SINGLE – 04
INSTALLATION GUIDE

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Note: The program has not been tested with a diffractometer under Win-2000 or XP. It does run in " dummy mode" under Win-2000.

IMPORTANT: If you are installing this version as an upgrade to a previous version of Single04, do not follow section 1. Go instead to section 2, NOW!

1: NEW INSTALLATION

1.1: The normal distribution consists of:

- One or more executables, *single04_*.exe*
- One or more diffractometer profile files; *difprof_*.dat*
- several *dll* files
- *gino.con*
- *pdf* files of manuals
- *pdf* file of distribution notes

1.2: Choose the executable appropriate for you instrument. There are currently two versions:

- *single04_smc9000.exe* for Huber SMC-9000 controllers of Huber diffractometers (as installed in the Bayerisches Geoinstitut, University College London, ETH Zurich)
- *single04_ams.exe* for AMS motor controllers and a Keithly counter card as installed in the Virginia Tech Crystallography Lab.

Copy the appropriate exe file to a directory such as C:\SINGLE04 or to a directory under \my documents. If you are only using the program in dummy mode (without diffractometer) you can use either executable.

1.3: Copy the following files to the same directory:

- the appropriate diffractometer profile file. Rename it *difprof.dat*
- all the *dll* files
- *gino.con*

This directory should be kept for the program files only. Data files should be placed in other directories.

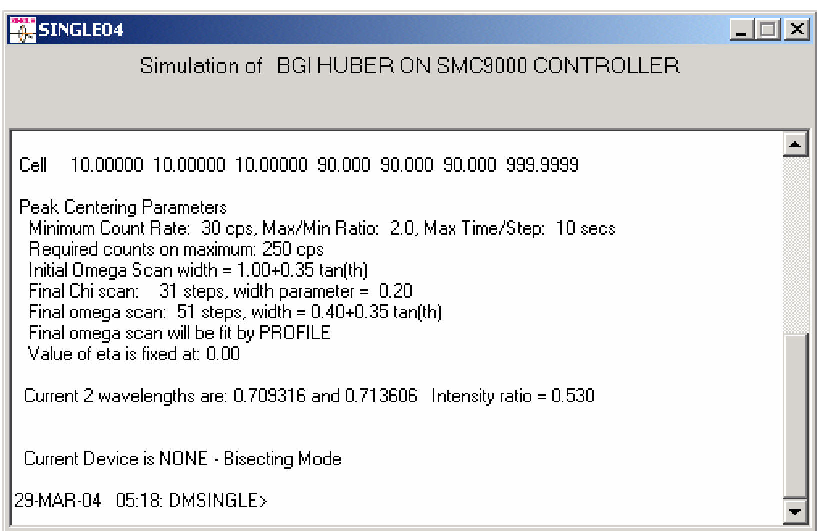
1.4: Copy the *pdf* files to a separate directory (e.g. \Single04\docs)

1.5: Create a directory for test data.

1.6: Create a shortcut to the executable on the desktop.

1.7: Test the installation. Start the program from the desktop. Answer "NO" to the question about running a "real" diffractometer. If the installation is ok, a file browser will appear. Proceed as follows:

- Use the browser to select the data directory created in 1.4
- Type in the name for the experiment ("mat") file. Do not use an extension.
- Answer "YES" to do you want to create a new matfile
- The program should now load some default parameters and the window should look something like this:



1.7: If this window appears then you have installed the program successfully, and it can be used to do crystallographic calculations without a connection to a diffractometer. See the single manual for details of commands and functions. If you now want to control a real diffractometer, type "exit" at the prompt to close the program and proceed to step 3.

2: UPGRADE

2.1: If you are just upgrading the program from a previous version of SINGLE04, do not overwrite the previous version of the single executable! (This allows you to use the old version if the new version does not work.)

2.2: Copy the distribution notes to the documents directory. Read them for any specific instructions. Take care not to over-write old versions of files!

2.3: Unless the distribution notes say otherwise, you only need to copy the new Single executable to a new filename in the same directory as the previous executable. A good name would be single04_*date* where *date* is the date of installation. Redirect the desktop shortcut to point at the new executable and run the program. The new program will use the old difprof.dat file.

2.4: Do not overwrite your copy of *difprof.dat* with the distribution version unless the distribution notes say otherwise! The version of this file in the distribution is generic and **will not contain data specific for your diffractometer!**

2.4: Now proceed to section 5 to test the new version of Single.

3: COMMUNICATIONS FOR HUBER SMC-9000 CONTROLLER

3.1: Make up an RS232 cable to connect a COM port on your PC to the serial port on the SMC9000. The socket on the SMC-9000 accepts a 25-way D-plug. The serial line on your PC may be a 9-way or a 25 way.

Wiring:

At PC end of the cable short together CTS, DSR and DTR. On a 25-way plug these are pins 5,6, and 20. On a 9-way plug these are pins 8,6, and 4.

At Huber end of the cable short together pins 5,8, 25.

Connect through cable:	PC end pin	2	to Huber end pin	3
		3		2
		7		7

3.2: Initially configure the SMC9000 communications to the following:

1200 baud
8 data bits
1 stop bit
parity none
terminator CR/LF

3.3: Edit the difprof.dat file and change the COMPORT line to read (for using port 1, usually com1:):

COMPORT 1,1200,0,8,0

The first number is the port number, so change that if necessary. It is best to start at 1200 baud as the slower communications rate is more reliable for debugging problems. Once you are sure things are working, then you can exit the program and reset the SMC9000 controller to a higher baud rate and the value on this COMPORT line to the same value. See below for the meaning of the other numbers.

4: DIFFRACTOMETER PROFILE FILE

The diffractometer profile file named difprof.dat contains two types of information:

- Diffractometer characteristics (motor settings, hardware limits, configurations)
- Default scan and data collection parameters

First, you will have to modify the difprof.dat file to match your hardware and the settings on your SMC9000 controller. These entries in the file that you need to change are listed under section 4.1. Later you can modify the default scan and data collection parameters listed under section 4.2.

The difprof.dat file contains a number of lines, which may be present in any order. Each line begins with a 6 letter code word, or 6 blank spaces. All lines with 6 blank spaces at the beginning are ignored; they can be used to insert comments. The allowed code words (in bold) with examples from the Bayreuth Huber are:

4.1 Diffractometer characteristics.

DIFNAM Name of the diffractometer. This will appear on the GUI.

CONFIG -1,-1,-1,-1,0,0,0,1,0,0,500,1
parity(4),ndcp,nspcchi,i_mono,dslit,hslit,ifilt,shtdly,ishut_check
- interface and diffractometer configuration:

Parity(4)	Parities (+/-1) of the 4 circles, relative to a Busing-Levy diffractometer
ndcp	ndcp=1 for a diffractometer interface which treats omega as absolute, 0 for an interface that works with omega relative (eg a Picker).
nspcchi	compensation on phi for chi drives (for Picker)
i_mono	1 if monochromator installed, 0 otherwise
dslit	1 if motor driven slits available, 0 otherwise
hslit	1 if half-slits available, 0 otherwise
ifilt	1 if filter-wheel available, 0 otherwise
shtdly	shutter delay in msecs. (Delay before next motor drive or scan)
ishut_check	1 if shutter status feedback is enabled, 0 otherwise

GONCON 470.0,370.0
-diffractometer size:

U0 source to crystal distance in millimetres

V0 crystal to detector distance in millimetres

COMPORT 1,9600,0,8,0

-communications parameters (*all integers*). **These must match the settings in the Huber SMC-9000 interface**. See the Huber manual for the SMC-9000 for details. In order the parameters are:

iport	port number (see PC manual). COM1: or COMA: is usually iport=1
baud	baud rate for the com line. Set equal to the value for the SMC-9000
parity	0= none,1=odd,2=even,3=mark,4=space (normally 0)
databits	number of databits on the comport (normally 8)
stopbits	The stop bits for the port (0, 1, 2 = 1, 1.5, 2 respectively). Normally set 0 to mean 1 stop bit.

MOTORn

Motor parameters. These depend on the diffractometer. For the Huber SMC-9000 interface they are (in order):

Steps/degree
Slew Rate (in Hz)
Ramp rate to slewing rate
Med speed rate (in Hz)
Ramp rate to medium speed
Minimum circle limit
Maximum circle limit

For omega, the last number is the minimum angle of inclination between the 2theta arm and the plane of the chi circle.

Motors are numbered 1 thru 4 for 2theta, omega, chi and phi. If you have motorised slits on your detector, these are motors 6 for the ones defining the theta-2theta width of the detector and 5 for the other ones. Motor drive instructions are always rounded off explicitly to the resolution of the diffractometer as defined in the file *difprof.dat*. Note that the Bayreuth/Huber version of the code does not normally read back motor positions from the motor controller.

For motorised slits, only the stpdg (interpreted as step per mm) and the limits are used.

The steps/deg in the difprof.dat file must match those loaded directly to the SMC-9000 interface. The supplied difprof.dat works with the following SMC-9000 settings:

Axis	**Type**	**GRN**	**GRD**	**Ref Off**	**Ref Freq**	**Run Freq**	**Fast freq**
1	Goniometer	1000	1	90.0	10,000	1,000	10,000
2	Goniometer	1000	1	0.0	10,000	1,000	10,000

3	Linear Table	1000	1	0.0	8,000	1,000	8,000
4	Linear Table	1000	2	0.0	10,000	1,000	10,000
5	Slit screen	400	1	0.0	1,000	200	1,000
6	Slit screen	400	1	0.0	1,000	200	1,000

Notes:
1: The gear ratios GRN and GRD define the number of motor steps per degree. GRN/GRD = 1000/1 means 1000 steps per degree, or 0.001^{o}/step. GRN/GRD = 1000/2 is 0.002^{o}/step, and stpdg for motor 4 in the difprof.dat file is therefore 500. See the Huber manual for the SMC-9000 series controller for more details.

2: All motors on the BGI Huber have level for positive direction =0 and limit switch attachment=1

The circle limits should be set in the difprof.dat file to be slightly less than the position at which the physical limit switches are activated. Once you have got the program talking to the diffractometer, check these values carefully! The values for the omega limits in the file are absolute omega.

MONOC 12.2,90.0,0
Monochromator information. Not needed if no monochromator (imono=0 on config card). If monochromator is present, the numbers are:
- Monochromator 2theta value
- Monchromator dihedral angle (0 if monochromator diffraction plane is parallel to sample plane, 90 if perpendicular)
- Iparallel. Only needed if dihedral angle=0. Then 1 means dispersions of crystal and monochromator add on positive 2theta side of diffractometer, -1 if dispersions add on negative side.

WAVEL 2,0.709316,0.713606,0.52
NWAVE,WAVE1,WAVE2,WRATIO

NWAVE is the number of wavelengths in the incident beam, WAVE1 and WAVE2 their wavelengths, and WRATIO the intensity ratio I(WAVE2)/I(WAVE1). Determine WRATIO from fitting scans of well-resolved high-angle strong reflections.

VIEW 30.,0., 0.,50.
Preset 2theta,omega,chi, and phi for the view position. Adjust these so that the slides of the goniometer head are parallel and perpendicular to the axis of the telescope at the view position.

PARK 0. ,0.,90.,0.,2.,9.
Preset 2theta,omega,chi, phi and slit positions for the park position to which the diffractometer is driven on normal program termination.

PHOTO 0. ,0., 0.,0., 300.
Preset 2theta,omega,chi, and phi for the photo position, and the crystal-to-film

distance.

OFFSET 0. ,0., 0.,0., 0.,0.,0.

Offsets of diffraction-defined zero positions of the circles from the zero positions of the motor controllers. The diffractometer is driven to a position of the angle *plus* the offset. Thus, if the direct beam hits the detector when the controller value for 2theta value is +10^o the offset for 2theta should be +10^o. When you use the software to then drive to +15^o 2theta, the motor controller will drive to +25^o. The omega offset is specified in absolute omega.

4.2 Default scan parameters

SCANS1 30.0,2.0,10.,250.,1.0,0.35,2.0,9.0
-default parameters for centering reflections, only used when creating a new mat file. In order:

MINCT minimum count on a centering scan for a reflection to be centered
MAXMIN the required ratio of the max to min counts on a centering scan
TMAX Max allowed time per step
maxct Required counts at max
OMW_I1,OMW_I2 Width of the first omega scan during the centering procedure is OMW_I1 + OMW_I2*tan(theta))
M5slit width of the vertical detector slit for zref
M6slit width of the horizontal detector slit for zref

SCANS2 31,51,0,0,0.2,0.4,0.35,0.0
- more default parameters for centering reflections, only used when creating a new mat file. In order:
N_CHI,N_OMEGA,IBAD,FIXETA,CHIW,OMW_F1,OMW_F2,ETAVAL

Final chi scans in centering are performed with N_CHI steps, and a width given by CHIW*sqrt(slit5width/sin(theta)).

Final omega scans in centering are performed with N_OMEGA steps, and a width OMW_F1+OMW_F2*tan(theta).

If ibad=0 then the final omega scans are fitted with a Pseudo-Voigt function with ETAVAL as the default value of the mixing parameter (ETAVAL=0 is Gaussian, ETAVAL=1 is Lorentzian). The value of eta is fixed in the fitting routine if fixeta=0, and refined if fixeta=1

If ibad=1 then the final omega scans are fitted with a parabolic function instead of a pseudo-voigt. Recommended for crystals with very poor peak profiles.

SGROUP 0 P 1
space group for data collection. The leading integer is the lattice type number, 0=P

DSCAN1 1.00 0.35

the scan width for data collection. The first number is the basic width and the second term is the dispersion term. The example will set a width of $1.0+0.35\tan\theta$. The width is either omega or 2theta, depending on the scan type specified in the DSCAN2 line.

DSCAN2 1.000 0.000 0.020 5.000 0.000 10.00 1.000 0.000 0.000 0.000 0. 0.
Data collection parameters, in order:

- Scan type, 1 for omega scan, =2 for om/2th
- Number of psi positions collected for each reflections
- Step size for scan
- Time per step (secs) for initial scan
- Mode: 0 for single scan, 1 for constant precision
- required precision I/sigI
- maximum no. repeat scans in constant precision mode
- space group absence flag: =1 collect only sp gp allowed, =2 collect only sp gp absent, =0 ignore sp gp, collect all
- increment in psi for psi scans
- reorientation flag; =0 no reorientation, 1 otherwise (Not currently used)
- minimum I/sigI to allow rescan in constant precision mode.
- = 0 for no psi scans, =1 for psi scans

HKLORD 0 1 0 1 0 1 1 2 3
Limits and sequence for indices.

ALLSTD 2 100
Number of standards and frequency (here 2 standards every 100 reflections)

HKLST 1 1 0 1 1
Standard reflection; standard number followed by *hkl* and use flag

SHELL 1 0. 60. 1
Shell information; shell number followed by 2theta limits (min, max) to shell, and use flag.

5: COMMUNICATIONS TEST

5.1: Before you try to drive the diffractometer, run through this checklist:

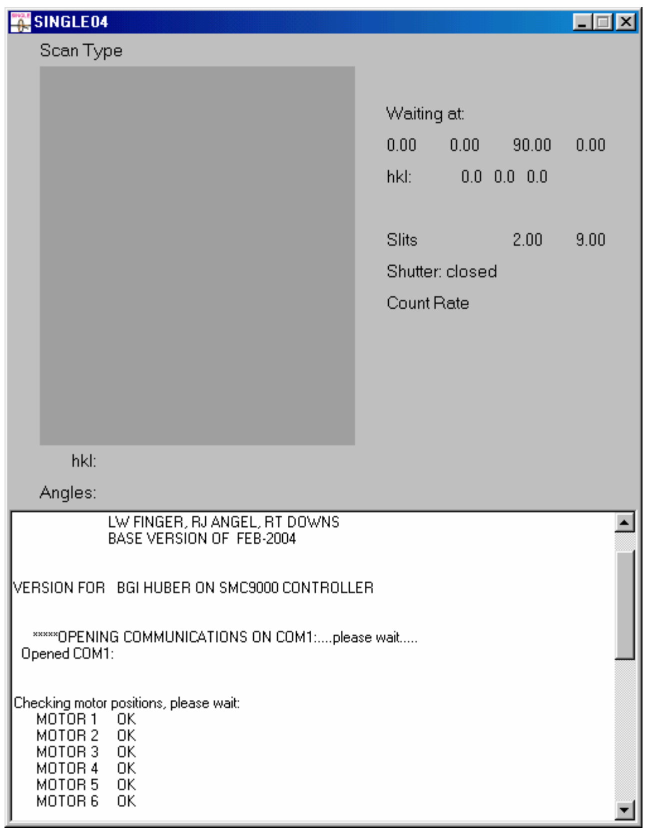
- The Single program works in “dummy” mode (section 1.5)
- The cable is properly connected between the motor controller and the com port.
- The com port and the com port parameters are correctly written in to the diffractometer profile file.
- The motor controller communications parameters are set to the same values as those in the *difprof*.dat file.

5.2: Start the Single program from the shortcut on the desktop

5.3: Answer “YES” to the question about running a “real” diffractometer. If the installation is ok the main window will appear. In the scrolling region of the window, the following messages will appear in order:

- “OPENING COMMUNICATIONS ON COM**n** ....please wait ”
  - check that **n** is the com port number you want
  - if the com port cannot be opened, or comms stall, then you will either get an error message or nothing will happen. Close the program and fix the problem – it is probably a parameter set incorrectly in the comport line of difprof.dat
  - if successful you will see “Opened COM**n**” and it will proceed to...

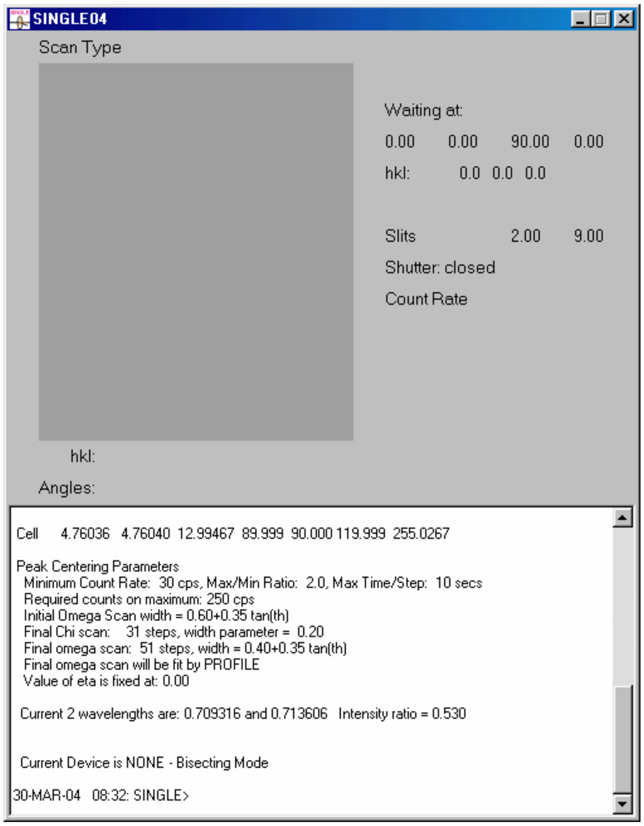
- “Checking motor positions, please wait:”
  - For each motor you should see either “OK”
  - or a message to say that the stored position in the controller does not match the park position
  - or some other error message. Correct the problem and restart the program.



5.4: Then the positions and shutter state will be updated on the display, and a file browser will appear. Proceed as follows:

- Use the browser to select your data directory.
- Type in the name for the experiment (“mat”) file. Do not use an extension.
- Answer “YES” to do you want to create a new matfile

- The program should now load some default parameters and the window should look something like this:



5.5: The program is now in communication with the SMC-9000 controller. Do the following, carefully to determine whether the program is working correctly:

- mot command (test phi first! Make sure that the physical movement, the position on the SMC-9000 controller, and the display on the Single GUI agree)
- ldmt command
- open/close shutter: check error handling when enclosure door open
- drives: check error handling when limit switch is activated during a drive (after doing this you will have to press the "reset" button on the SMC-9000 and may have to reinitialise the controller. See local instructions).
- Carefully (I mean it) check that the motor limits in difprof.dat are correct. If they are, then you should be able to drive the diffractometer to close to the limit switches, but not on to them. Double, double check for collisions not protected by limit switches, such as the detector arm on to the chi circle, and the phi cradle on to either the collimator or the detector arm. If you need to change the limits you must first close the program, then edit the difprof.dat file, and restart the program.
- count
- prof (all of omega, omega/2theta, and 2theta only)

If all of these facilities work then try, in order, the following centering routines for one strong low-angle reflection. Time the process-

- cent
- cntr
- zref

You may have to adjust the scan parameters (use set center) to get the best results. As a guide, the motor 5 slit should be adjusted so as to get a symmetric and close to Gaussian peak shapes on the final chi scan of centering. The final omega scan should be of a width so that the peak occupies about 1/3-1/2 of the total scan width. Once you have good parameters, edit them into the difprof.dat file.

If OK, then switch to fixed-phi mode and repeat.

The last check is then to do a zref on a set of reflections from your standard crystal and examine the resulting scans with the WinIntegrStp program. You should obtain good unit-cell parameters, and there should be no significant variation in the delta-d/d plot. If there is variation, then you may need to adjust either the alpha1/alpha2 intensity ratio and/or the profile parameter eta. Adjust these with WinIntegrStp until you minimise the slope and scatter in the delta-d/d plot. Once you have good values, edit them back into difprof.dat and repeat the measurement.