Siemens D5005 manual

part 1. reflectometry

starting up

Start up the computer, login user d5005, password d5005

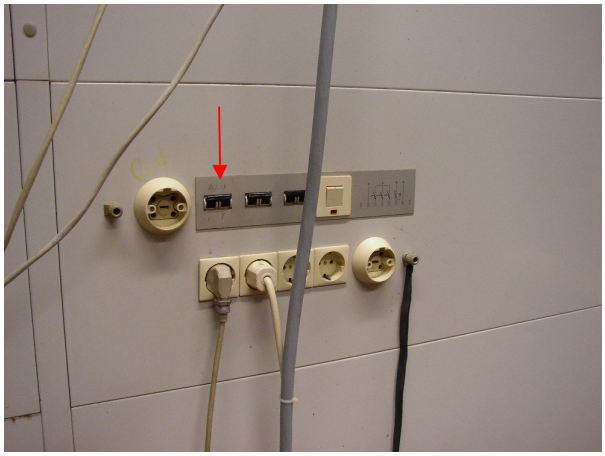
Sratup the D5005:

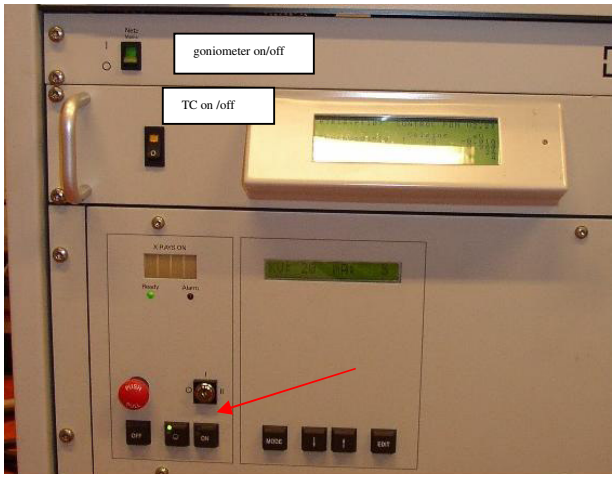
When the 220 V mains power was shut down, the system needs a special startup sequence:

1) water cooling on, manual valves + electrical valve (on wall behind D5005)
2) goniometer (netz) on ,
3) TC on,
4) generator on, 3 buttons below key, press middle button, wait until green light stops blinking, press right button

5a) computer: go to icon TC to initialize communication with TC, click OK,

5b) go to immediate measurement icon and double click

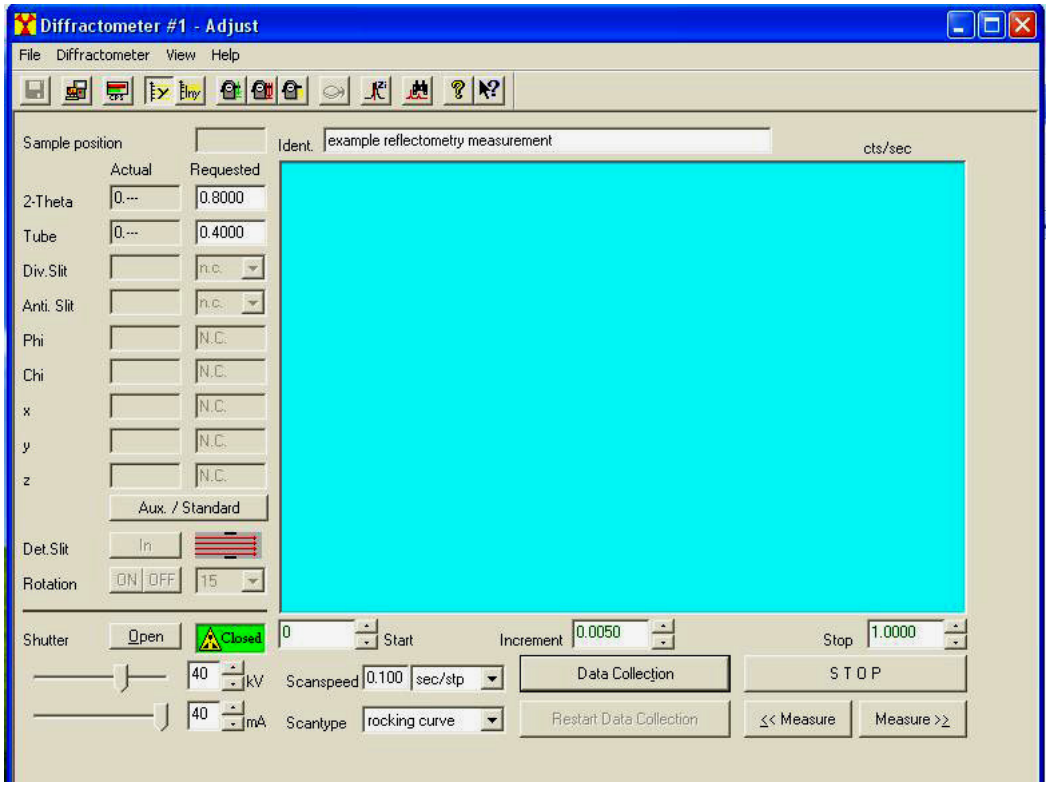




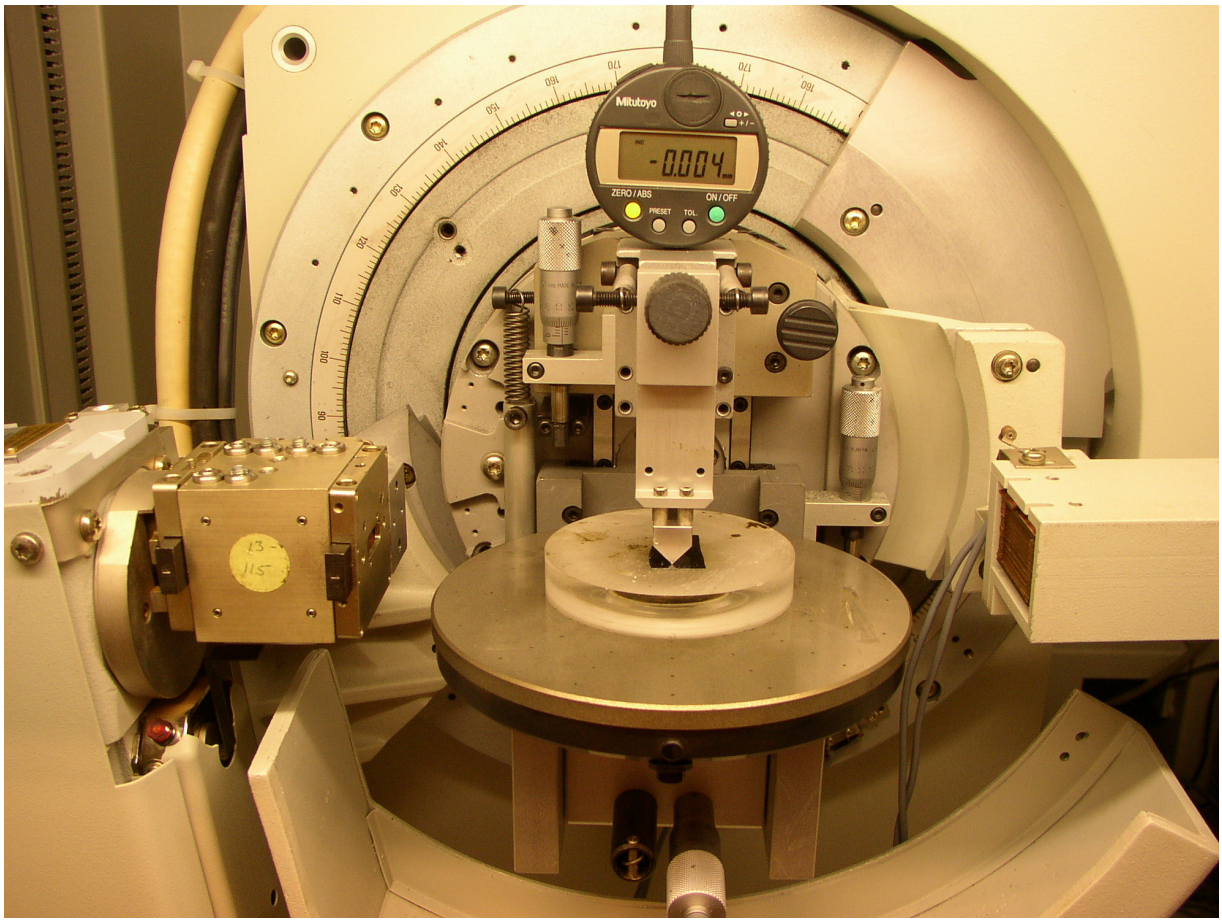
normal startup:

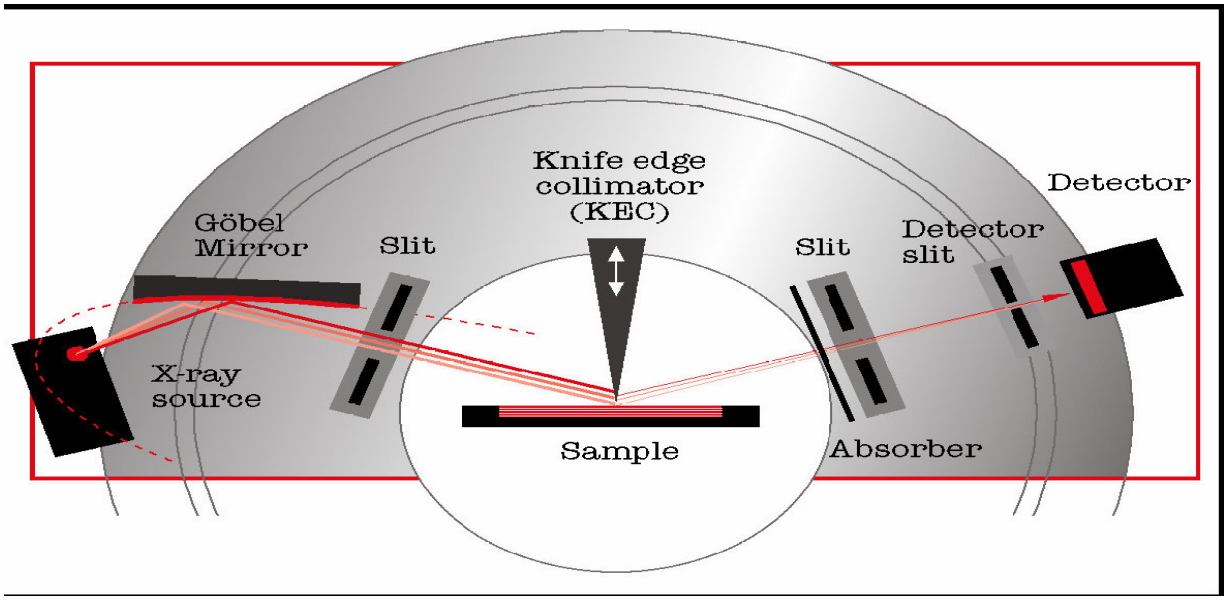
1) water cooling on, manual valves + electrical valve

2) generator on, 3 buttons below key, press middle button, wait until green light stops blinking, press right button
3) go to immediate measurement icon and double click



Acquiring reflectivity data with the Bruker D5005





The method described below is what is required only for the simplest reflectivity measurements. Bruker's Refsim can be used to model the data obtained. To obtain a reflectivity curve it is first necessary to align the reflectometer to the sample, this is essential because data must be collected with the angle of incidence being equal to the angle of reflection. The alignment needs to be very precise because the data is collected at small angles. Once the alignment is complete the data can be acquired in one of two programs either using immediate measurement (the simplest method) or job measurement (not described here)

Aligning the reflectometer to the sample

1) The sample must then be placed at the correct height. Lower the knife edge to almost on the sample (take care not to crash it into your sample).This is done using the electronic height meter, observing the amount of light still passing through the slit.

2) To enable you to make the beam parallel to your sample a rocking curve must be obtained. To do this set the scan type to rocking curve, continuous, the scan speed to 0.1 sec/step, the start value to 0.0 and the stop value to 1.0, and a 2θ value of 0.8 °. Start the scan by selecting Data Collection. A peak of about 20000 counts and a full width at half maximum of less than 0.1^{o} should be observed, allow the system to collect for sufficient time and then press stop. To determine the peak position click the Zi button in the top row of buttons (or select Determine Zi Value in the Diffractometer menu) a Zi determination box will appear. Note the Calculated Peak Position and make sure that Enter theoretical position box contains the value of θ= 0.4, click Save and Send new ZI. A warning box appears "All circles will be initialized "(check crashing possibility)!

3) Step 2 has only changed the angle of the tube and hence you now need to apply the same correction but in the other direction to the detector. To do this enter 0.4 for the θ-val (tube). Perform a detector scan from 0 to 2.0. To determine the peak position click the Zi button in the top row of buttons (or select Determine Zi Value in the Diffractometer menu) a Zi determination box will appear. Note the Calculated Peak Position and make sure that Enter theoretical position box contains the value of 2θ= 0.8, click Save and Send new ZI. A warning box appears "All circles will be initialized "(check crashing possibility)!

N.B. For samples with low angle of total reflection choose a value smaller than the angle of total reflection for θ and twice that value for 2θ.

4) Step 3 has now changed the angle of the detector and hence you might need to apply a small correction to the tube again. To do this enter 0.4 for the 2θ-val (2-ThetA). Perform a tube scan from 0 to 1.0. To determine the peak position click the Zi button in the top row of buttons (or select Determine Zi Value in the Diffractometer menu) a Zi determination box will appear. Note the Calculated Peak Position and make sure that Enter theoretical position box contains the value of θ= 0.4, click Save and Send new ZI. A warning box appears "All circles will be initialized "(check crashing possibility)!

5) Perform a Locked Coupled scan from 0.4 to 1 °. For checking the absolute position of the angles compare the measured reflectivity curve with the one simulated by REFSIM. The critical angle (total reflection) should be correct within 0.01 °.

If not, note the difference between measured and calculated curve (in 2θ °). Go to the icon "Config"and double click.

Password DIFFRAC. Go to "*options" , "axes settings*". Add (subtract) from these values the difference value (divided by two, θ value) you noted. Save and download.

Repeat now Step 2 to 5.

Obtaining a reflectivity curve using Immediate Measurement

To obtain a reflectivity curve set the scan type to locked coupled, continuous and the scan speed, start, increment and stop parameters to what you want for your sample. Most used values will be from 0.4 to 3.0 ° twoθ, 2 sec/step. The data can be displayed on a linear scale by clicking the graph button with a y on it or on a logarithmic scale by clicking the graph button with lny on it (although this displays as base 10 logarithmic plot!). Once sufficient a number of scans have been collected for you to resolve the features you are interested in have been completed click Stop. To save the data select Save As from the File menu and choose a file name and then save it into your data directory.

Data analysis of the reflectometry measurement

The XRR spectra are analyzed with the REFSIM software (Bruker) in order to extract information on the density, thickness and surface and interface roughness.

How to use this program?

1. Enter new compounds in the database

In most cases the compounds used are already listed, if not continue item 1, else proceed with 2.
The program is delivered together with two databases.
The element database contains already all the information required for most applications. You will only rarely have to change data in this database. In order to solve the first problem in this tutorial (SiO2 film on Si substrate) you have to click (left) on the database field. The program offers access to the element database and to the compound database. Drag the mouse (while the left mouse button is pressed) to the compound field. The submenu database : compounds is opened. The cursor appears in a field where you can enter the chemical name of your new compound: Enter e.g. silicon oxide.
Enter a chemical formula into the next field, e.g. SiO2, Enter the density (g/cm3) of your new compound (if you know it) in the next field, e.g. 2.65. These three input fields serve mostly as reminders and reference for your future work. You still have to enter the correct stoichiometry of your compound. The input columns on the right side of the menu serve for this purpose.
Select in the topmost line the first element in your compound (scroll or select with first letter), e.g. Si.
Specify the fractional part of this element in the compound, e.g. 1 (1 Si in SiO2).
Select in the second line the second element in your compound, e.g. O.
Specify the fractional part of this element in the compound, e.g. 2 (2 O in SiO2).
Ten input fields for stoichiometry are present (10 possible elements per compound). You may as well specify stoichiometries like Si0.96O1.45H2.21. Once you have specified a new compound, you may add it to the database with the Add button. Compounds already stored in the database can be marked with the mouse. All parameters connected with the marked compound appear in the input fields. These parameters can be changed, and the old compound can be replaced with the Replace button. The marked compound can also be deleted with the Delete button. Check whether the second compound we need for our example (Si) is in the database. If not, enter it. Your work in this menu is now finished. Click on EXIT.

2. Edit the simulation parameters

The main menu contains a field Edit. The upper submenu Simulation Parameters allows the choice of the simulation range (incident angle in °) and step width. Specify a range from 0° to 2°.
The program is set to copper radiation by default. Absorption is by default being taken into account.
The simulation mode is by default set to specular reflection (other modes are not operating yet).
Other input is at this time not necessary. Click on OK.

3. Edit the sample

Enter the field Edit Sample.
a) substrate :
Click on the field layer type (right top), and select sub (substrate). The field name is a window in your compound database. Select silicon from the database. Press the Add button to define the substrate.
b) layer Click on the field layer type and select uni (homogeneous layer). Pick from the name window silicon oxide. Enter thickness, roughness and density for this layer (e.g. 100nm, 0.5nm, 2.45g/cm3). Press the Add button to deposit the layer on the substrate. Leave this menu with the OK button.

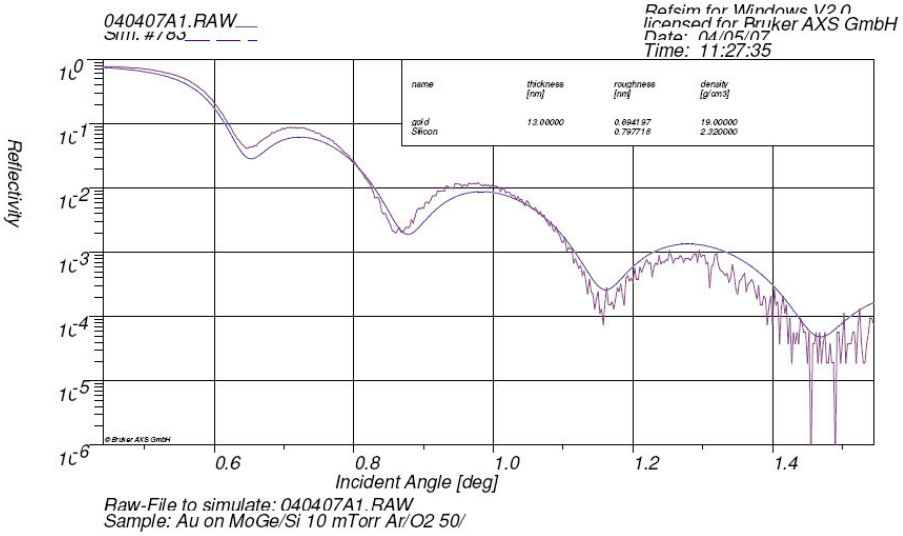
4 Run the Simulation

Click Calculation on the main menu. Drag mouse to Simulation. Click Start.
The program now calculates and displays the reflectivity curve for the specified sample.
You can also specify an increment or decrement for one or more parameters and apply this change every time you click on Start. Try out some of the commands offered in the main menu command

Graphics. The simulation can be printed. The sample setup can be brought into shape and printed via any editor you like (specification of the editor in the main menu command System / Software Settings). The simulated curve can be stored as binary file (*.SIM) or ASCII file (*.ASC) (main menu command File / Save). The stored file can be reread and displayed like a raw data file. displayed file.

5. Compare/Fit with measured curve

Go to “File”, “Open”, select the measurement *. raw file. Go to “Calculation”, “Refinement”, “Simplex”. Click on the parameters you want to refine, and “Start”.



part 2. measurement of lattice parameters of a thin epitaxial film

Aligning the sample. (as an example we chose (LaCa)MnO3 on SrTiO3).

1) remove the knife edge assembly after adjustment of the sample surface at zero height (0.000).

2) From tables/ literature find the most intense peak of the substrate, e.g. the <0 0 2> peak of SrTiO3 is to be found at 46.4834° 2θ, (or the <0 0 4> of silicon at 69.00 °). To align the beam parallel to the chosen crystal plane of the substrate first a rocking curve must be obtained. To do this set the scan type to rocking curve, continuous, the scan speed to 0.1 sec/step, the start value to θ – 1 ° and the stop value to θ + 1°, and a 2θ value of 46.483 °. Start the scan by selecting Data Collection. A peak of up to 100000 counts and a full width at half maximum of less than 0.1^o should be observed, To determine the peak position click the Zi button in the top row of buttons (or select Determine Zi Value in the Diffractometer menu) a Zi determination box will appear. Note the Calculated Peak Position and make sure that Enter theoretical position box contains the value of θ= 46.4834/2=23.2417, click Save and Send new ZI. A warning box appears "All circles will be initialized ".

3) Step 2 has only changed the angle of the tube and hence you now need to apply the same correction but in the other direction to the detector. To do this enter 23.2417 for θ. Perform a detector scan from 2θ – 1 ° up to 2θ + 1 °. To determine the peak position click the Zi button in the top row of buttons (or select Determine Zi Value in the Diffractometer menu) a Zi determination box will appear. Note the Calculated Peak Position and make sure that Enter theoretical position box contains the value of 2θ= 46.4834, click Save and Send new ZI. A warning box appears All circles will be initialized.

4) Step 3 has now changed the angle of the detector and hence you might need to apply a small correction to the tube again. To do this enter 23.2417 for 2θ. Perform a tube scan from to θ – 1 ° to to θ + 1 °. To determine the peak position click the Zi button in the top row of buttons (or select Determine Zi Value in the Diffractometer menu) a Zi determination box will appear. Note the Calculated Peak Position and make sure that Enter theoretical position box contains the value of θ =23.2417 °, click Save and Send new ZI. A warning box appears "All circles will be initialized "

5) Perform a Locked Coupled scan from to 2θ – 1 ° up to 2θ + 1 °.. The peak maximum should be correct (46.4834) within 0.001 °.

If not, note the difference between measured and calculated curve (in 2θ °). Go to the icon "Config"and double click.

Password DIFFRAC. Go to "*options" , "axes settings*". Add (subtract) from these values the difference value (divided by two, θ value) you noted. Save and download.

Repeat now Step 2 to 5.

6) Measure a 2θ range from 46 up to 50 °. In this range the <0 0 2> peak of the (LaCa)MnO3 will appear. Using the EVA (Evaluation) program the peak position can be determined.

2dsin θ=λ λ=1.5406 lattice parameter = d*sqrt(h^2+k^2+ l^2) where h=0 , k=0, l=2. (SrTiO3 is cubic)
For the tetragonal (LaCa)MnO3 film this approximation is OK for the <0 0 l> reflections.

Thus the lattice parameter c can be calculated. In practice, because of gaining accuracy and eliminating small zero-offset in 2θ, also the <0 0 1> and <0 0 3> reflections are measured.

Troubleshooting/ configuration

D5005 is connected to COM1 at the backside of the PC

Error messages:

error=5, 100 uF/63V Elco on motherboard generator has to be replaced
error=14 , water flow, clean filter in water container
error=22, pull up switch at the rear of the generator (inside the d5005)
error=30, means alarm light on top cabinet fails, change lamp bulb
error=40 , tube surge, renew cooling water in container of secondary circuit

Direct mode commands

(in program immediate measurement)

<IN> to initialize D5005

<TU100,1> tube at rate 100 goes up or down when pressing <up> or <down> and <tuning> on remote control box inside the xray cabinet

<TU100,2> detector at rate 100 goes up or down when pressing <up> or <down> and <tuning> on remote control box inside the xray cabinet

<FRU,1> <FRD,1> <FRU,2> <FRD,2>

when the detector and tube are slowly moving down (reference points are lost).

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Installation diffrac software

Diffrac software runs under Windows NT 4.0 and Windows XP

In order to establish communication with the D5005 you first have to activate the high temperature electronics:

you can put under an icon after creating a shortcut

d:\diffplus\TCload.exe /d:1 /f:HTK16.BIN

<immediate measure> which can be found as adjust.exe does not need dongle or license

You can put under an icon after creating a shortcut

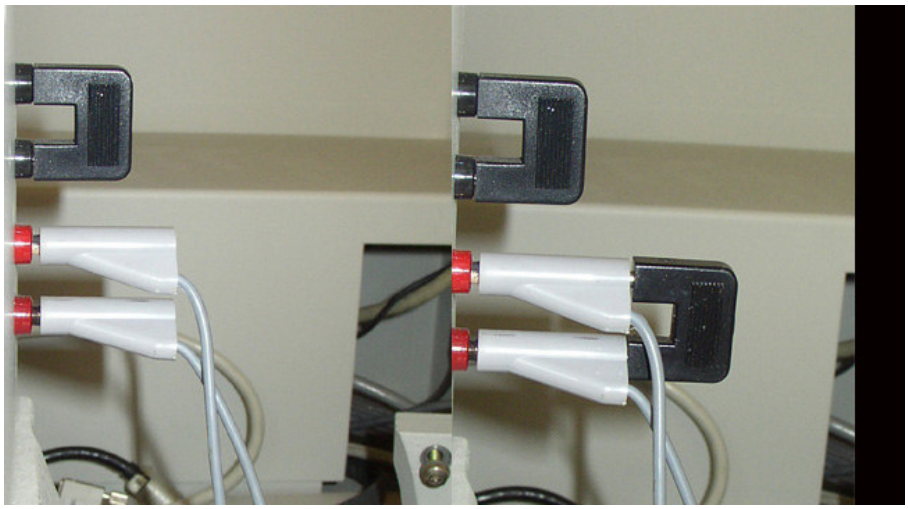
In <windows explorer> do <create shortcut> and type in “d:\diffplus\adjust.exe /d:1

<configure>

In <windows explorer> do <create shortcut> and type in “d:\diffplus\sag_cnf.exe /d:1

Crashing the tube and/or detector against end switch

In most cases this should not happen because of software limits, however when the detector or tube crashes against the hardware end switch, first find the cable going to the end switch, follow it to the banana connectors at the goniometer frame, short circuit the connection, in the “immediate measurement” program do to direct command and enter “in”, initialize. Immediately after this remove the short circuit!



Configuration file diff_no1.cnf

```
FORMAT :SAG Diffractometer Configuration File - free format style.
VERSION:1    CREATED;07/25/07   13:17:03 PROGRAM:SAG_CNF SYSTEM:WNT
ALARM  :A1:F A2:F A3:F A4:F A5:F A6:F A7:F A8:F A9:W A10:W A11:W
A12:W A13:W    ALARM  :A14:I A15:W
DET_NO1:Scintillation HV:840.0 AG:300.0 LL:0.7 WW:2.3 LS:N IM:N
DEADTIME:350.0  DET_NO2:None HV:825.0 AG:300.0 LL:0.7 WW:2.3 LS:N
IM:N DEADTIME:350.0           DEAD_EQ:None
TEMPCTL:PORT:Integrated LIMITS:10.0 1400.0 100.0 UNITS:C
COM1   :BAUD:9600 BITS:8 PARITY:None STOPBITS:1 FLOW:Xon/Xoff
COM2   :BAUD:9600 BITS:8 PARITY:None STOPBITS:1 FLOW:Xon/Xoff
COM3   :BAUD:9600 BITS:8 PARITY:None STOPBITS:1 FLOW:Xon/Xoff
COM4   :BAUD:9600 BITS:8 PARITY:None STOPBITS:1 FLOW:Xon/Xoff
PHI    ;0.0 4.0 5400.0 43200.0 0.5 17.0
CHI    :90.0 400.0 50.0 150.0 0.5 115.0
X      :0.0 200.0 2.0 10.0 0.5 121.0
Y      :0.0 200.0 2.0 10.0 0.5 121.0
Z      :0.0 200.0 2.0 10.0 0.5 121.0
DIVSLIT:345.0
ANTSLIT:345.0
AUX1   :0.0 10.0 2.0 10.0 0.5 0.0
AUX2   :0.0 10.0 2.0 10.0 0.5 0.0
AUX3   :0.0 10.0 2.0 10.0 0.5 0.0
2THETA :59.5715001883507 5000 100 600 0.5 42
THETA  :32.0135983105659 5000 100 600 0.5 42
SITE   :Universiteit Leiden
SITE   :Kamerlingh Onnes lab.
USER   :D5005 user
MODEL  :TH/TH D5000 CONTROLLER:SMD6+TC PORT:COM1 STAGE:Standard
CHANGER:None    MODEL  :RADIUS:217.5
MOTORS :2T:Y TH:Y PH:N CH:N X:N Y:N Z:N DS:N AS:N SC:N A1:N A2:N A3:N
SYNCH:NoneOPTICSI:NEARSLIT:1.0 DIVSLIT:1.0 SOLLER:N
MONOCHROMAT:Goebel Mirror             OPTICSD:ANTSLIT:2.0 DETSLIT:0.1
SOLLER:Y THINFILM:N FILTER:N ANALYZER:None      GENERAT:Sealed tube
PORT:Integrated LIMITS:20.0 50.0 5.0 40.0 2.0 WARMUP:60.0
GENERAT:STANDBY:20.0 5.0 300.0 DEFAULT:40.0 40.0
WAVELEN:CuKav:1.5418 CuKa1:1.5406 CuKa2:1.54439 CuKb1:1.39222
Cua2/a1:0.08      WAVELEN:UNITS:A
LIMITS :TH:-5.0 46.0 2T:-5.0 92.0 PH:-360.0 360.0 CH:-360.0 360.0 X:-
30.0 30.0  LIMITS :Y:-30.0 30.0 Z:-30.0 30.0 DS:0.0 450.0 AS:0.0
450.0 TH-2T:-720.0 720.0  LIMITS :TH+2T:-720.0 720.0 A1:0.0 0.0
A2:0.0 0.0 A3:0.0 0.0                    FILENAM:diff_no1.cnf
WORD   :4096
DATA   :END OF FILE
```

In case of serious problem, contact:

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