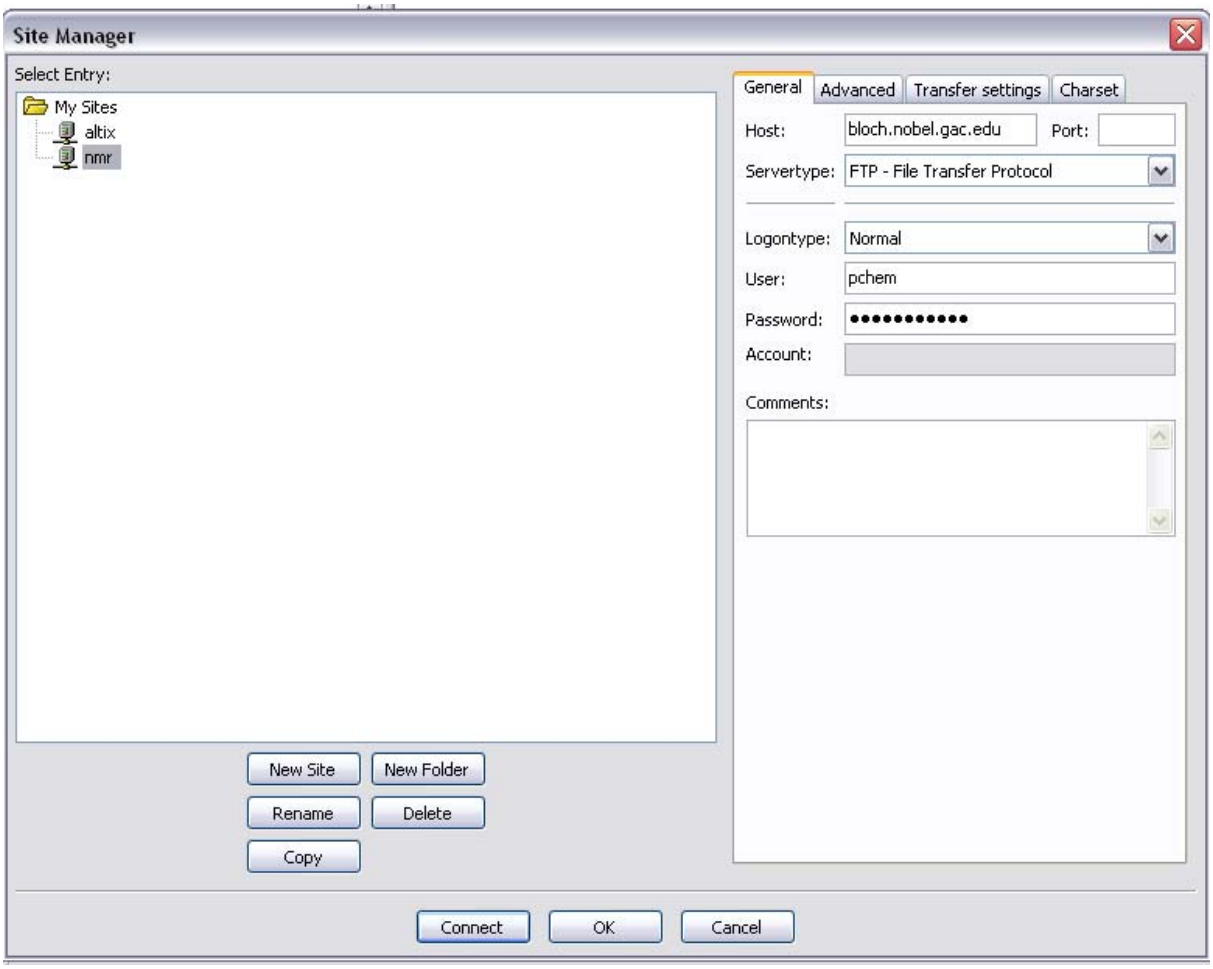
Fitting NMR peaks for N,N-DMA

Importing the FID file to your local system

Any ftp program may be used to transfer the FID file from the NMR computer. The description below will take you through the process using Filezilla (a freeware ftp/sftp program).

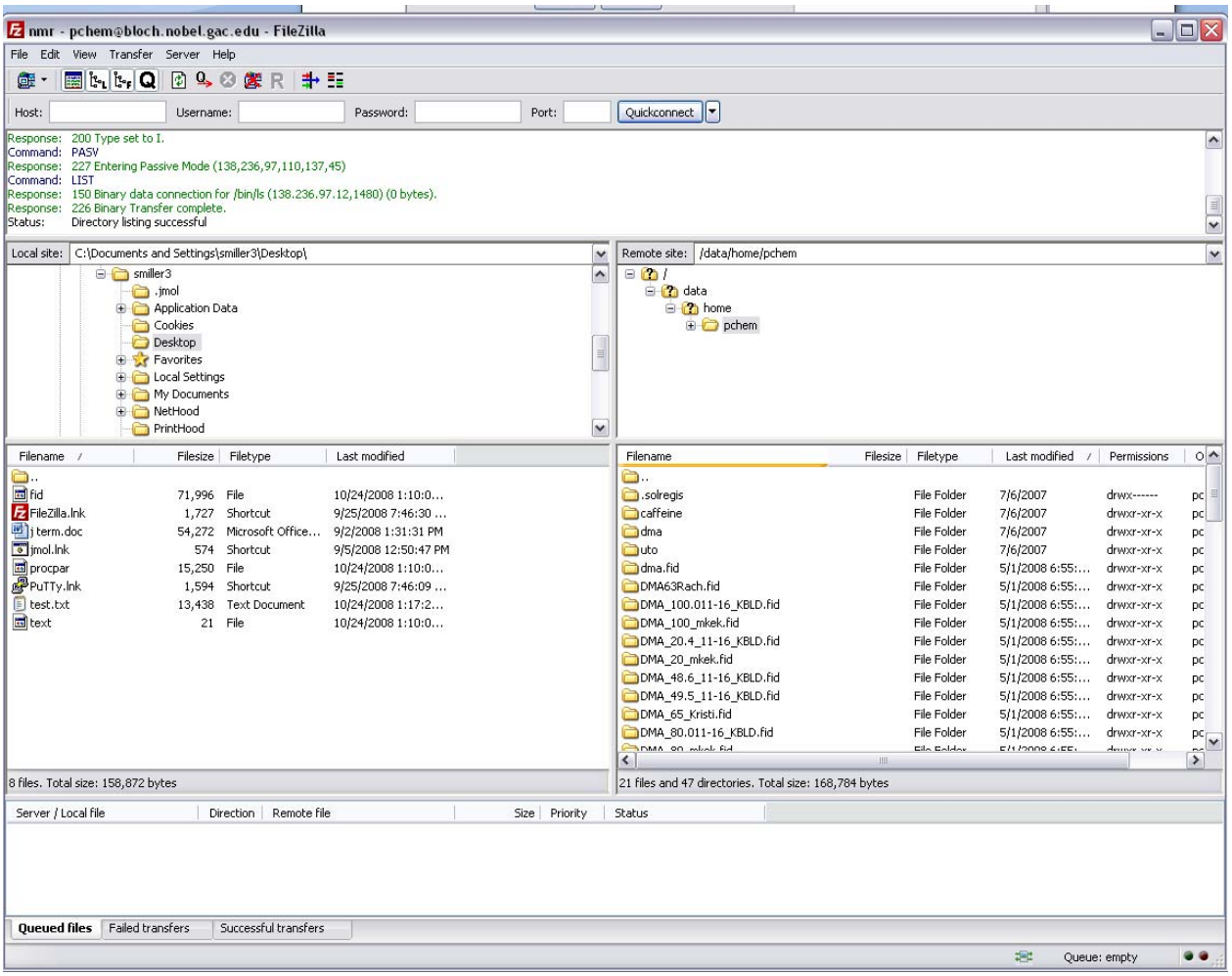
1. Open Filezilla (if the program is not on your computer, simply Google "filezilla", and you will find many download sites. Download and installation should be straightforward).

2. Under the "File" tab, select "Site manager". A new window should appear:



If you have not set up a connection to the NMR server, click "new site" and enter a name in the left hand pane ("nmr" in the example above). Enter bloch.nobel.gac.edu for the host, ftp for the servertype and normal for the logontype (all as shown above). Enter the username and password you used when logging onto the NMR to collect the spectrum originally and click "Connect". Note: if you have already done this, you can go into the site manager, select the correct site from the left hand pane, and click "Connect".

3. The Filezilla window should now contain two separate file trees, one for the system you are on (left) and a second for the NMR computer (right):

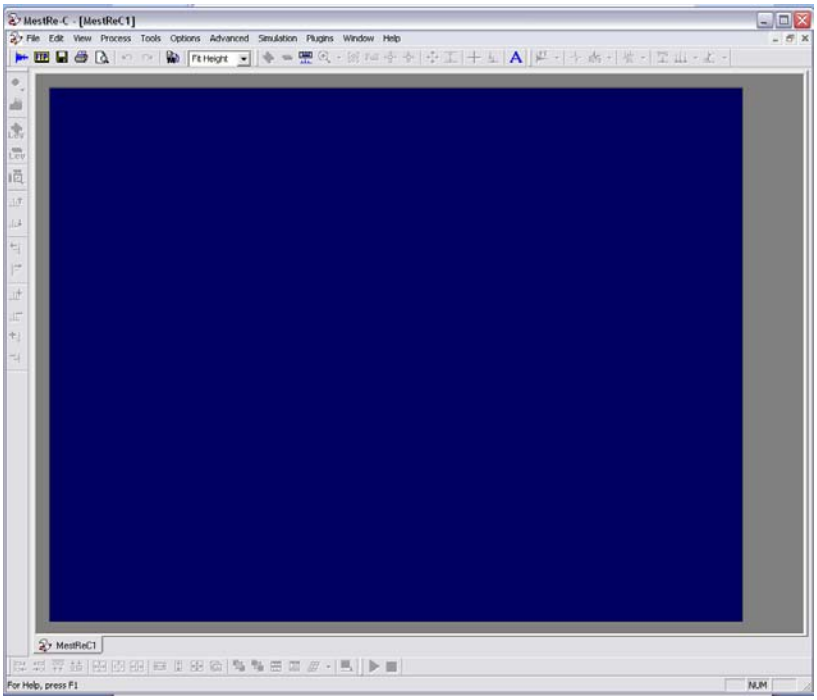


In the left hand side of the Filezilla window, browse to the folder/location on your computer you wish to move your NMR files to. Then, in the right hand side of the Filezilla window, browse to the folder on the NMR computer which contains your data. There should be three files in the folder, named “fid”, “procpar” and “text”.

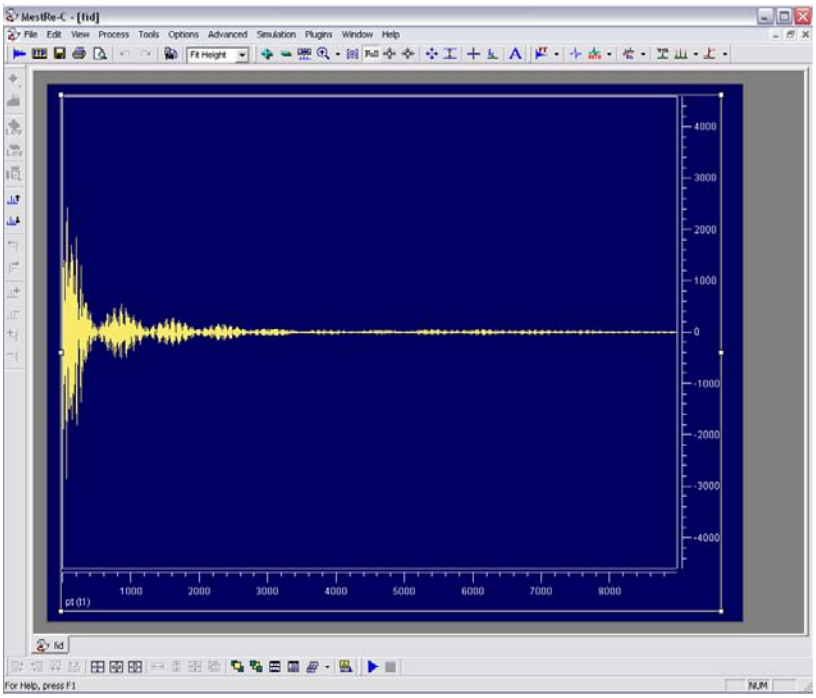
4. Under the “Transfer” tab , select “Transfer type > binary”. Next, highlight and drag the three files (fid, procpar and text) to the selected location on the left hand side of the Filezilla screen. Once the files are successfully transferred (i.e. they are no longer listed in the “Queued files” area at the bottom), you may exit Filezilla.

Using MestRe-C to generate data for fitting

1. Open MestRe-C. The basic screen should look like

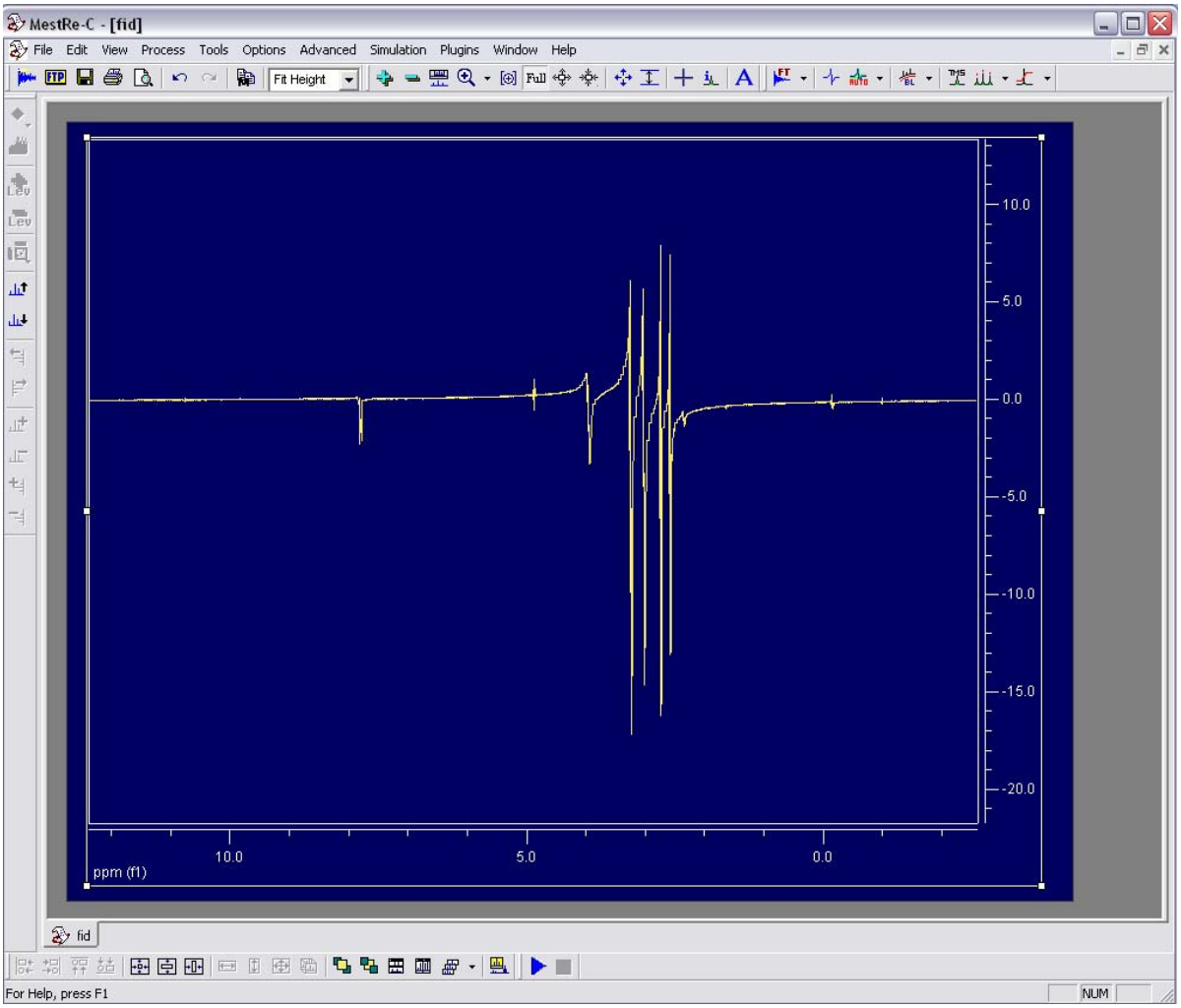


2. Click “File > Open…” and browse to the location where you put the fid, procpar and text files. If necessary, set the filetype to “All files”. Select the “fid” file and click “Open”. A non Fourier transformed plot should appear, which should look something like

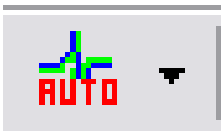


3. To perform a Fourier transform, click the

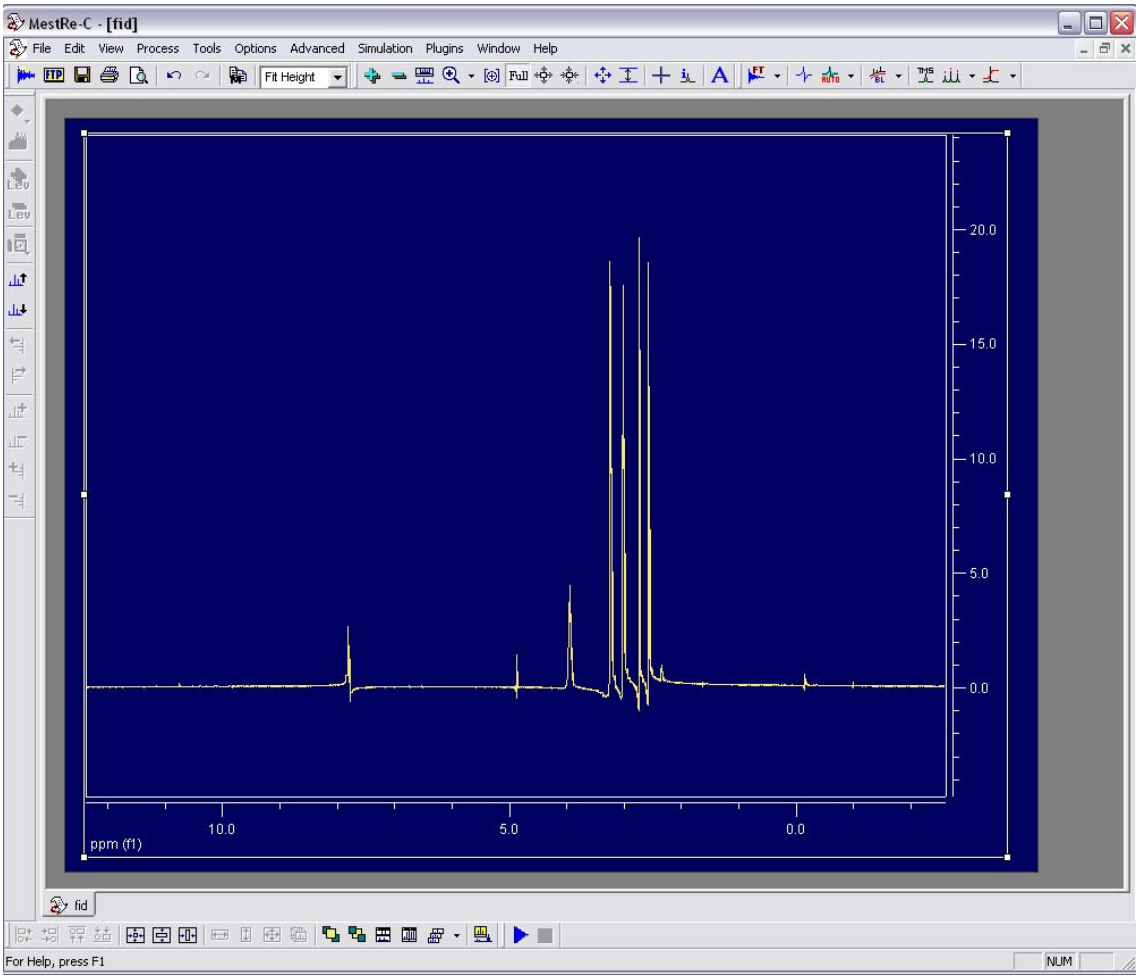
button and click on the “Apply along t1” button in the window which pops up. The result should look something like



4. Next you will need to phase the spectrum. Clicking on the autophase button

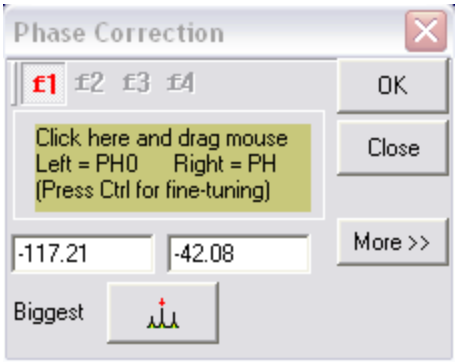


should result in a spectrum which looks more or less recognizable as an NMR spectrum.

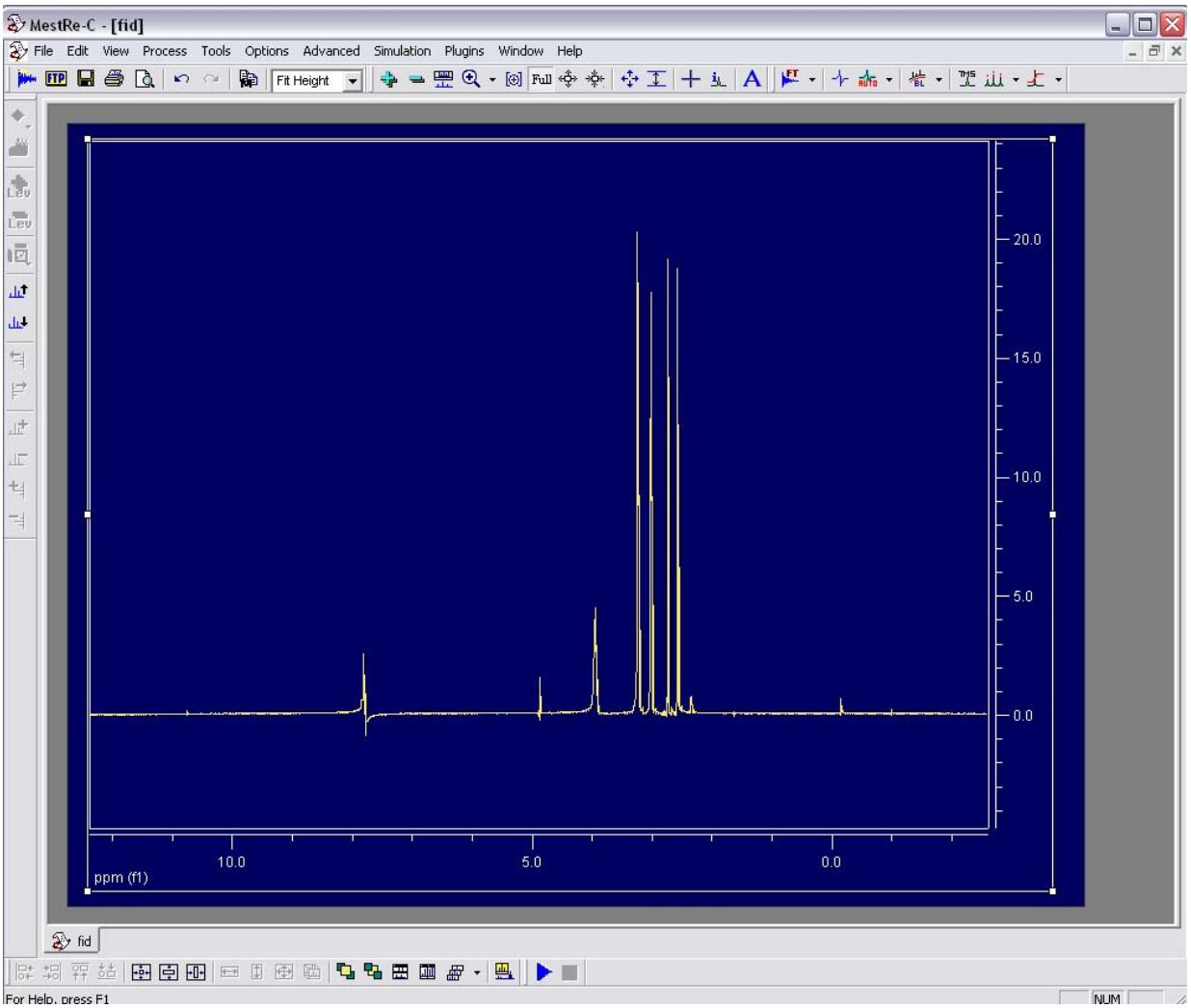


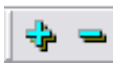
5. Chances are that the autophase routine will not generate a good baseline (as it didn't in the figure above). If this is the case, you may use the manual phasing option by clicking on the

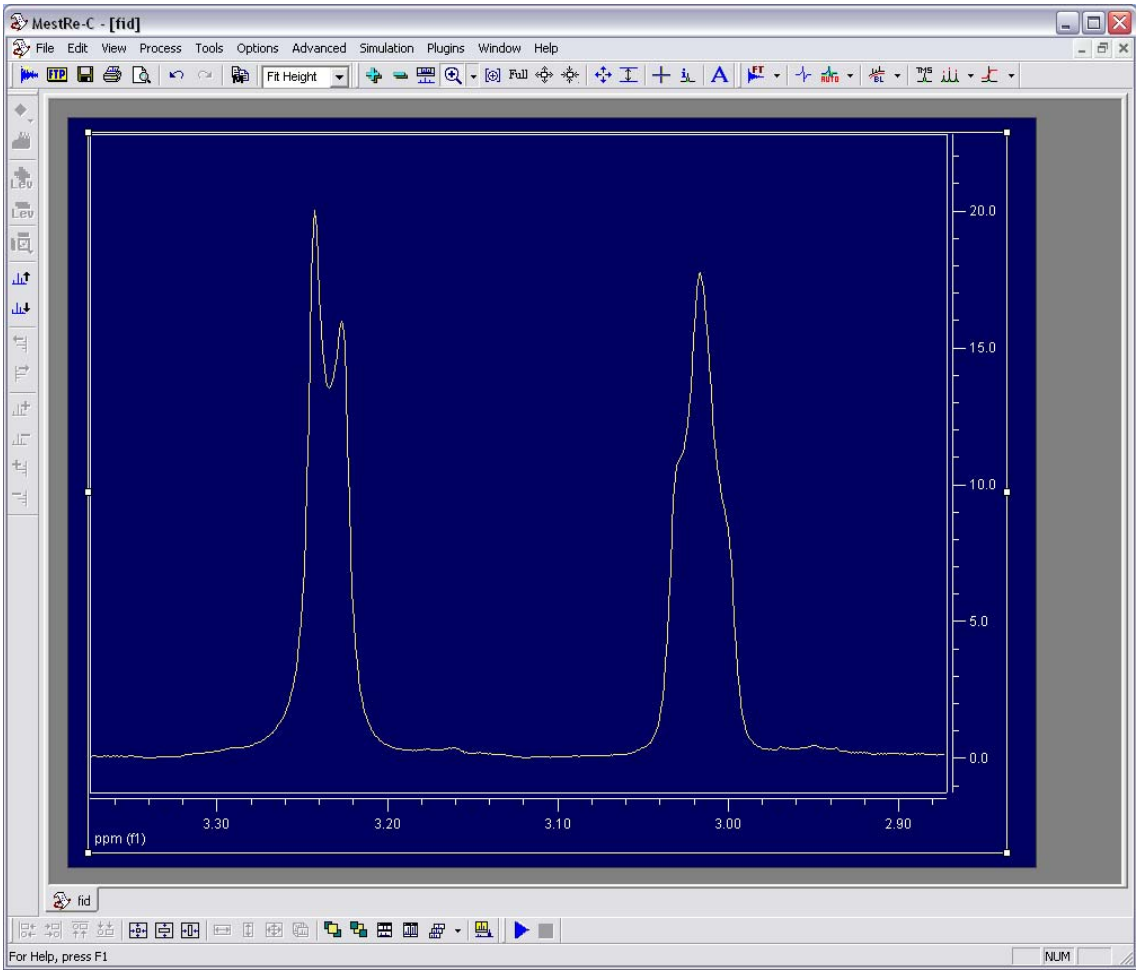
button. This brings up the interactive phase correction box:



If you click (and hold) the left mouse button and move it up and down in the yellow space, the baseline will change; holding down the ctrl key will allow you to make fine adjustments. Adjust the phase (as needed) until you have an acceptable baseline.



6. You can now zoom in on the portion of the spectrum you are interested in; when you save your data later, only the portion of the spectrum being displayed will be written to the data file. First, you may want to move the spectrum up/down to occupy the window as much as possible. If you move the cursor over the baseline, you can simply click (and hold) to drag the spectrum up or down. The zoom icons  (or the wheel of a wheel mouse) will allow you to zoom in/out. The magnifying glass icon  will allow you to select the portion of the x axis which you would like to enlarge. Suppose you want to look at the two peaks slightly above 3 ppm in the spectrum above. Changing the baseline and zooming in might yield the spectrum below:

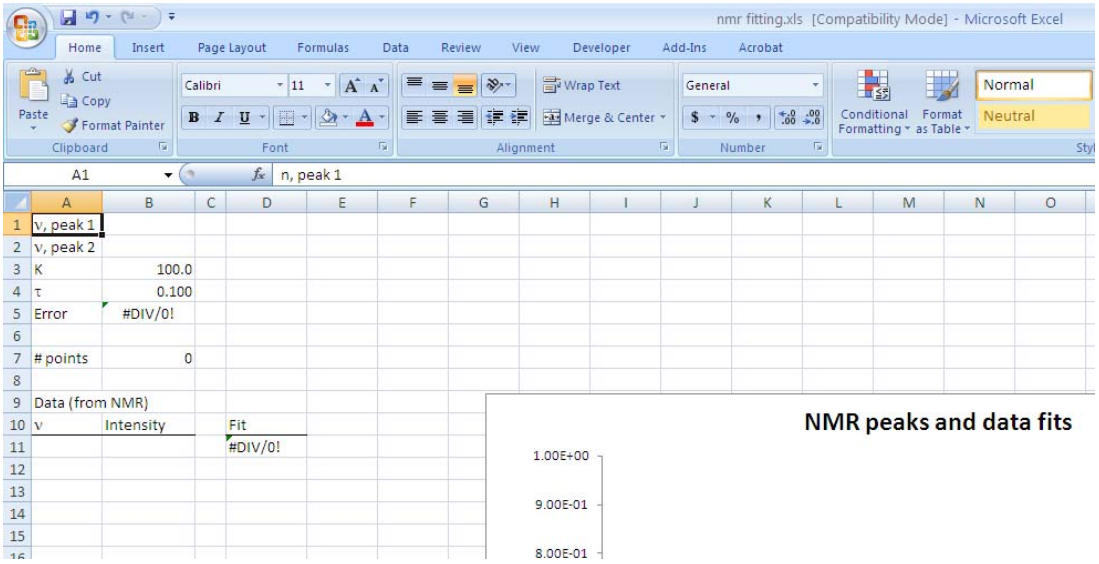


7. If the spectrum above is sufficient for fitting, you can now export the data; if it is not, you may modify it as needed. To export the data into a form usable by the data fitting spreadsheet in Excel, click “File > Export file > ASCII”. In the pop up window, select “Export only real part”, “Export only current displayed data”, “Include header” and “Include units”. Also select “Hz” in the “Include units” pulldown menu (since the analysis requires frequencies, not shifts in ppm).

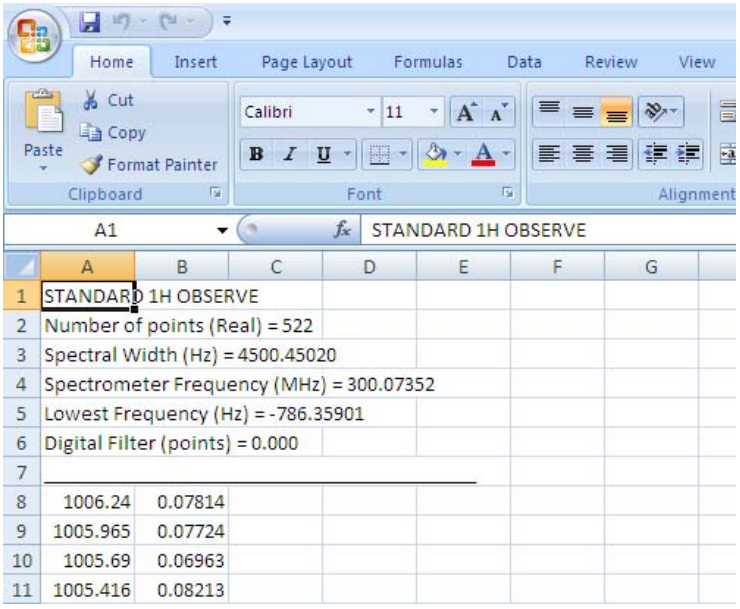
Click OK, and select a name and location for the data file. At this point, you are done with MestRe-C; you may want to wait to close it until you are certain that your spectrum is adequate. When you close MestRe-C, it is recommended that you do not save the changes to the FID file.

Opening the data in Excel and fitting the peaks

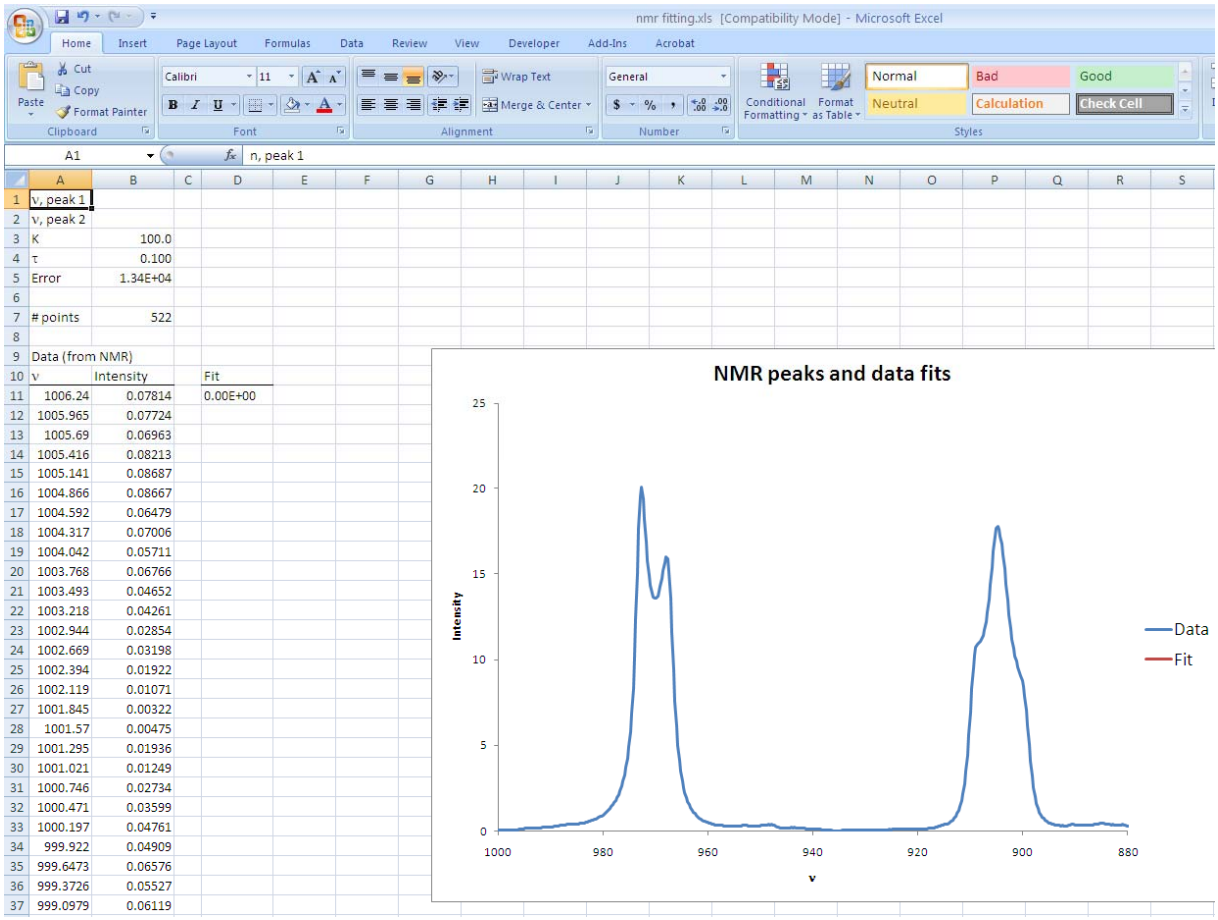
1. Open the Excel sheet named “nmr fitting.xls”; the file should be compatible with older versions of Excel.



2. Open the ASCII file you saved in MestRe-C. In the first screen of the text import wizard, select “Delimited” and click “Next”. In the next screen select “Tab” and click “Finish”. You should now have two columns of data in Excel



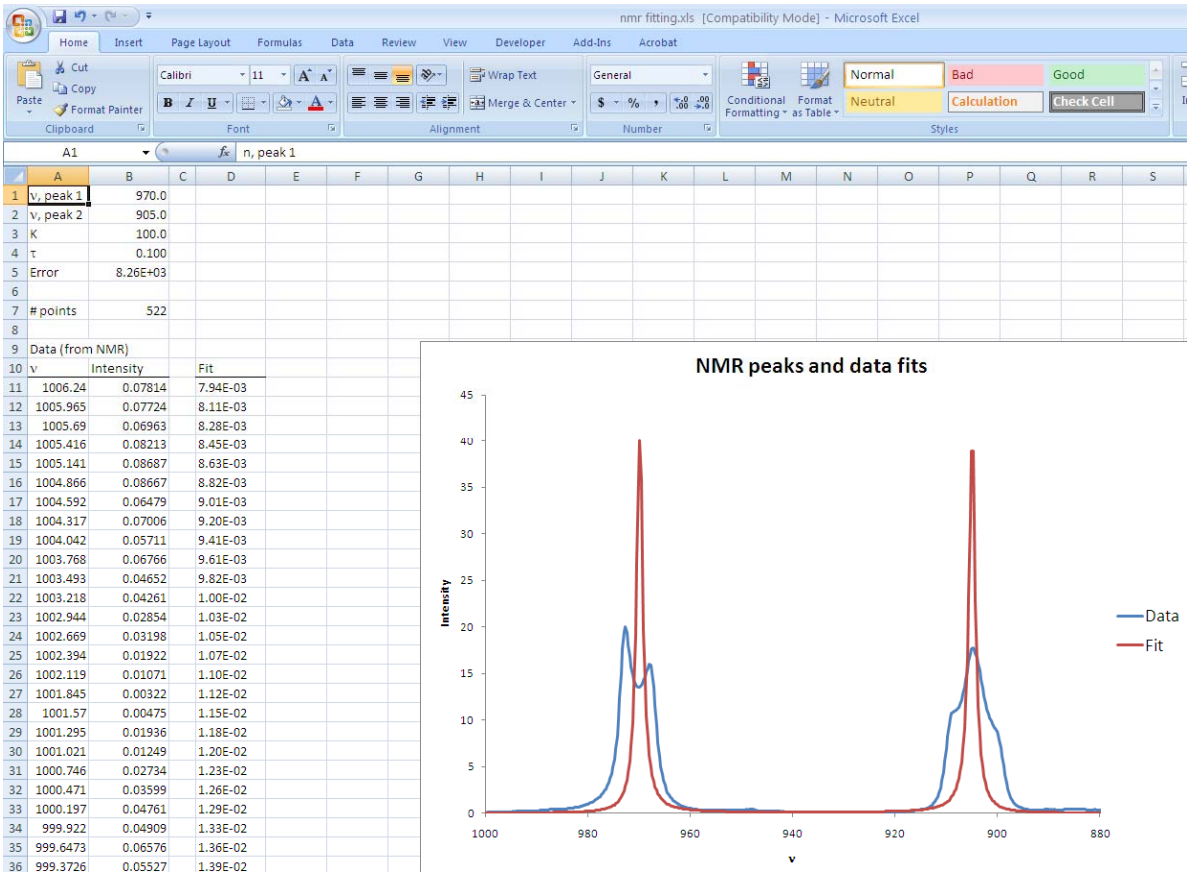
3. The first column (A) contains the frequency data, and the second column (B) contains the intensity data; copy only the data (starting in cells A8 and B8 in the example above) and past it into “nmr fitting.xls” starting in cell A11. If necessary, adjust the limits of the x axis of the plot in “nmr fitting.xls” so that you can see both peaks.



	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	S
1	ν, peak 1																		
2	ν, peak 2																		
3	K	100.0																	
4	τ	0.100																	
5	Error	1.34E+04																	
6																			
7	# points	522																	
8																			
9	Data (from NMR)																		
10	ν	Intensity		Fit															
11	1006.24	0.07814		0.00E+00															
12	1005.965	0.07724																	
13	1005.69	0.06963																	
14	1005.416	0.08213																	
15	1005.141	0.08687																	
16	1004.866	0.08667																	
17	1004.592	0.06479																	
18	1004.317	0.07006																	
19	1004.042	0.05711																	
20	1003.768	0.06766																	
21	1003.493	0.04652																	
22	1003.218	0.04261																	
23	1002.944	0.02854																	
24	1002.669	0.03198																	
25	1002.394	0.01922																	
26	1002.119	0.01071																	
27	1001.845	0.00322																	
28	1001.57	0.00475																	
29	1001.295	0.01936																	
30	1001.021	0.01249																	
31	1000.746	0.02734																	
32	1000.471	0.03599																	
33	1000.197	0.04761																	
34	999.922	0.04909																	
35	999.6473	0.06576																	
36	999.3726	0.05527																	
37	999.0979	0.06119																	

4. In “nmr fitting.xls”, copy and paste cell D11 down to the last row containing data in columns A and B. For example, if your last data points occur in cells A500 and B500, copy cell D11 and paste it into cells D12 through D500.

5. Enter the approximate frequencies of the two peaks in the plot into cells B1 and B2. In the example above, good guesses might be 900 s^{-1} and 970 s^{-1}. When you do this, a second line (red/brown) should appear on the graph. This is a plot of the fitting function; keep in mind that it has not yet been fit properly though, so it might not look very good.



6. Ensure that the Solver Add-In function is installed. If it is not, search "solver" in the help function in Excel and follow the installation steps.

7. Highlight cell B5 (to the right of "Error"). Open the solver dialog box and select the following:

Set target cell: $B$5 (can be set by clicking the box with the red arrow and highlighting the cell)
Equal to: Min
By changing cells: $B$1:$B$4

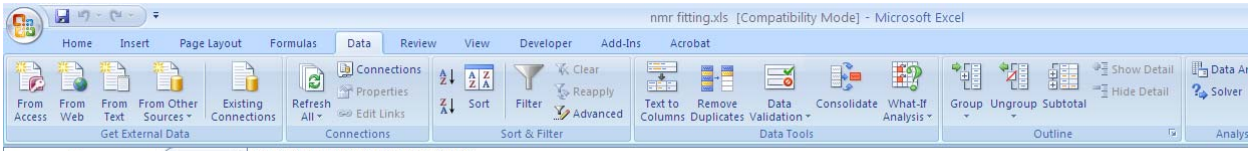
8. Click the "Options" button and make the following changes (as necessary):

Iterations: 1000
Derivatives: Central
Search: Conjugate

Then click "OK"

9. Click "Solve"; it may take a little while, depending on the size of your data set. When it is done, the "Solver Results" box should open. If the fit is reasonable (look at the graph), select "Keep solver solution" and click "OK". If the fit does not look good, you may select "Restore original values" and try again (but this is unlikely).

10. The best-fit values of ν_A, ν_B, K and τ will now appear in cells B1 through B4, respectively.



B5 {=SUM((B11:B11672-D11:D11672)^2)}

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	S
1	ν, peak 1	971.5																	
2	ν, peak 2	904.3																	
3	K	182.4																	
4	τ	0.025																	
5	Error	8.82E+02																	
6																			
7	# points	522																	
8																			
9	Data (from NMR)																		
10	ν	Intensity		Fit															
11	1006.24	0.07814		6.66E-02															
12	1005.965	0.07724		6.80E-02															
13	1005.69	0.06963		6.94E-02															
14	1005.416	0.08213		7.09E-02															
15	1005.141	0.08687		7.25E-02															
16	1004.866	0.08667		7.41E-02															
17	1004.592	0.06479		7.57E-02															
18	1004.317	0.07006		7.74E-02															
19	1004.042	0.05711		7.91E-02															
20	1003.768	0.06766		8.09E-02															
21	1003.493	0.04652		8.27E-02															
22	1003.218	0.04261		8.46E-02															
23	1002.944	0.02854		8.65E-02															
24	1002.669	0.03198		8.85E-02															
25	1002.394	0.01922		9.06E-02															
26	1002.119	0.01071		9.27E-02															
27	1001.845	0.00322		9.49E-02															
28	1001.57	0.00475		9.72E-02															
29	1001.295	0.01936		9.95E-02															
30	1001.021	0.01249		1.02E-01															
31	1000.746	0.02734		1.04E-01															
32	1000.471	0.03599		1.07E-01															
33	1000.197	0.04761		1.10E-01															
34	999.922	0.04909		1.12E-01															
35	999.6473	0.06576		1.15E-01															
36	999.3726	0.05527		1.18E-01