Pulse Programs/ Command ased 8

The Pulse Programs “zg” and “zg30” 8.1

As mentioned in the section on acquisition parameters (***"Basic Acquisition Parameters: The “eda” Table" on page 72***), pulse programs are used to define the pulse sequence with which the sample is to be excited. A newly delivered AVANCE with SGU spectrometer will already have a “library” of standard pulse programs installed. The list of pulse programs may be viewed by entering the command ’edpul’. Scroll through the list until the program “zg30” is found. Click on “zg30” and the program, reproduced in ***Figure 8.1.***, will be displayed.

A detailed description of pulse programming is beyond the scope of this manual, although a few comments on the program entitled “zg30” should help the user understand which parameters are important and why. A standard 1-D experiment would use a 90 degree pulse to excite the NMR signal to maximize the emitted signal. However, this also maximizes the duration that must elapse between successive pulses in order for the sample to relax. It can be shown that in the case of repeated excitation of a sample, a more efficient method is to excite the sample with a 30 degree pulse and reduce the relaxation delay accordingly. Although each individual emitted signal is weaker, the faster accumulation of data that the 30 degree pulse allows results in an overall increase in sensitivity. The two standard programs are "zg“ for a 90 degree excitation pulse and "zg30” for a 30 degree excitation pulse.

For more information on the conventional nomenclature used for power levels, pulses, delays and loop counters throughout the Bruker series of pulse programs, refer to the text file **$TopspinHome/exp/stan/nmr/lists/pp/Param.info**. For information on the conventional nomenclature used in the names of Bruker pulse programs refer to **$TopspinHome/exp/stan/nmr/lists/pp/Pulprog.info**. Finally, a useful file for checking changes in the latest software release is **$TopspinHome/exp/stan/nmr/lists/pp/update.info**.

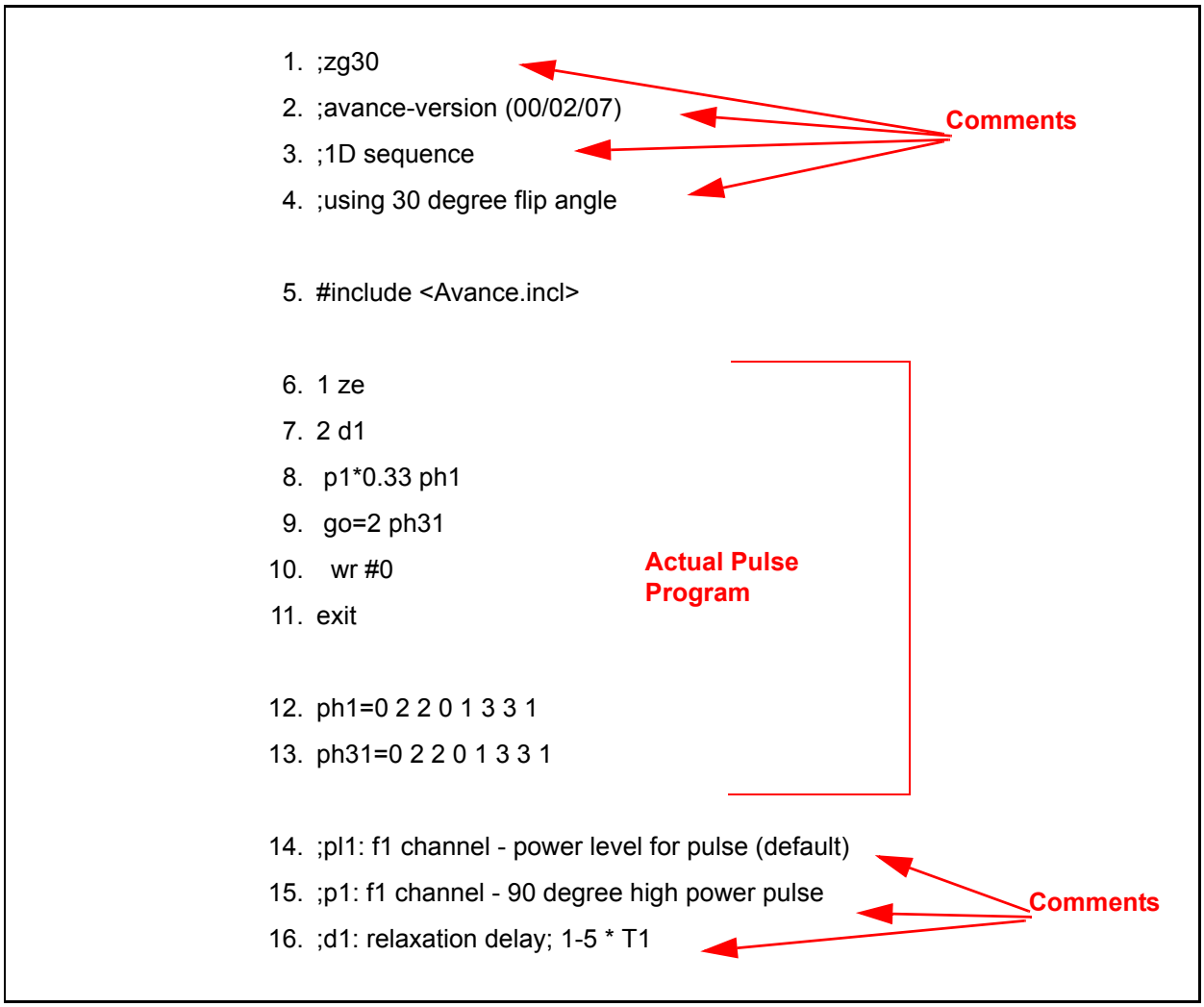
$TopspinHome stands for the name of the directory in which the Topspin program was installed (C:\Bruker\Topspin under Windows and /opt/Topspin under Linux).

Details of the “zg30” Program 8.2

Any entries in a pulse program that are preceded by a semicolon are simply comments which are included to assist the user. The program compiler will ignore the contents of any line that begins with a semicolon. For the purposes of this description each line of the “zg30” program has been assigned a number which however will not appear on the Topspin screen whenever the pulse program is displayed.

The first four lines are examples of comments. Line 1 simply gives the title of the pulse program and line 2 the version. Line 3 suggests that this pulse program is suitable for a 1-D NMR experiment and line 4 reminds the user that instead of a 90 degree excitation pulse the program is designed to use a 30 degree pulse (and hence the name of the program).

Figure 8.1. The Pulse Program “zg30 “



Line 5 is not a comment, but rather a standard inclusion dealing with pulse programming text defined in separate files.

Line 6: 1 ze - This line is not preceded by a semicolon and is actually the first line of the program proper. Any line within a program can be numbered to facilitate loops and this one is naturally numbered as line 1. The command "ze" (zero) is a command that causes any data currently in memory to be replaced by data acquired during the first scan. Data acquired during subsequent scans will then be added in the memory. The command "ze" effectively clears, or zeroes, the memory in preparation for data that will be acquired during an experiment.

Line 7: 2 d1 - This line is a delay command. The delay is called "d1“. A delay is simply a pause during which no pulses are transmitted. The length of d1 can be specified by entering a suitable value in the delay table of the "eda“ menu.

Line 8: p1*0.33 ph1 - The command p1*0.33 instructs the spectrometer to emit a pulse with length p1 multiplied by 0.33. This is the 30 degree excitation pulse, since conventionally p1 is the 90 degree pulse. Any pulse will have three principle characteristics: 1) length, 2) amplitude, and 3) phase.

The **length** or duration of the pulse may be set by entering a suitable value of p1 in the pulse table of the eda menu.

The **amplitude** of the pulse is often referred to as its power level. In this case, no power setting is specified, so the spectrometer defaults to the value assigned to pl1. The value of “pl1” may be set by entering a suitable value in the power level table of the “eda” menu.

It should also be mentioned, that when no channel is specified for a particular pulse, the pulse is automatically sent to the observe channel (i.e. channel F1). Line 8 also assigns the **phase** to the pulse with the command “ph1”.

Line 9: go=2 ph31 - This single command starts a train of processes including the opening of the receiver and digitalization of NMR signals. When the acquisition has been completed, i.e. after TD points have been digitized, the program loops back to the line beginning with "2" which is Line 7. The entire process is repeated NS times (assuming that DS=0). If NS=8 then lines 7, 8, and 9 will be looped through 8 times. Line 9 also sets the phase of the receiver with the command ph31.

The need for the delay “d1” in Line 7 should now be more apparent. Exciting an NMR sample with a series of pulses and insufficient relaxation delay causes it to become saturated. In other words it absorbs more energy than it can release. To give the sample time to release more energy, the delay “d1” precedes each pulse. This is referred to as allowing the sample sufficient time to relax.

Line 10: wr #0 - This command instructs the computer to store the acquired data on the hard disk. The data is stored in the current data set as defined by the parameters NAME, PROCNO, EXPNO, etc.

Line 11: Exit - This command tells the computer that the end of the pulse program has been reached.

Line 12 and 13: These two lines define the phase cycling sequence for both the transmitter (ph1)and receiver (ph31).

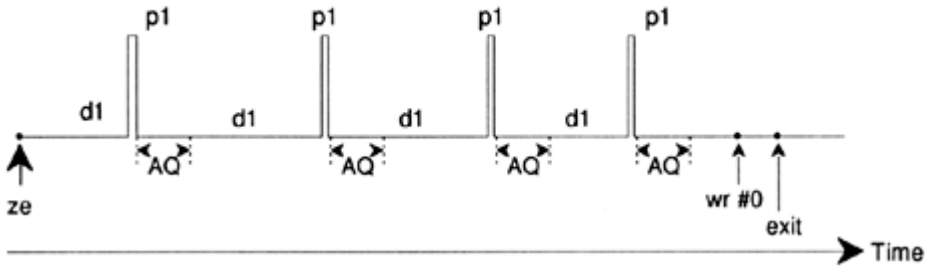
The above description has been included to give the reader some idea of the parameters that must be set when running the pulse program “zg30”, namely “d1”, “p1”, and “pl1”. Note that the last three lines of the pulse program are comments used to explain the significance of these three parameters to the user. The precise value assigned to “p1”, “d1”, and “pl1” will depend on the sample to be analyzed and the type of probe to be used.

The common pulse program entitled “zg” is identical to “zg30” except that a 90 degree pulse is transmitted rather than a 30 degree pulse.

Figure 8.1. is a pictorial representation of the pulse program “zg”, with NS=4 and DS = 0.

At this point, the reader should have gained a basic understanding of the main elements involved in acquisition. The rest of this manual will be concerned with step by step accounts of how to actually acquire spectra.

Figure 8.2. The Pulse Program “zg” where NS=4 and DS=0.



The Command “ased” *8.3*

Now that you are more familiar with the acquisition software, and some basic pulse programs, it might be opportune to introduce a further command: “ased”. The command may be used to set the acquisition parameters for a particular pulse program, e.g. pulses, delays, etc.

The command refers to the current pulse program i.e. the program entered for PULPROG in the “eda” table. Having ensured that “zg30” is the current pulse program, choose the ‚AcquPars‘ tab of the data window, and press the button in the toolbar or the question mark next to the puls program line in the ‚AcquPars‘ table or enter .’ased’.

The display changes to an “eda” type table. Note, however, that the “eda” table presents the complete delay and pulse arrays, and that the “ased” table simply contains “p1” and “d1”. This is because the pulse program “zg30” uses no other pulses or delays. The parameters in the “ased” table may now be modified to suit the experiment. Any changes will be carried over into the “eda” table. Click on SAVE to return to the ‚AcquPars‘ table. Thus, “ased” is a useful command in that it concentrates on all relevant parameters for a specific experiment.

Finally, do not forget that any individual parameters may be adjusted directly from the Topspin command line. This is particularly useful when a “fine tuning” of an experiment is required.