



Data Collection Step Size Selection

HOW TO DECIDE STEP SIZE, TIME PER STEP, AND COLLECTION TIME ON
PANALYTICAL X-PERT PRO POWDER

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Introduction

In this document we will discuss how a user decides on step size, time per step, and total collection time for a powder x-ray diffraction (p-xrd, pxrd) data collection. This write-up is specifically for Panalytical X-Pert Pro powder machine. The information given here is appropriate for Panalytical Data Collector software. However, the discussions are applicable to other pxrd data collections.

Procedure

The general procedure will be divided in the following steps:

- We will first collect a *Quick Run* pattern
- We will determine the Full Width at Half Maximum (FWHM) for a prominent peak
- We will then decide on the appropriate step size
- We will then adjust the time per step to give us the total desired data collection time
- Finally, we will collect the data again to confirm our strategy

❖ QUICK RUN

The first step to determine the data collection strategy is to do a *Quick Run*. However, if your sample is radiation sensitive or if you already know some details about your sample, you may be able to skip the *Quick Run* step.

During the *Quick Run*, the user typically collects the data about 5 to 10 minutes for a 2-theta range of 10 to 60° using the **Panalytical Data Collector** software. Once the *Quick Run* is completed, the user selects a prominent peak for full width at half maximum analysis. The selection of peak and its shape is important and it is preferable that the peak is symmetric and without any sub-peaks or shoulder.

❖ FULL WIDTH AT HALF MAXIMA (FWHM)

Open the *Quick Run* pattern in **Panalytical Data Viewer** software and you will see a pattern similar to the one shown in Figure 1 below:

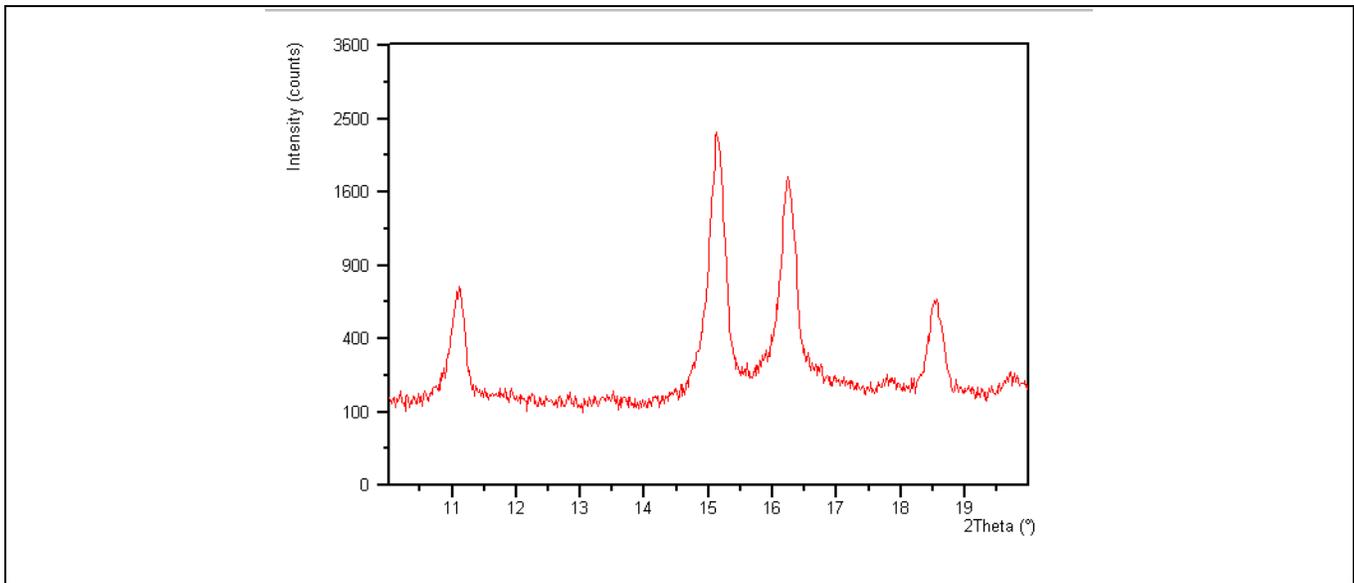


Figure 1 Quick Run Pattern as seen in Data Viewer

Now, bring the mouse pointer near the selected peak, in this case the one centered around 11° two-theta. The mouse pointer will change from cross-hair (\oplus) to an arrow (\blacktriangleright). Now right click the mouse and select “Peak Parameters”. This will draw a background line, peak position, and Full width at half maximum line and will display in text the Peak Parameters including: Position, Full Width at Half Maximum (both in degrees), and Net height and Background (both in counts or counts/sec) as shown in Figure 2. If there is a need, the background values on the left and right (black squares; ■) can be changed to produce a more symmetric peak. Once a satisfactory profile is created make a note of the FWHM, in our example it is 0.173°.

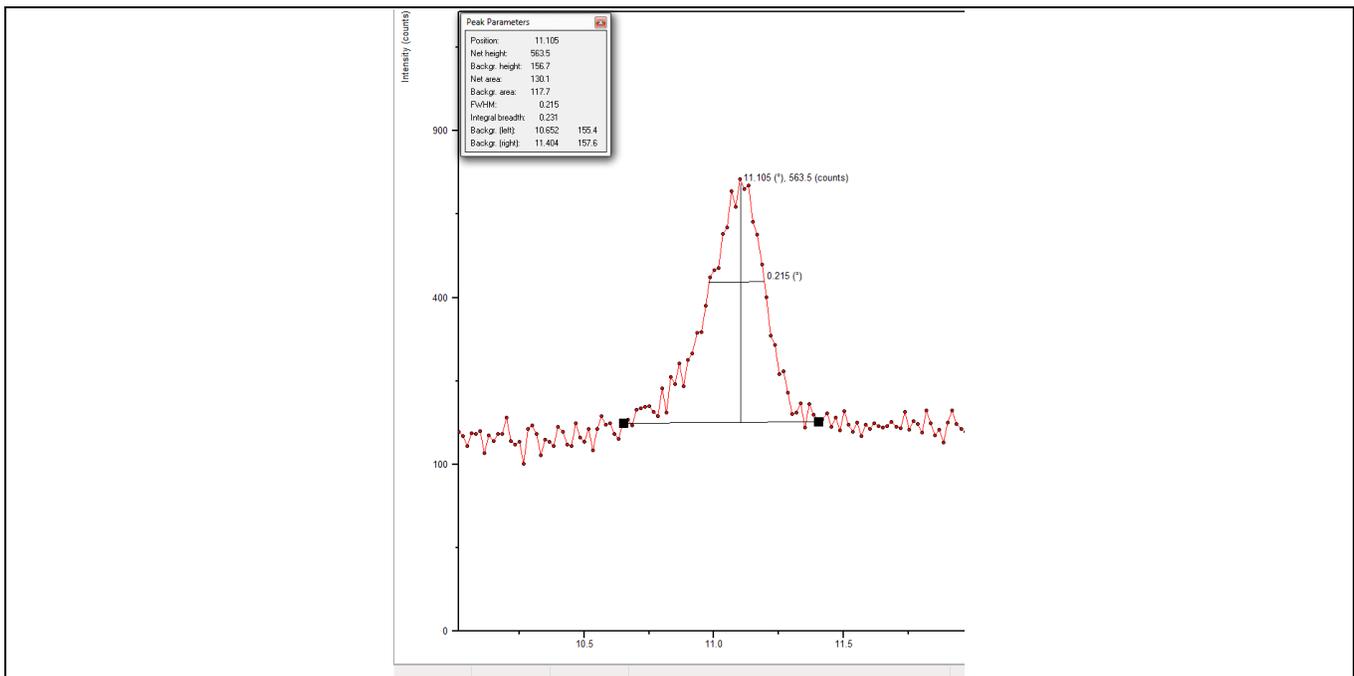


Figure 2 Peak Parameters in Data Viewer

❖ STEP SIZE

Now that we found out the FWHM for our sample, the next step is deciding on the Step Size, i.e., at what intervals of 2-theta the experimental data will be acquired. The FWHM for our test sample is 0.173° . So it is essential we make the Step Size at least $1/10 - 1/12^{\text{th}}$ the value of the FWHM. This way we will have 10-12 points between FWHM and 30-36 data points between two background levels. Please note that FWHM is slightly larger than two sigma, the standard deviation for Gaussian peak (σ ; http://ned.ipac.caltech.edu/level5/Leo/Stats2_3.html) and with 10-12 points between FWHM it is likely the peak position will be determined accurately. So in this example the Step Size will be 0.016° , i.e., $1/10^{\text{th}}$ the value of FWHM (In Panalytical X-Pert Pro w/ X'Celerator Detector the step sizes are best chosen in multiples of eights).

❖ 2-THETA RANGE

Next step is deciding the 2-theta ranged. Based on the *Quick Run* and some knowledge of the material at hand, the user needs to decide on the 2-theta range for data collection. Typically, they range between $5-100^\circ$. Of course, individual samples may either have shorter or longer range. If the sample is a "known sample", powder diffraction files (PDFs) can be consulted before deciding the 2-theta range.

❖ NUMBER OF STEPS

Once the 2-theta range is determined, it is easy to find out how many steps are required for the scan range since we already determined the Step Size

$$\text{Number of Steps} = \text{Scan Range in 2-theta} / \text{Step Size}$$

For our example the data was collected between 10 and 20° in 2-theta ($20-10=10^\circ$ Scan Range). So for this Scan Range (10°) and Step Size (0.016°) the Number of steps will be

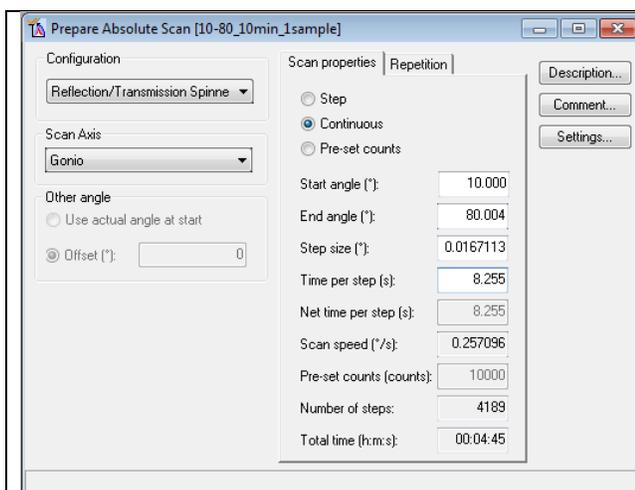
$$10^\circ / 0.016^\circ = 625$$

So the number of steps will be 625.

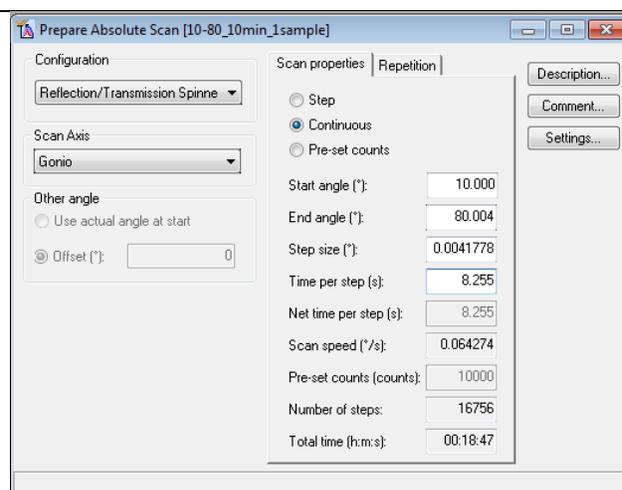
❖ TIME PER STEP AND TOTAL DATA COLLECTION TIME

Now that we determined the Step Size, 2-theta Scan Range, and Number of Steps, we need to decide on the Time per Step. Of course deciding on Time Per Step is linked to the Total Data Collection Time.

So let us look at an example where the Step Size was determined to be 0.01671° , the range was from 10 to 80° in 2-theta (70° total), and Number of Steps were 4189. The Prepare Absolute Scan window in Program Menu has some specific input requirements. You can play with them to see the Total Collection Time. For this example, inputting 8.255 seconds for Time Per Step yields a Total Data Collection Time of $\sim 4 \frac{3}{4}$ minutes.

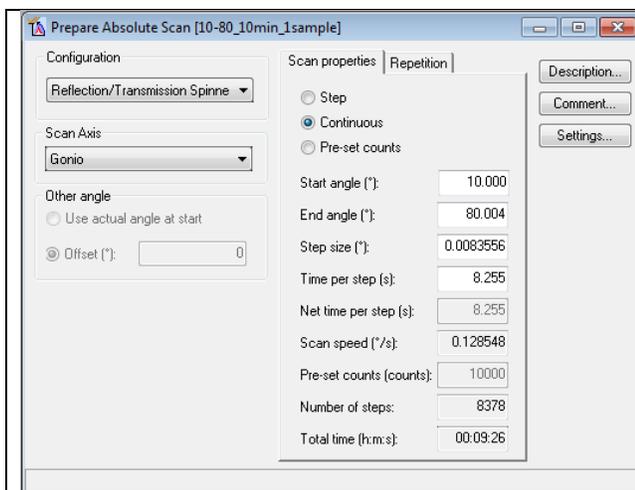


Step Size 0.01671° , range 70° , Time/Step 8.3 s for a Total Data Collection Time of $4\frac{3}{4}$ minutes

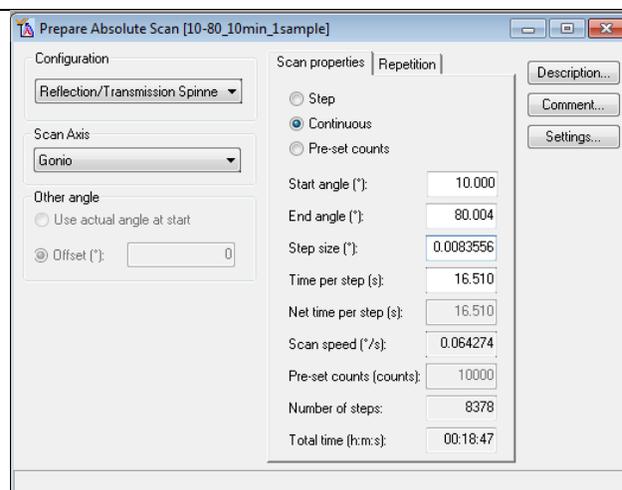


Step Size 0.00417° , range 70° , Time/Step 8.25 s for a Total Data Collection Time of $18\frac{3}{4}$ minutes

So let us look at another example where the Step Size was determined to be 0.008355° , the range was from 10 to 80° in 2 -theta (70° total), and Number of Steps were 8378. The Prepare Absolute Scan window in Program Menu has some specific input requirements. You can play with them to see the Total Collection Time. For this example, inputting 8.255 seconds for Time Per Step yields a Total Data Collection Time of $\sim 9\frac{1}{2}$ minutes. Inputting 16.510 seconds yields almost double the Total Data Collection Time of $\sim 18\frac{3}{4}$ minutes.



Step Size 0.008° , range 70° , Time/Step 8.3 s for a Total Data Collection Time of $9\frac{1}{2}$ minutes



Step Size 0.016° , range 70° , Time/Step 16.5 s for a Total Data Collection Time of $18\frac{3}{4}$ minutes

As seen in the input dialog lower the Time/Step means faster scan rate of $0.128^\circ/s$ and higher Time/Step means lower scan rate of $0.0642^\circ/s$.

Final confirmation is achieved when the user runs the full range of data and here it is shown for NIST powder Si standard sample for 10 - 80° 2 -theta and for a 10 minute run with Ni-filter.

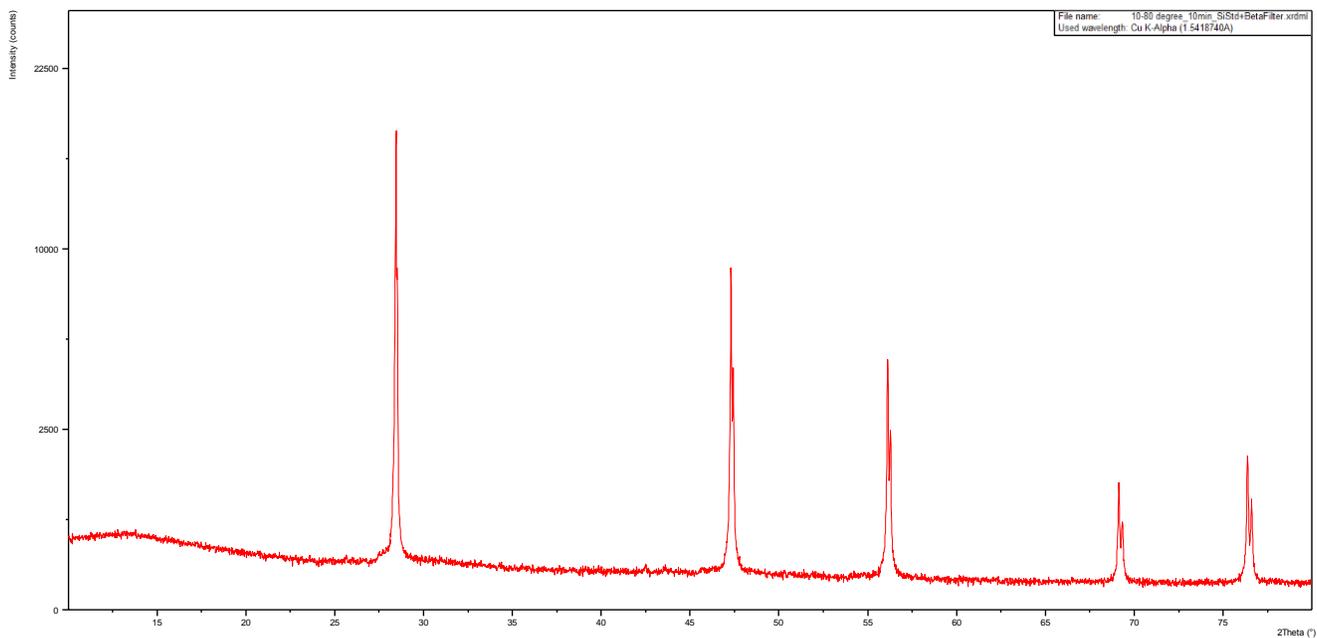


Figure 3 NIST powder Si standard sample for 10-80° 2-theta and for a 10 minute run with Ni-filter

Conclusion

Based on the discussions above it is hoped that regular users of XRD will be able to decide on the Step Size, 2-Theta Range, Number of Steps, and Total Collection Time.

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