PELCO X-Checker®

Product No. 602-01, -02, -03, -20 and -21

DESCRIPTION:

Congratulations on your purchase of the PELCO X-Checker®. When used regularly, the PELCO X-Checker® allows you to monitor the performance of your EDS/SEM system and helps find potential problems before they have a chance to affect your analysis. The PELCO X-Checker® provides multiple tests, which you can perform periodically to verify your system performance. The tests available vary depending on your particular model of the PELCO X-Checker®. There are four models:

#602-01 PELCO X-Checker®

#602-02 PELCO X-Checker® B

#602-03 PELCO X-Checker® Extra

#602-20, 602-21 PELCO X-Checker® Wafer

Each of the 602-01, 602-02 and 602-03 are mounted on a 9.5mm diameter, 2mm thick disc. These can then be ordered mounted on any of our standard SEM mounts. The following four sections show the layout of the elements on the four models.

PELCO X-Checker® (#602-01):

The following tests can be performed with the PELCO X-Checker® (shown in ***Figure 1***):

1. Spectrum calibration with aluminum, copper and nickel.
2. Low energy spectrum calibration with carbon and aluminum (for windowless and ultra-thin window detectors).
3. Manganese (in Mn88/Ni12 alloy) full width at half max (FWHM) resolution.
4. Low energy sensitivity check.
5. Low magnification imaging calibration.
6. High magnification imaging calibration.

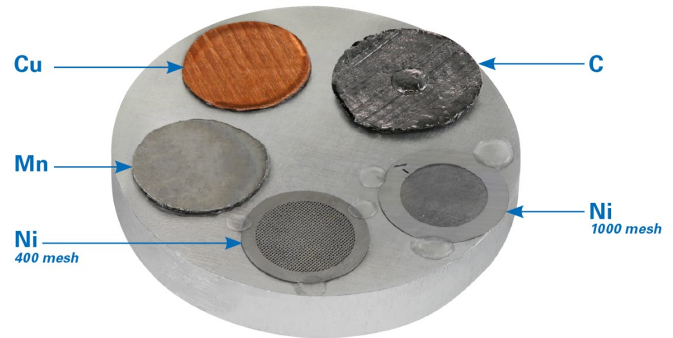

Figure 1*: #602-01 PELCO X-Checker™*

PELCO X-Checker® B (#602-02):

The PELCO X-Checker® B (***Figure 2***) adds boron nitride as a more sensitive monitor of low-end performance on thin window and windowless detectors.

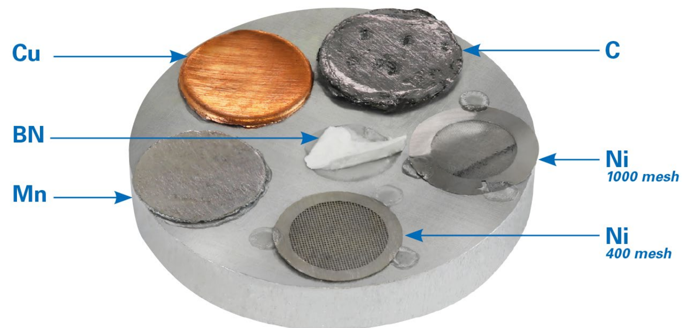

Figure 2*: #602-02 PELCO X-Checker™ B*

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PELCO X-Checker® Extra (#602-03):

The PELCO X-Checker® Extra (***Figure 3***) adds the following tests:

1. PTFE as a fluorine source to measure low-end resolution.
2. A beryllium half grid (replaces the nickel high mag grid) for verifying the low-end sensitivity of high performance detectors.

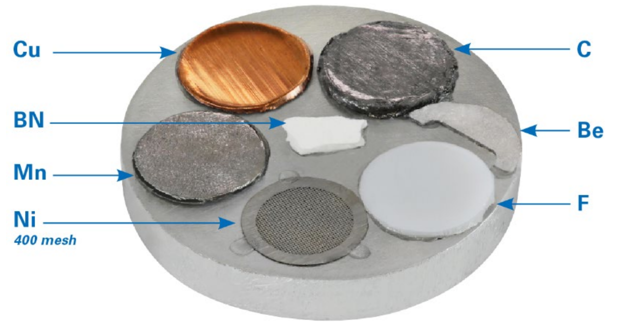

Figure 3*: #602-03 PELCO X-Checker™ Extra*

PELCO X-Checker® Wafer (#602-20, 602-21):

The PELCO X-Checker® Wafer (***Figure 4***) adds aluminum foil for low energy spectrum calibration as well as stainless steel (SS - 304 Stainless) for analysis:

Cr	18.13%	Ni	8.26%
S	0.01%	Mn	1.78%
Si	0.46%	C	0.02%
P	0.03%	Fe	71.31%

The #602-20 model comes mounted on an 8” (200mm) wafer, whereas the #602-21 is mounted on a 12” (300mm) wafer.

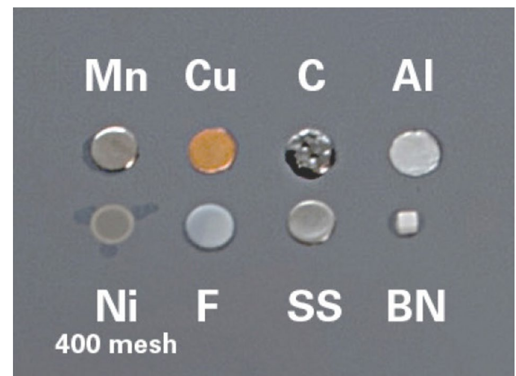

Figure 4*: #602-20 PELCO X-Checker™ Wafer*

SPECTRUM CALIBRATION

Most EDS systems can be calibrated on the aluminum K-alpha and copper K-alpha peaks (1.49 keV and 8.04 keV respectively). To perform a spectrum calibration using a PELCO X-Checker®, set the SEM to its lowest magnification and orient the copper under the beam so it fills ¾ of the screen and the remainder with the aluminum. The accelerating voltage should be at 20 kV and the scan speed at TV or rapid scan. Adjust the condenser lens (spot size) and final aperture to achieve a count rate of about 1000 X-ray counts per second*. On the spectrometer, adjust the ratio of aluminum and copper on the SEM screen, if necessary, by moving the stage or changing the magnification so the aluminum and copper peaks are about the same height.

For the PELCO X-Checker® Wafer, the aluminum is a separate standard but the silicon K-alpha peak (1.74 keV) can be used for lower energy calibration. Alternatively, the copper L-alpha and K-alpha lines (0.93 and 8.04 keV) should provide a good full-scale calibration for general use. This calibration should be performed at 10 kV. Once you have obtained a good spectrum with this procedure, follow the calibration instructions for your EDS system as outlined by the manufacturer.

**count rate for a pure element*

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LOW ENERGY SPECTRUM CALIBRATION

Windowless and ultra-thin window detectors may require special calibration at the low end of the spectrum to ensure proper calibration when analyzing low energy X-rays. For these systems, calibration can be performed on the carbon K-alpha and aluminum K-alpha peaks (0.28 and 1.49 keV respectively). Orient the PELCO X-Checker® so that carbon is visible on the SEM and fills about ¾ of the screen. Set the accelerating voltage to 5 kV. You may have to vary the proportion of carbon and aluminum in the field of view until the two peaks are about the same size. Adjust the condenser lens (spot size) and final aperture to achieve a count rate of about 1000 counts per second*. Follow the same system calibration procedure you used for the copper and aluminum.

NOTE: On some systems, this low energy calibration (while giving optimum results for low energy peaks) may result in peak shifts at higher energy. If this is true with your system, and if your analysis requires both low and high energy peak identifications, you may want to perform the calibration using just copper and calibrating on the L-alpha and K-alpha lines (0.93 and 8.04 keV respectively) as described under "Spectrum Calibration" on the previous page. However, this may not be as accurate as the two previous methods. If you have the PELCO X-Checker® B, PELCO X-Checker® Extra, or PELCO X-Checker® Wafer, you have the option of using the boron nitride or even the beryllium (PELCO X-Checker® Extra only) peaks for low-end calibration.

NOTE: This may be difficult due to their low signal strength.

**count rate for a pure element*

RESOLUTION

The resolution of your EDS system should be checked periodically to ensure correct operation of your detector and electronics. This is easily accomplished using the manganese standard by measuring the full width at half max (FWHM) of the manganese K-alpha peak. If you have the PELCO X-Checker® Extra, you can also perform a resolution test on the fluorine K-alpha peak with the PTFE standard. The test described here is valid for either the manganese peak or the fluorine peak.

To measure FWHM, orient the manganese (or fluorine) standard under the beam and set the magnification to 100X. Set the accelerating voltage to 15 kV for manganese (or 5 kV for fluorine). Adjust the condenser lens (spot size) and final aperture to get a count rate of about 1000 counts per second*. If your EDS system has an adjustment to vary the signal processing time constant, select the setting with the longest time constant to provide the best resolution of the spectrometer. Acquire a spectrum until there are at least 2000 counts in the centroid (middle) channel of the manganese (or fluorine) peak.

Determine the vertical midpoint of the peak - this is the half max. Measure the width of the peak at half max - this is the full width at half max (see ***Figure 5***). Record this value in the "RESOLUTION DATE/ eV" table on page 16 of the PELCO X-Checker® manual. Resolution should be checked periodically and this table will provide a handy record of your detector performance over time. It is recommended that you check the resolution of your system every six months.

**count rate for a pure element*

Many factors can cause a degradation of resolution. Small variations in resolution (10 eV or less) can result from statistical error. A small one-time variation in resolution probably does not indicate a problem, but a steady degradation over time is a sure sign of a detector or system problem. If your measurements show an increase in FWHM over time, you should notify the EDS system manufacturer.

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NOTE: It is an accepted practice in the EDS industry to specify the resolution of X-ray detectors using radioactive Fe-55 source, which has a photon emission close to the same energy as the manganese K-alpha peak. Measurements taken with the PELCO X-Checker® may vary slightly from those made with an Fe-55 source due to background radiation (Bremsstrahlung) X-rays produced in the bulk sample. Remember that the PELCO X-Checker® is designed to detect changes in resolution over time and is not a substitute for an Fe-55 source. However, if the background is subtracted from the manganese peak before the FWHM measurement is made, the resolution should be very close to that measured with an Fe-55 source. The manganese standard on your PELCO X-Checker® contains about 12% nickel so it is normal to see small nickel peaks in your spectrum.

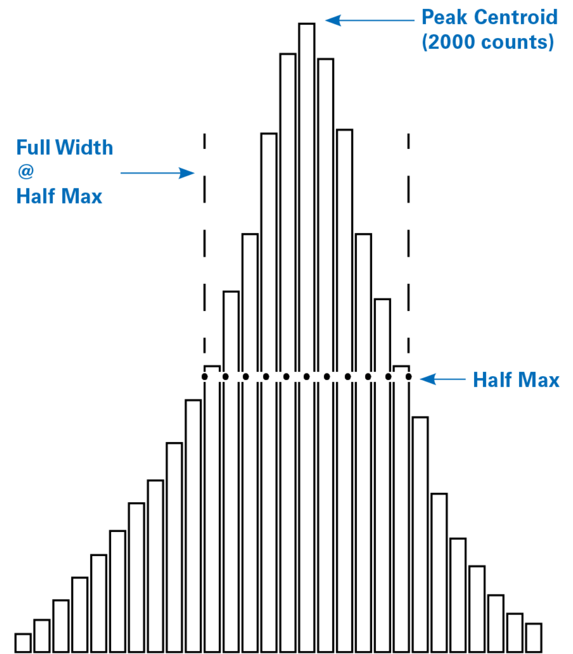

***Figure 5**: FWHM Peak Determination*

SENSITIVITY

Detector windows can become coated with vacuum pump oil or other contaminants that can attenuate the incoming X-ray signal. This is especially serious when analyzing low energy X-rays. Unless you monitor your system's sensitivity on a regular basis, you may not be aware that your window is dirty and you are losing some of your signal. There are several ways to monitor system sensitivity but the simplest is to perform a peak ratio measurement using a high and low energy peak.

The most common test is performed using the manganese standard. Since this standard contains about 12% nickel, you can ratio the manganese K-alpha to the nickel L-alpha peak. As the detector window gets dirty, the manganese K peak will appear larger relative to the nickel L-alpha peak because the nickel L X-rays will become increasingly absorbed by the contamination on the window. If you have a windowless or thin window detector, you may notice a small oxygen peak; this is normal.

Set the accelerating voltage to 10 kV and put the beam in the center of the manganese standard. Adjust the condenser lens (spot size) to achieve a count rate of about 1000 counts per second* and acquire a spectrum for 100 seconds. If you need to tilt the sample, make sure to record the tilt angle since this test must be performed at the same tilt angle each time. Using your system software, or by manually measuring the peak heights, calculate the peak ratio of the manganese K-alpha peak to the nickel L-alpha peak. Record this value in the "**SENSITIVITY Date / Mn-L : Ni-L Tilt**" table on page 17 of the PELCO X-Checker® manual.

**count rate for a pure element*

NOTE: For beryllium window detectors, the peak ratio will be about 2:1. For ultra-thin window detectors, the ratio will be closer to 1:1. It is critical that you determine the sensitivity of your detector and use that value as a benchmark for your system. The values quoted here are only examples and your ratio value may vary considerably. All detectors and windows are slightly different and sensitivity values in the form of peak ratios can vary from one system to another. It is best to perform the benchmark measurement when the detector is new or right after the window has been cleaned. This way you will be able to measure future performance against an optimum condition. Detectors already in service may already have some contamination on the window, usually vacuum pump oil.

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DO NOT ATTEMPT TO CLEAN YOUR DETECTOR WINDOW WITHOUT FIRST CONTACTING THE MANUFACTURER.

This test is not designed to measure absolute sensitivity of your detector, but rather a degradation of sensitivity over time. For this reason, it is important to check sensitivity at least every six months. This test should be performed whenever you check the FWHM resolution since both tests are designed to show changes in system performance over time.

IMAGE CALIBRATION

Many EDS systems come with image analysis software to measure particle size distributions and perform other size measurements. These software packages should be checked to ensure they are reporting accurate data.

The PELCO X-Checker® and PELCO X-Checker® B models come with two nickel grids that can be used to check the length and magnification calibration of your image analysis software. The 400 mesh grid is for low magnification calibration and provides a pattern shown in ***Figure 6***. The 1000 mesh grid is for calibration at higher magnification and provides a pattern shown in ***Figure** 7*. The PELCO X-Checker® Wafer has a 400 mesh nickel grid only.

NOTE: The PELCO X-Checker® Extra substitutes a beryllium half grid for the high magnification grid.

To perform a size check, place the appropriate grid under the beam, acquire an image in your EDS software. Use your image analysis software to measure the grid pattern and calculate the average grid opening area. You can do this at various magnifications, but it is a good idea to perform this test at or near the magnification with which you intend to work. Perform measurements in both X and Y directions. Any discrepancy with the micron marker on your SEM should be checked with a standard recommended by the SEM manufacturer. Some grids may have an arrow in the center of the grid; this arrow should not be used in your measurements. As there is some variation in grid opening size, make sure you include at least 30 grid openings in your measurement.

NOTE: This test is designed to see if your image processing software is working properly. It is not intended as an absolute test of the accuracy of the measurement displayed on the SEM. Your SEM manufacturer can recommend a high precision standard you can use to verify the performance of your microscope.

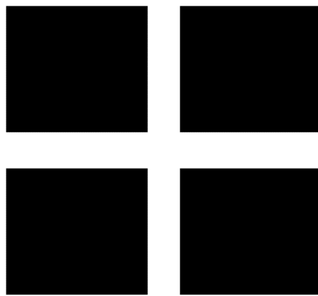

***Figure 6**: 400 Mesh Grid*

Grid opening = 38 x 38µm. Average grid area = 1,444µm² ±5% (2σ).

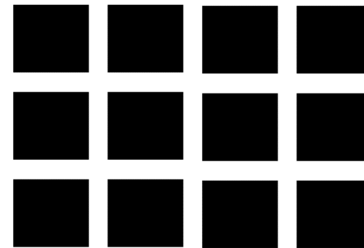

***Figure** 7: 1000 Mesh Grid*

Grid opening = 19 x 19µm. Average grid area = 361µm² ±5% (2σ).

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