Electron
Microscopy
Sciences

INSTRUCTIONAL MANUAL
CAT. 80058-ST X-Checker™ – Standard
CAT. 80058-BN X-Checker™ – Boron Nitride
CAT. 80058-EX X-Checker™ – Extra



Electron Microscopy Sciences

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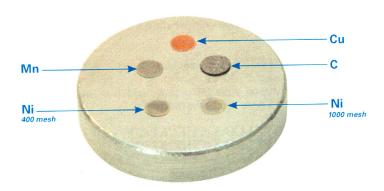
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Introduction & Features

With the X-CheckerTM, you can monitor the performance of your EDS/SEM systems in order to better and more quickly detect potential problems before they affect your analyses. This item provides various tests, which can perform periodically to verify the performance of your system. Please note that the tests vary depending on your particular model of the X-CheckerTM.

This manual describes three models: (1) CAT. 80058-ST X-Checker[™], Standard (2) CAT. 80058-BN X-Checker[™], with Boron Nitride, and (3) CAT. 80058-EX X-Checker[™], Extra. CAT. 80058-BN adds boron nitride and a 1000 mesh beryllium grid in place of the 1000 mesh nickel grid, to test low end performance and fluorine to measure low end resolution. All X-Checker[™] models mount materials on an aluminum stub.

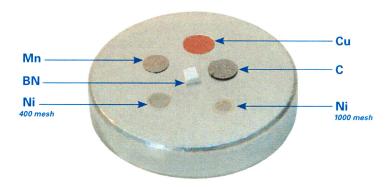
To clean your aluminum stub, rub it gently with a cotton swab and alcohol. Do not touch the cotton swab to the grids as they are very delicate and prone to damages.



80058-ST X-Checker™ - Standard

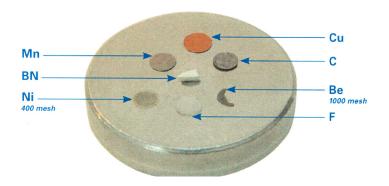
The follow tests can be performed with the X-Checker $^{\text{TM}}$, Standard model.

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



80058-BN X-Checker™ - Boron Nitride

The X-Checker[™] with Boron Nitride adds boron nitride as a more sensitive monitor of low end performance on thin window and windowless protectors.



80058-EX X-Checker™ - Extra

The X-Checker™ Extra model adds the following tests:

- 1. Boron nitride to test low end performance/peak separation.
- PTFE as a fluorine source to measure low end resolution.
- 3. A 1000 mesh beryllium grid (replaces the nickel high mag grid) for verifying the low end sensiti

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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:

- 1. Set the SEM to its lowest magnification and orient the X-Checker[™] with copper under the beam so that on the view CRT you observe approximately ¾ copper and the remainder aluminum. The accelerating voltage should be at 20 kV and the scan speed at TV or rapid scan.
- 2. Adjust the condenser lens (spot size) and final aperture to achieve a count rate of about 1000 X-ray counts per second (count rate for a pure element).
- 3. On the spectrometer, adjust the ration of aluminum and copper on the SEM CRT, if necessary, but moving the stage or changing the magnification so the aluminum and copper peaks are about the same height.
- 4. Once you have obtained a satisfactory spectrum with this procedure, follow the calibration instructions for your EDS system as outlined by the manufacturer.

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).

- 1. Orient the X-Checker™ so that carbon is visible on the SEM and fills about ¾ of the screen.
- 2. 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.
- 3. Adjust the condenser lens (spot size) and final aperture to achieve a count rate of about 1000 counts per second (count rate for a pure element). Follow the same system calibration procedure used for the copper and aluminum procedure.

NOTE: On some systems, this low energy calibration, while delivering 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 copper alone, and calibrating on the L-alpha and K-alpha lines (0.93 and 8.04 keV respectively). This should provide a sufficient scale calibration that may not be good for general use. This calibration should be performed at 10 kV.

If you have the X-Checker[™] with Boron Nitride model or the X-Checker[™] Extra model, you have the option of using both boron nitride and even the beryllium (X-Checker[™] Extra model only) peaks for low end calibration.

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

Resolution

The resolution of your EDS system should be checked frequently to ensure the correct operation of your detector, electronics, and all of its pars. This is easily done using the manganese standards by measuring the full width at half max (FWHM) of the manganese K-alpha peak. If you have the X-Checker™ Extra model, you can also perform a resolution test on the fluorine K-alpha peak with the PTFE standard. The test described in the aforementioned is valid for either the manganese peak or the fluorine peak.

To measure FWMH:

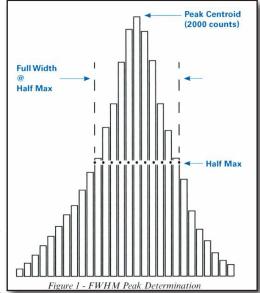
- 1. Orient the manganese (or fluorine) standard under the beam and set the magnification to 100X.
- 2. Set the accelerating voltage to 15 kV for manganese (or 5 kV for fluorine).
- 3. Adjust the condenser lens (spot size) and final aperture to get a count rate of about 1000 counts per second (count rate for a pure element). 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.
- 4. Acquire a spectrum until there are at least 2000 counts in the centroid (middle) channel of the manganese (or fluorine) peak.

To 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 1. Record this value in the resolution table. Resolution should be checked periodically and this table will provide a hand record of your detector performance over time.

It is recommended that you check the resolution of your system every six months.

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 most likely will not indicate a problem, however, 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 EDS system manufacturer.



NOTE: It is accepted in practice in the EDS industry to specify the resolution of all detectors of the manganese K-alpha peak using radioactive Fe-55 source. Measurements taken with the X-Checker™ are 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 X-Checker™ contains about 12% nickel so it is normal to see small nickel peaks in your spectrum.

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.

You will perform this test 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 dirt on the window. If you have a windowless or thin window detector, you may notice a small oxygen peak; this is normal.

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Sensitivity, continued



- 1. Set the accelerating voltage to 10 kV and put the beam in the center of the manganese standard.
- 2. Adjust the condenser lens (spot size) to achieve a count rate of about 1000 counts per second (count rate for a pure element) and acquire a spectrum for 100 seconds. If you need to tilt the sample, make sure to record the title angle since this test must be performed at the same tilt angle each time.
- 3. 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 table.

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 to monitor 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. Doing so will allow you measure future performance against an optimum condition. Detectors already in service will most likely have some contamination on the window, usually vacuum pump oil.

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 the 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

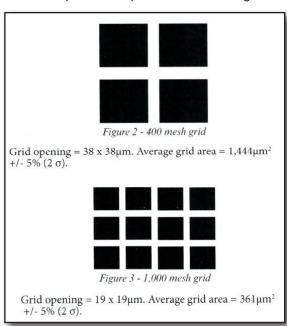
Many EDS systems come with image analysis software to measure particular size distributions and perform other size measurements. These software packages should be checked to ensure they are reporting accurate data,

The X-Checker[™] and the X-Checker[™] with Boron Nitride models come with two nickel grids that can be used to check size calibration of your image analysis software. The 400 mesh grid is for low magnification calibration and provides a pattern shown in Figure 2. The 1000 mesh grid is for calibration at higher magnification and provides a pattern shown in Figure 3.

NOTE: The X-Checker™ Extra substitutes a 1000 mesh beryllium grid for the high magnification grid. Thus grid should not be used for image calibration.

To perform a size check:

- 1. Place the appropriate grid under the beam and acquire an image in your EDS software.
- Using 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 advised to perform thus test at or near the magnification with which you intend to work.
- 3. 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 to 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 that you can use to verify the performance of your microscope.

Resolution Date/eV Sensitivity Date / Mn-K : Ni-L Tilt _

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