



## THERMO NORAN SYSTEM SIX ENERGY DISPERSIVE X-RAY SPECTROMETER

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*Insert Nickname Here*

# Operating Instructions

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## **Introduction**

**T**he Thermo NORAN System Six is a state-of-the-art energy dispersive x-ray spectrometer that is used in conjunction with the scanning electron microscope. The EDS system allows the user to acquire chemical composition information via x-ray collection, qualitatively or quantitatively analyze the chemical composition of a sample, and map the locations of specific elements in an image. The basic features of the EDS system are easy to use, and most operations are performed by clicking toolbar buttons.

## **Safety**

The EDS system is a safe instrument. You can do much more damage to it than it can do to you. When the scanning electron microscope electron beam is turned on, some x-rays are produced as a result of electron beam interaction with the sample, but these x-rays are of relatively low energy and do not escape the sample chamber.

## **Samples**

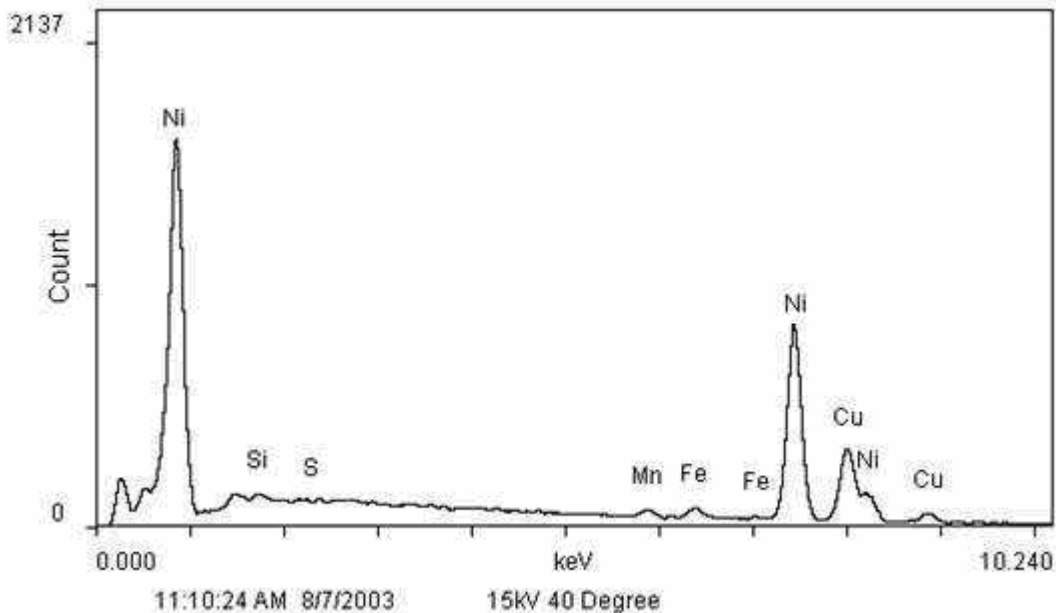
The EDS system may be used to analyze the chemical composition of any sample or sample area observed in the SEM.

## Background

**NOTE:** The following text is taken directly or adapted from the Corrosion Testing Laboratories webpage. See Resources for link.

One of the instruments most commonly used in conjunction with the SEM is the Energy Dispersive X-ray Spectrometer (EDS). Combining the EDS system with the SEM allows the identification, at microstructural level, of compositional gradients at grain boundaries, second phases, impurities, inclusions, and small amounts of material. In the scanning mode, the SEM/EDS unit can be used to produce maps of element location, concentration, and distribution.

The x-ray spectrometer converts an x-ray photon into an electrical pulse with specific characteristics of amplitude and width. After analysis of the electrical pulses, the output is a histogram of the x-ray energy received by the detector, with individual "peaks," the heights of which are proportional to the amount of a particular element in the specimen being analyzed. Below is an example output spectrum from an EDS scan of a copper-nickel alloy.



The locations of the peaks are directly related to the particular x-ray "fingerprint" of the elements present. Consequently, the presence of a peak, its height, and several other factors, allow the analyst to identify elements within a sample. With the use of appropriate standards and software, a quantitative analysis can be made of elements with atomic number of four (carbon) or greater.

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Since the sensitivity (ability to detect the presence of an element above background noise) of the EDS is very poor for low atomic weight elements, elements lighter than carbon cannot be detected. Further, resolution limits of the x-ray energy levels may affect the positive identification of certain elements (i.e., molybdenum and sulfur) due to overlapping energy slots.

Quantitative analysis is usually limited to flat, polished specimens. Unusual geometries, such as fracture surfaces, individual particles, and films on substrates may be analyzed, but with considerably greater uncertainty. *The standard guide for the performance of energy dispersive x-ray spectroscopy is covered in ASTM E 1508, "Quantitative Analysis by Energy-Dispersive Spectroscopy."*

## **References**

- J. I. Goldstein, et al., Scanning Electron Microscopy and X-Ray Microanalysis, Plenum Press, New York (1981).
- <http://www.corrosionlab.com/facilities/EDS.htm>
- ASTM E1508

## Instrument Operation

This section will walk you through the basic operation of the EDS system. You will follow a series of tasks that includes

- Powering up the instrument,
- Starting the software,
- Acquiring a spectrum,
- Identifying spectrum peaks, and
- Quantitatively analyzing your sample.

### Instrument Startup

1. Ensure that the SEM is on and functioning properly. The best way to ensure proper SEM function is to turn on the electron gun (HT ON) and obtain an image on the SEM screen. See the separate SEM operating instructions document for more information on SEM operation.
2. Check to see if the Noran System Six computer (stainless steel box) and EDS control computer (Dell on the right) are turned on. If both are already turned on, skip to the next section. If they are off, continue to Step 2.
3. Turn on the stainless steel NORAN System Six box by pressing the black button on the front panel. Wait for the blinking green light to appear next to “Active” in the front panel window.
4. Once the green Active light is blinking, turn on the Dell computer (the one next to the stainless steel box) and wait for it to boot.

### Software Startup

1. Once the computer has booted, open the EDS software by double clicking on the NORAN System Six desktop icon.
2. Open the project explorer by clicking on the File...Project Explorer pull down menu.
3. Create a new project under the *C:\Program Files\Thermo Electron\NORAN System SIX\0803 Olin Install\* directory by clicking on the “0803 Olin Install” folder and the **Create a new project** toolbar button (the first button on the left).
4. Name the project by clicking on the **Rename a project or folder** toolbar button and typing a new folder name.
5. Type your name in the **Author** box.
6. Hit **OK** to close the project explorer window.

## Collecting a Spectrum

1. Click on the **Spectrum** button in the left toolbar. In **Spectrum** mode, you should see a large window with a blue background at the top of the screen, a periodic table at the bottom left, and a window with **Attributes/Notes/Detector Status** tabs at the bottom right.
2. Before you start collecting a spectrum, give your data a title by typing a name in the white **Base name:** box at the left of the window.
3. To start collecting a spectrum, click on the **Start** Acquisition (black triangle) toward the right side of the toolbar. You should see a yellow spectrum appear.
4. The EDS detector was set up to work best at a **10 mm working distance and a 0° tilt**. The working distance is controlled by adjusting the z-axis knob on the sample stage, and this knob is mounted to a shaft that shows the approximate working distance value. A good way to set the working distance is to increase the sample height in increments of a few millimeters, refocus the image after each adjustment, and observe the change in the WD value on the SEM status bar. Continue the incremental sample height adjustment until your WD value is 10. This method works great for smaller or flatter samples that are aligned with the top of the sample holder, but it is not the safest method to use with large or irregularly-shaped samples.
5. Acquisition and microscope information is shown in the status bar at the bottom of the screen. Observe the **DT** value in the status bar. This number is the detector dead time, and its value should be approximately about 30 (plus or minus 5 or so). If it is not close to 30, adjust the SEM spot size until it is.
  - If DT is too low, slowly increase the spot size until DT is about 30.
  - If DT is too high, slowly decrease the spot size until DT is about 30.After you have adjusted the dead time, stop and re-start the spectrum acquisition. The acquisition will take about 1 minute.
6. Wait until the acquisition terminates according to the criteria set up in the EDS Acquisition Properties dialog box, or click the **Stop** button (black square) on the toolbar if you are impatient. The default acquisition time is approximately 1 minute.
  - You may view the peak identification during the acquisition by clicking on blue plot background and then clicking the **Identify Spectrum** (red fingerprint) button on the toolbar.
  - If you do not click the red fingerprint button during acquisition, the peaks will be automatically labeled after acquisition is complete.

## Identifying Peaks

**WARNING:** Basing your quantification solely on the automatically identified peaks is generally not the best thing to do. Use your intuition, your knowledge, your experience, and perhaps a little help from your local experts.

As mentioned in the previous section, the software will automatically identify and label the spectrum peaks. The positively identified elements (at least according to the software) will show up in **green** on the periodic table. Possible hits will be highlighted in **orange**. If the automatically determined elements do not look correct, try the following procedures:

1. Go back up to the spectrum and activate the Energy Cursor by clicking on the Energy Cursor toolbar button (the button with the light blue vertical line...close to the black triangle button you used to start the acquisition). This should cause a light blue, vertical line to appear in your spectrum window.
2. Grab the light blue line and try moving it with the mouse (click and drag).
3. As you drag the light blue line over one of your spectrum peaks, notice what happens in the periodic table window. Elements with x-ray energies close to that of the peak are highlighted in light blue on the periodic table! Isn't that cool? This should give you a better shot at guessing the elements that produced your spectrum peaks.

## Quantifying a Spectrum

1. To automatically generate quantitative data from a spectrum, click the **Quantify Spectrum**, button in the middle of the top toolbar (it looks like a % symbol on a balance).
2. Click the **Quant Results** tab to view the elemental breakdown of your spectrum.
3. Save your results by clicking the **Save** toolbar button or the File...Save pull down menu option.

**NOTES on O, C, and Excluded Elements:** Oxygen and carbon peaks often show up for metals that have been handled and exposed to atmospheric conditions. If you're not interested in O or C, or you know your sample is an alloy with very little C or O, you may want to **exclude** these element from the quantification. To exclude elements, click the atomic symbol on the periodic table until the element is shaded in striped gray.

The **Excluded** option may be used for any elements that are erroneously identified by the software, or those that you simply are not interested in quantifying.

**STOLK'S BEDTIME STORIES, Part 1:** Assuming the computer is right is not the best thing to do. Why, just the other day, I placed a brass sample into the SEM, collected an EDS spectrum, and automatically quantified the results, the system provided the following breakdown:

Element	Weight Percent
C	2.05
O	1.35
Al	0.12
Cu	32.48
Tc	2.41
Re	60.37
Os	1.22

What exciting results these were! Given these data, I had no choice but to track down the source of this magical brass. I was going to be rich, I tell you! Rich! The rhenium alone could make me a bundle. That stuff ain't cheap! And neither is the osmium! But the real money maker here has



got to be the technetium. Technetium...oh, my goodness. I may be the first person alive to find naturally-occurring technetium, and it's mine all mine!

Now, I'm no dummy. I know that technetium, rhenium, and osmium are not common alloying elements for copper. A more careful analysis of the spectrum (using the sliding blue eV line) reveals that the Re, Os, and Tc lines overlap with lines from more common elements. For example, rhenium overlaps with a zinc peak. Think zinc is a more likely candidate for this chunk of brass? Yeah, me too. Similarly, the osmium (8.910) L line almost perfectly overlaps with the Cu (8.907) K line, so I could pretty safely assume that the gobs of Os were more likely gobs of Cu.

I also recognized that the carbon and oxygen are likely on the surface of my brass sample and probably not alloying elements in the brass. So, what should I do? Careful scientist that I am, I decided to ignore the oxygen and carbon. I'm guessing they were caused by surface contaminants and a surface oxide layer.

After appropriate changes to the periodic table and a re-run of the quantification, we see the following results:

Element	Weight Percent
Al	0.71
Cu	57.82
Zn	39.84
Pb	1.63

Ahhh, that's better. Looks like a brass...pretty good match with a common, leaded brass...perhaps Alloy 370 or Alloy 377.

## System Shutdown

Under normal circumstances, the EDS system may remain turned on, and shutting down the EDS system is not necessary. If you wish to shutdown the system,

1. Close the software.
2. Turn off the Dell computer used with the EDS system.
3. Turn off the stainless steel NORAN System Six computer.



## X-Ray Mapping

**X**-ray mapping is a powerful but somewhat time consuming EDS analytical technique. EDS x-ray mapping allows one to identify and map the locations of specific elements in a microstructure. If you wish to learn to the x-ray mapping feature, please see your instructor.