Examination of the Fluorine K Line and Iron L Line Overlap by EDS and WDS

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Key Words
MagnaRay, EDS, Fluorine, Iron, Overlapping Peaks, Peak Deconvolution, WDS

From time to time when analyzing an iron rich material, an analyst may see the peak identification include fluorine (F) even though that element is not likely to be present. This document will explore why that happens.

The heart of the problem is that the x-ray lines for F-K and Fe-L are quite close in energy.

F-Kα 677 eV
Fe-Lα 704 eV

The difference is 27 eV, which is fairly close but not an insurmountable challenge for modern EDS detectors and peak detection algorithms. Depending on the detector type and the amplifier settings the peak widths in this region are on the order of 60 or 70 eV.

Although it may be difficult to visually confirm the presence of fluorine in a sample of iron, it should be relatively easy for the peak fitting algorithms to separate these peaks given sufficient precision in the data.

That last conditional is a key to the likely reason why F may be misidentified in a sample. EDS spectra are built up by counting individual photons and adding them one by one to the spectrum. A characteristic of this kind of data is that the precision of the peak centroid and peak width are tied to the number of counts acquired. The same applies to the overall shape of the peak. The result is that when there are only a small number of counts in the peak then it is easy for the software to see the presence of F when there is none. This is similar to driving in a thick fog where it is difficult to see the road ahead. On a sunny day the same road is easy to navigate. When a lot more data has been acquired the identification problem becomes much easier and the software will generally correct itself and remove the F label – if, in fact, F is not present.

Several spectra reproduced here illustrate this peak overlap. EDS spectra were taken from samples of manganese, iron, bixbyite, triplite, zinnwaldite and fluorite. The following table lists the combinations of Mn, F and Fe in these samples.

Sample	Mn	F	Fe
Manganese	✓		
Fluorite		✓	
Iron			✓
Bixbyite	✓		✓
Triplite	✓	✓	✓
Zinnwaldite	✓		✓

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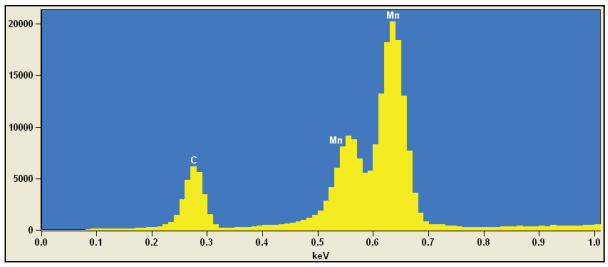
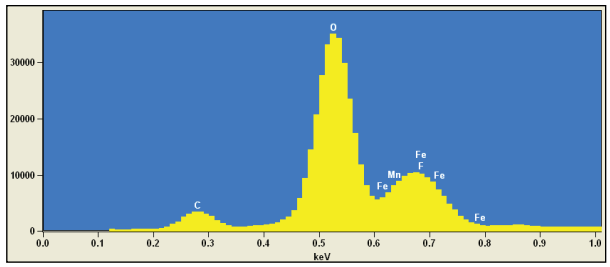
This group of samples provides all combinations of Fe and F present or not with the added complication of the possible presence of the Mn-L line, which at 636 eV is 41 eV lower in energy than the F-K line. This is far enough away that it doesn't cause any issue with misidentifying F in the presence of Mn.

The following figures display these six spectra for easy comparison. Viewing the spectra confirms that visually it is virtually impossible to determine what elements are present or not in this region of the spectrum.

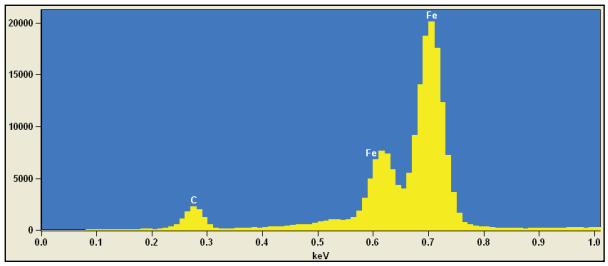
Collection of EDS spectra



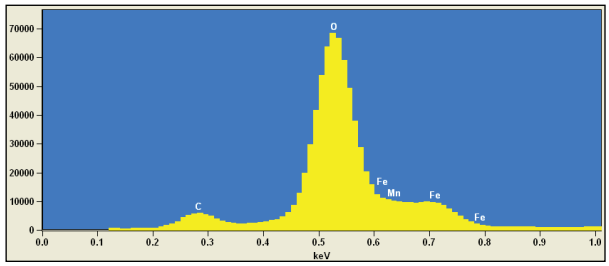
Pure manganese



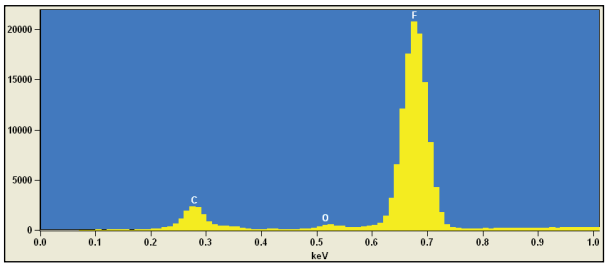
Triplite



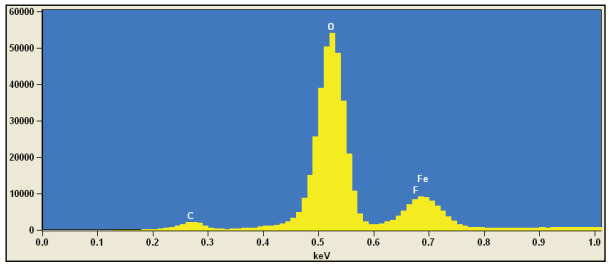
Iron



Bixbyite



Fluorite



Zinnwaldite

Using a Thermo Scientific™ MagnaRay™ WDS spectrometer, readily confirms the presence or absence of F in these samples. The MagnaRay spectrometer features much higher resolution than an EDS detector. In this region of the spectrum the MagnaRay peak widths are about five times narrower than the EDS peaks. This makes it possible to view all of the lines in question without concern for the overlap. For the WDS spectrum a sample of magnetite was used in place of the pure iron which was used to collect the EDS spectrum of iron.

Nominal compositions of the mineral samples analyzed here.

Bixbyite

Manganese	52.05%
Iron	17.64%
Oxygen	30.32%

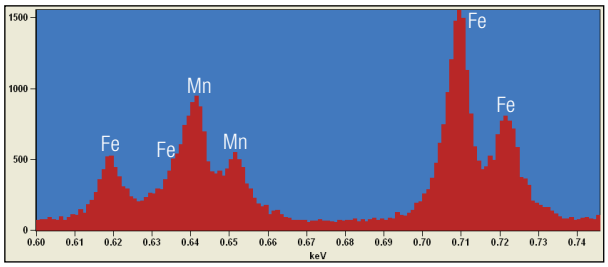
Triplite

Calcium	3.79%
Magnesium	3.45%
Manganese	25.96%
Iron	13.19%
Phosphorus	14.64%
Hydrogen	0.12%
Oxygen	32.13%
Fluorine	6.73%

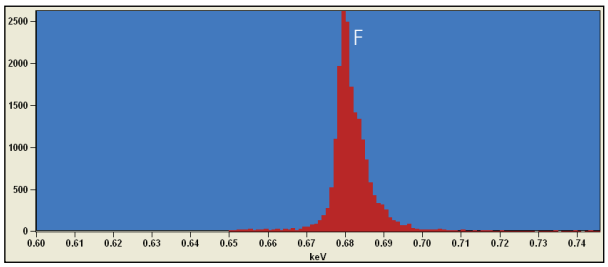
Zinnwaldite

Potassium	8.94%
Lithium	1.59%
Aluminum	12.35%
Iron	12.78%
Silicon	19.28%
Hydrogen	0.12%
Oxygen	38.43%
Fluorine	6.52%

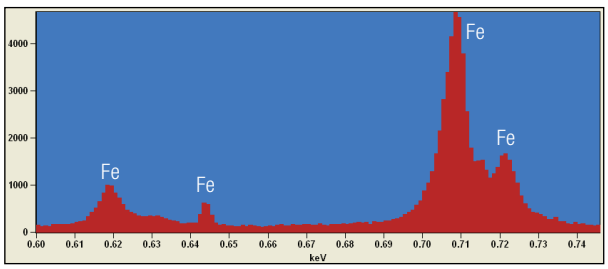
Collection of WDS spectra



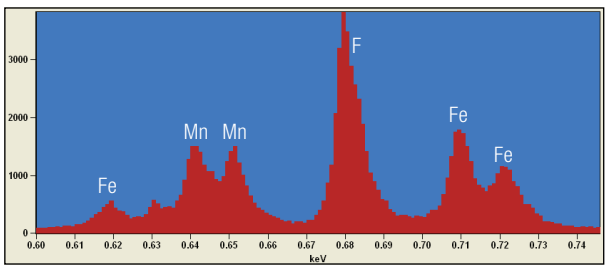
Bixbyite



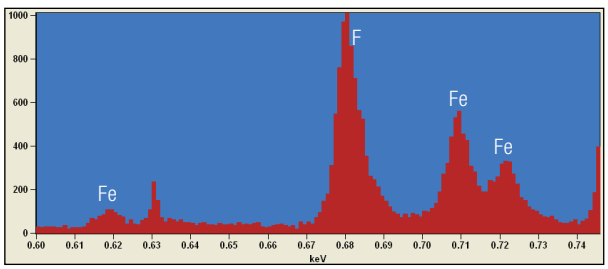
Fluorite



Magnetite



Triplite



Zinnwaldite

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