



Technical Note #2001

TRACER 5 Spectrometer Mode

Introduction

This note is intended to give advice regarding Spectrometer Mode settings which will allow the measurement of a set of elements. There are several considerations which must be understood in order to safely measure any sample. These include the radiation safety practices as well as the illumination settings required.

Radiation Safety

The details of the radiation profile of the instrument are provided in the User Guide. It is important to understand that the X-ray beam generated by the instrument can cause harm to users if they are exposed directly to it. As the current and voltage are increased, the intensity of the beam will increase. The user should be aware of the direction of the beam as it exits the instrument and always configure the experiment to minimize the exposure to the operator.

When measuring thick metallic samples which completely cover the measurement window on the instrument, the risk of exposure to the beam is minimized. However, when measuring small samples which do not completely cover the measurement window or thin samples which do not stop all the X-rays, some of the beam will escape mostly in the direction of the beam. Further, when measuring plastic, liquid and powder samples, some of the beam will be scattered in all directions. For these reasons it is strongly recommended that all measurements be made in either the Desktop Stand with the radiation cover installed (Figure 1) or in the Benchtop Stand. These stands are designed to minimize the exposure outside of the measurement chamber.

Figure 1: TRACER 5g in Desktop Stand



Illumination Considerations

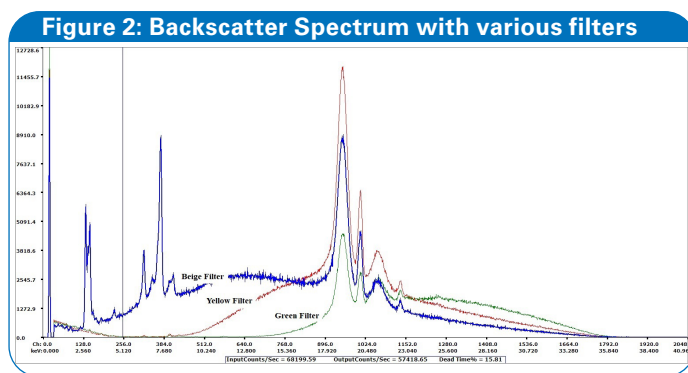
The basic illumination settings include the voltage, current and filter used for the measurement. In addition, the collimator and atmosphere used will influence the results obtained.

Voltage:

The excitation of each element is best achieved using an excitation voltage (keV) which is slightly above the absorption edge for the element. The absorption edge for each element is slightly above the emission energy of the element. Generally, a measurement is made of a series of elements with the excitation voltage being slightly higher energy than the highest energy of the group. Thus, low atomic number (Z) elements such as Mg, Al, Si will be analyzed using low excitation voltages such as 10-15 keV while high Z elements such as Fe, Cu, Mo will be analyzed using a high excitation voltage such as 40-50 keV.

Filter:

The filter employed helps to condition the X-rays used to excite the sample. It is desirable to have the background in the spectrum as low as possible at the energy of the element to be measured. Filters allow you to adjust the energy of the excitation beam and the background. If the Beige filter (no filter) is used with a 40 keV excitation voltage, much of the X-ray flux will be from the bremsstrahlung which will dramatically increase the background (Figure 2, Blue) in the spectrum. If a Yellow filter (25 μm Ti, 300 μm Al) is used, this background (Figure 2, Red) will be substantially decreased. If an even harder filter such as the Green filter (100 μm Cu, 25 μm Ti, 300 μm Al) is used, the background (Figure 2, Green) will be further reduced. Inclusion of a filter to modify the excitation beam dramatically reduces the number of X-rays which reach the sample. A complete list of the filters available on the TRACER 5, both in the built-in filter wheel and as manual filters is given in Appendix A.



Current:

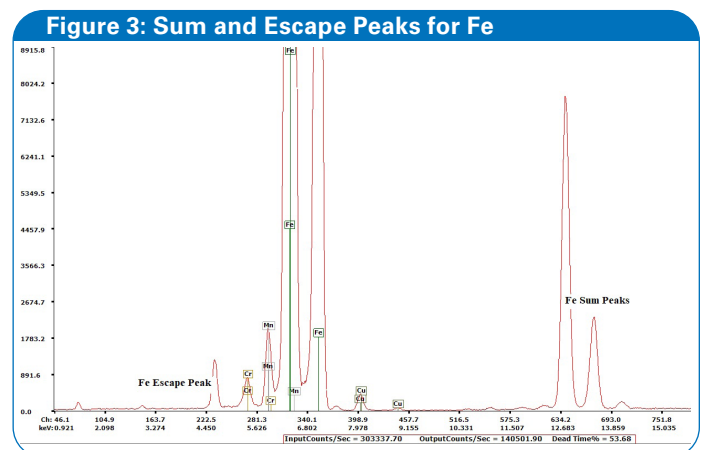
Current is the ultimate control of the excitation intensity and the resulting count rate of the measurement. As the current is increased, the count rate increases. As stated above, adding a filter to the excitation path will reduce the intensity of the excitation. The intensity can be increased while keeping the lower background by increasing the current.

The TRACER 5 source provides 4 watts of power at 40 keV (e.g. 40 keV, 100 μA). The current limit at each voltage is listed in Appendix A. The current is normally adjusted so that the output count rate is between 30,000 and 60,000 counts/second with a deadtime of less than 50%. This is the optimum compromise between measurement time and spectral artifacts. The precision of an XRF measurement is proportional to the square root of the number of counts in the element window of interest. Thus, higher count rates provide better precision for a given measurement time. There will be cases where the current limit of the instrument will limit the count rate. In these cases, simply use the maximum current available for the voltage being used.

As the count rate increases, artifacts such as sum peaks and escape peaks will occur. A sum peak is generated when two X-rays arrive at the detector almost simultaneously and are measured as one X-ray of higher energy. The probability that two X-rays will arrive at the detector and be seen as one, increases exponentially with count rate. They are commonly seen as a peak at twice the energy of the strongest peak in the spectrum or if there are multiple strong peaks, a peak at an energy equal to the sum of the two strongest peaks. If you see a sum peak from a $K\alpha$ peak it is likely that you will also see a sum peak from the $K\alpha + K\beta$ peaks that is much weaker. In the case of a strong $L\alpha$ and $L\beta$ set of peaks you will see three sum peaks.

Another artifact which may be seen in a spectrum is an escape peak. This is a peak at 1.74 keV lower energy than the parent peak. This peak is generated when the incoming X-ray fluoresces the silicon of which the detector is made. The Si $K\alpha$ fluorescence energy of 1.74 keV escapes the active detector area, leaving the rest of the initial X-ray energy to be measured. Figure 3 shows an example of a high-count rate spectra of iron. The sum peaks are at 12.8 keV ($K\alpha + K\alpha$) and 13.6 keV ($K\alpha + K\beta$). The escape peak is at 4.66 keV (6.40 keV - 1.74 keV).

Thus, the optimum count rate for any measurement is a compromise. High count rates can be used to maximize the precision of a measurement within a given measurement time; lower count rates will result in fewer and lower artifacts which will make qualitative analysis of the spectrum much easier.



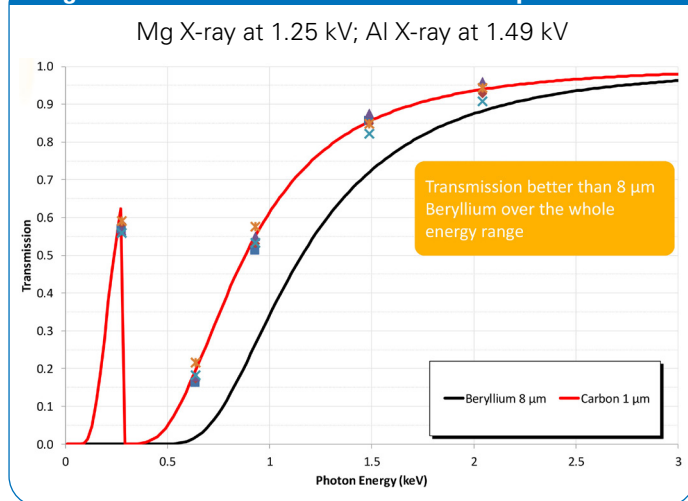
Collimator:

In addition to the three basic settings of the illumination, the collimator and the atmosphere also have an impact on the measurement. The TRACER 5 is supplied with two collimators, one which generates an 8 mm oval spot on the sample being measured and one which generates a 3 mm oval spot. The smaller spot size is commonly used to measure small samples which are smaller than the 8 mm spot. Given the same illumination settings, the use of the 3 mm collimator will reduce the excitation intensity by a factor of about seven (7) (the intensity is proportional to the area of the collimator) compared to the 8 mm spot.

Atmosphere:

Atmosphere will impact the results obtained in an XRF measurement, especially for light elements like Na, Mg, Al and Si. Any material between the sample and the detector will absorb some of the low-energy X-rays emitted by the sample before they arrive at the detector to be counted. This absorption will increase as the energy of the X-ray to be measured decreases. For example, the air path between the sample and detector will absorb about 75% of the X-rays emitted by silicon (1,740 eV) and will absorb about 90% of the X-rays emitted by magnesium (1,250 eV). Thus, materials like the detector window and the atmosphere will dramatically impact the number of X-rays which are detected. The TRACER 5i is equipped with 8 μm Be detector window while the TRACER 5g is equipped with a 1 μm Graphene window which has substantially better transmission for all elements from F to Si (see Figure 4). Similarly, the use of a He environment between the sample and detector will substantially improve the measurement of all elements from Na to Si (please see Appendix B).

Figure 4: Transmission Curve Be and Graphene



Suggested Measurement Conditions

The following section provides suggested measurement conditions for a variety of sample types and elements of interest. It serves as a guide to help you optimize conditions for your analytical objectives using the Spectrometer Mode.

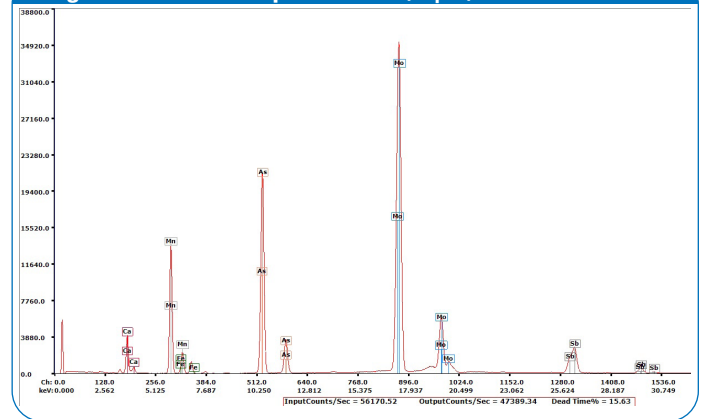
Screening:

High Z elements (Ti to U)

- Filter: 1-Yellow (25 μm Ti, 300 μm Al)
- Tube Voltage: 50 keV
- Tube Current: Adjust for count rate starting at 5 μA
- Atmosphere: Air

These settings will have the greatest effect on the heavier elements (Ti \rightarrow U) which are excited by the higher energy X-rays.

Figure 5: Glass sample at 50 kV, 5 μA , Yellow Filter



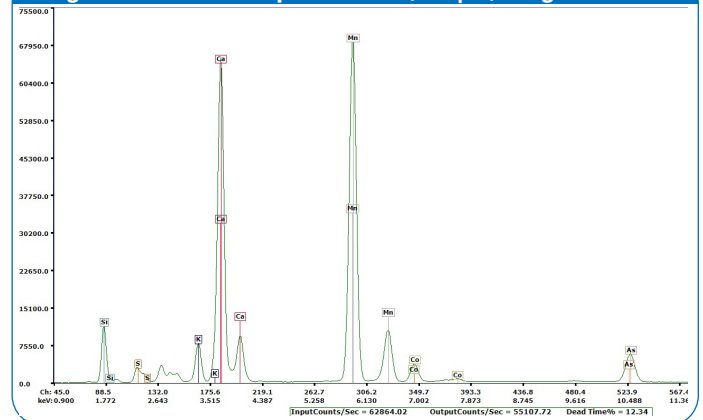
Screening:

Low Z elements (Na to Ca)

- Filter: 2-Beige (None)
- Tube Voltage: 15 keV
- Tube Current: Adjust for count rate starting at 15 μA
- Atmosphere: He or Vacuum if available

These settings provide efficient excitation of light elements from Na to Ca. As there is no filter in the excitation path the Rh $L\alpha$ X-rays at 2.7 keV will excite the light elements: Na – Si. The use of a He (or vacuum) atmosphere will enhance the sensitivity for very low Z elements such as Na, Mg, and Al.

Figure 6: Glass Sample at 15 kV, 15 μA , Beige Filter



Suggested Measurement Conditions (cont.)

Measurement of Heavy Metal Alloys:

Heavy elements (Ti to U)

- Filter: 1-Yellow (25 μm Ti, 300 μm Al)
- Tube Voltage: 40 keV
- Tube Current: Adjust for count rate starting at 5 μA
- Atmosphere: Air

These conditions will provide good excitation of the major elements in iron, cobalt, nickel and copper alloys and the presence of the Ti/Al filter reduces the background throughout the spectrum.

Measurement of Heavy Metal Alloys:

Light Elements (Mg to Zn)

- Filter: 2-Beige (None)
- Tube Voltage: 15 keV
- Tube Current: Adjust for count rate starting at 10 μA
- Atmosphere: Air

These conditions are good for exciting the low Z elements in alloys such as aluminum and silicon. These conditions are appropriate for measuring elements up to zinc (Z=30). He or vacuum is not required for these samples as the aluminum and silicon are generally at high enough concentration that they can be measured in the air atmosphere.

Poisons (Hg, Pb, As)

- Filter: 3-Violet (75 μm Cu; 25 μm Ti; 200 μm Al)
- Tube Voltage: 50 keV
- Tube Current: 20 μA
- Atmosphere: Air

These conditions give good excitation of the elements of interest and provide background reduction in the area of the peaks of interest. Note: when reviewing the spectra care must be taken to distinguish between lead and arsenic as the lead $L\alpha$ directly overlaps the arsenic $K\alpha$ peak. The lead $L\beta$ peak will distinguish the two.

Figure 9: Sample containing As and Pb; 50 kV, 30 μA , Violet Filter

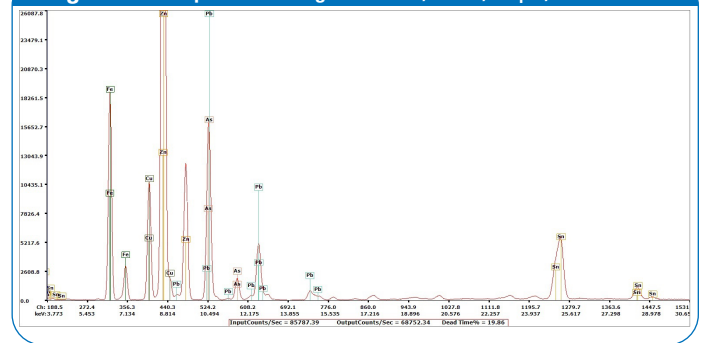
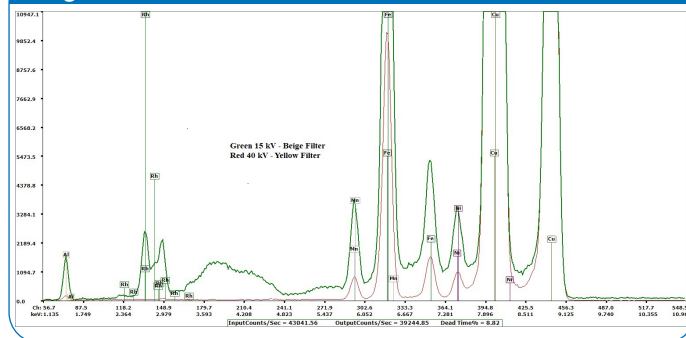


Figure 7: Aluminum Bronze; normalized on Cu $K\alpha$



Geological/Ceramics Majors

- Filter: 2-Beige (None)
- Tube Voltage: 15 keV
- Tube Current: Adjust for count rate starting at 15 μA
- Atmosphere: Air; He will give significant improvement for very light elements Na, Mg, Al

These conditions will provide excitation for the major components of geological and ceramic samples, e.g. Na to Ca. Note that a He (or vacuum) environment substantially improves the sensitivity for the very light elements such as Na, Mg and Al. Likewise, the use of the Graphene window detector of the TRACER 5g will improve the sensitivity for Na by about a factor of three.

Obsidian Sourcing (especially Rb, Sr, Y, Zr, Nb)

- Filter: 4-Green (100 μm Cu; 25 μm Ti; 300 μm Al)
- Tube Voltage: 50 keV
- Tube Current: 35 μA
- Atmosphere: Air

Figure 8: Obsidian Sample; 50 kV, 35 μA , Green Filter

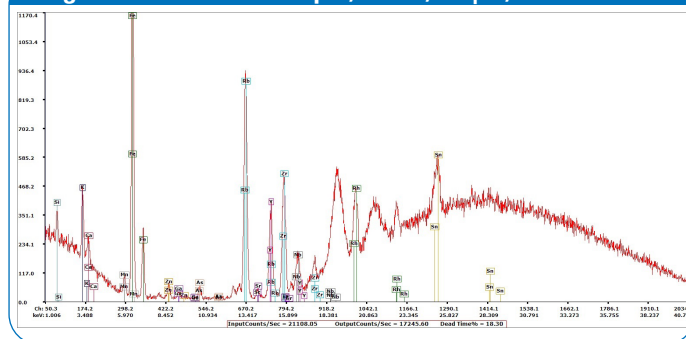
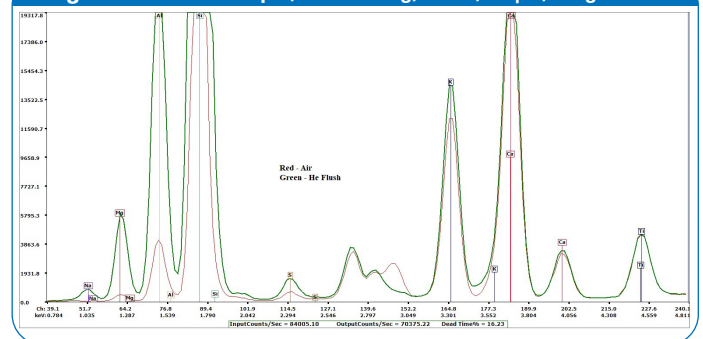


Figure 10: Rock sample; TRACER 5g, 15 kV, 15 μA , Beige Filter

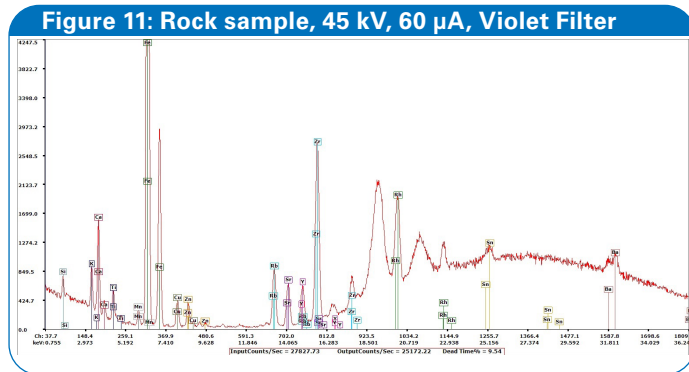


Suggested Measurement Conditions (cont.)

Geological/Ceramics Trace

- Filter: 3-Violet (75 μm Cu; 25 μm Ti; 200 μm Al)
- Tube Voltage: 45 keV
- Tube Current: start at 50 μA
- Atmosphere: Air

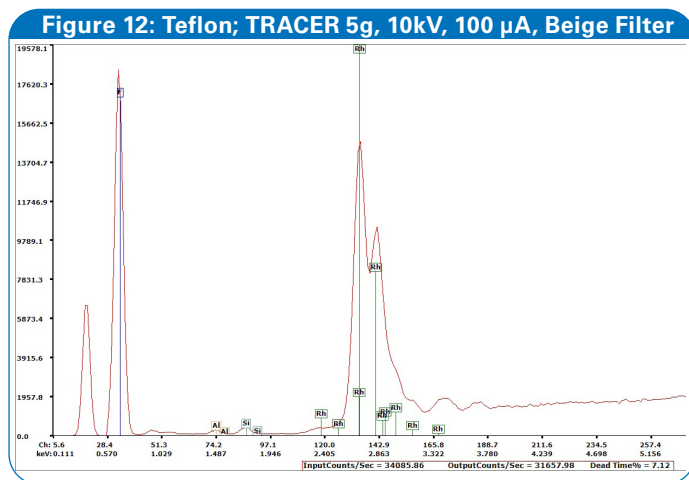
These conditions provide excitation of the heavy elements from Ti to U. Note that the high voltage allows detection of Ba using the K lines.



Fluorine Detection

- Filter: 2-Beige (None)
- Voltage: 10 keV
- Tube Current: 100 μA
- Atmosphere: He; Graphene window detector

This measurement must be made with a TRACER 5g in a nose down configuration with the standard Prolene window removed. See Appendix B for notes on operation in this configuration.



Appendix A: TRACER 5 Filters and Currents

TRACER 5 Filter Definition

LAYER 1	LAYER 2	LAYER 3	TOTAL	TRACER 5	COLOR
25 μm Ti	300 μm Al		325 μm	1	Yellow
				2	Beige
75 μm Cu	25 μm Ti	200 μm Al	300 μm	3	Violet
100 μm Cu	25 μm Ti	300 μm Al	425 μm	4	Green
38 μm Al			38 μm	5	Purple
25 μm Ti			25 μm	Included	Blue
200 μm Cu	25 μm Ti	300 μm Al	525 μm		Black
76 μm Al			76 μm		Orange
60 μm C	190 μm F		250 μm		White
25 μm Cu	25 μm Ti	300 μm Al	350 μm		Red

Filter #5 is 38.5 μm Al. Software does not show decimals in filter thickness.

TRACER 5 Current Limit

Voltage	Current
6 keV	195 μA
10 keV	180 μA
30 keV	130 μA
40 keV	100 μA
50 keV	35 μA

Current value at intermediate voltages is determined by interpolation; for example, at 35 keV the maximum current would be 115 μA .

Appendix B

Operation of TRACER 5 using He Atmosphere

In order to achieve the maximum sensitivity with the TRACER 5 it is necessary to use a helium atmosphere and to remove the Prolene window on the front of the instrument. When this is done it is essential to use the instrument in a nose down configuration (Figure 13). This configuration minimizes the chance that any foreign material will enter the volume around the detector and other important mechanical parts. The presence of foreign material in the front end of the instrument and attempting to remove such material could damage the detector or the mechanics of the instrument.

When operating with a He environment, the flow rate can be quite low. Set the flow regulator for 0.1-0.2 l/min. To flush the air from the front end run the flush for about 5 minutes before making the first measurement.

The instrument should not be used without the Prolene window with anything other than solid materials like rocks and metal samples. Do not use the instrument in this configuration when measuring powder or liquid samples. Powder samples can be disturbed by the flow and there is a possibility that the powder could enter the front end of the instrument and cause damage. Liquid samples are dangerous as it is possible that the liquid may evaporate and condense at the detector which is cooled. This could lead to significant damage to the instrument.

A He atmosphere can be used with the Prolene window installed when it is not possible to operate in a nose down configuration. The window will absorb some of the light element X-rays, but this configuration will give much better sensitivity for Na, Mg, Al and Si than using an air atmosphere.

When operating in a nose down configuration the best way to arrange the unit is to mount it in the desktop stand and raise the sample to the instrument using a scissor stand as shown in Figure 13. This allows the sample to be raised to meet the front of the instrument. In cases where the sample is not flat on both sides, molding clay is helpful in arranging the flat surface to be measured in the appropriate configuration.

Figure 13: Molding clay used to help level the measurement surface



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