XRF Instrumentation

Introduction to sources

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Instrument

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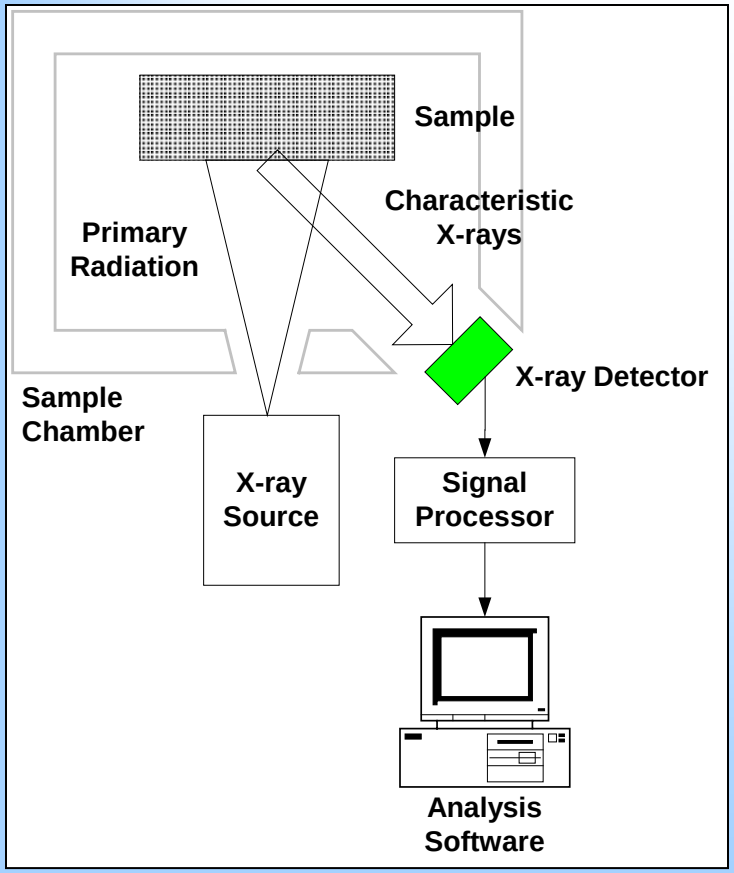

- Excitation source
  - X-ray tube or radioisotope
- Spectrometer
  - X-ray detector
  - Signal processing electronics
- Software
  - Spectrum correction and processing software
- Other
  - Radiation shielding
  - Sample fixture

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Excitation Source

Sources

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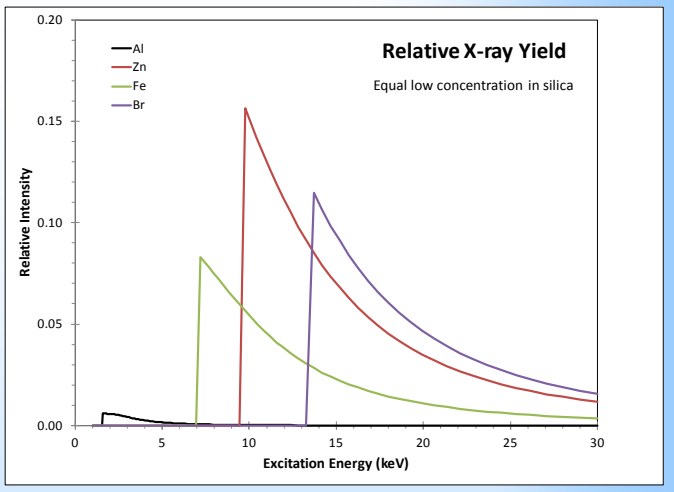
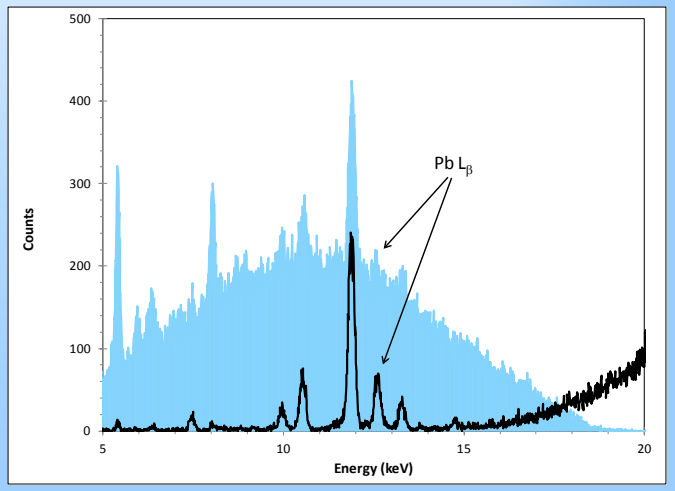
What is the purpose of the source?

- Excite elements to be analyzed
- Produces the signal



What is an ideal XRF source?

- Energy just above edge of analyte
- Monoenergetic → Low background
- Intense flux → Fast measurement
- Stable flux → Accuracy
- Can be turned off → Radiation safety
- Long life, inexpensive



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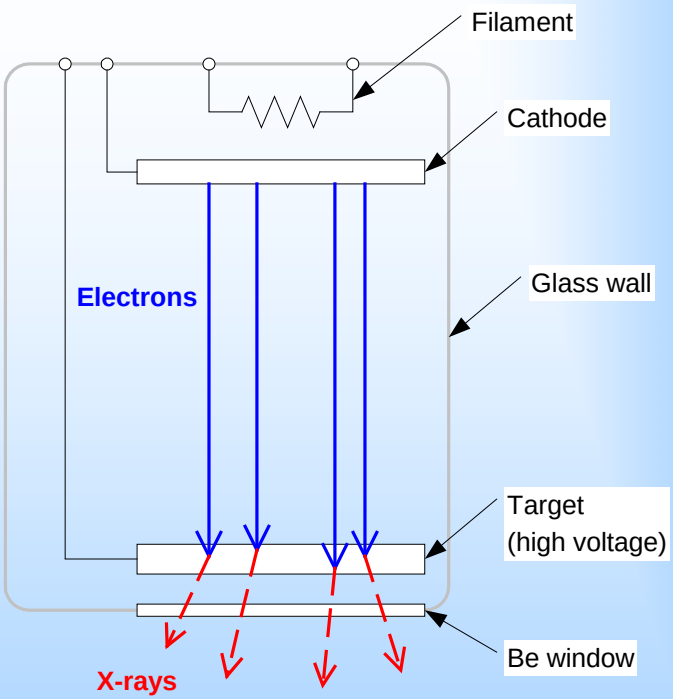
X-ray tubes

- Heated filament produces electrons
- High voltage (10 to 50 kV) target accelerates electrons
- Electrons hitting target → X-rays
  - Characteristic X-ray lines from target material
  - Bremsstrahlung continuum from electron scattering
- Many variations
  - Anode material
  - Maximum bias and current
  - Side window, end window, transmission
  - Microfocus, pulsed, DC



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X-ray tube variations

- Geometry
  - End window: Permits a very tight geometry but power is limited
  - Side window: Allows much higher power but also much larger
- Anode material
  - Produces characteristic X-rays. Can be useful for exciting sample or can interfere
  - Ag is very common (almost a standard). Rh, Pd, Au, W also used
- Maximum HV and current
  - HV setting is critical for exciting sample. High current permits fast measurement
  - Higher HV and current require more shielding and radiation safety issues
- Specialties
  - Microfocus
  - Pulsed

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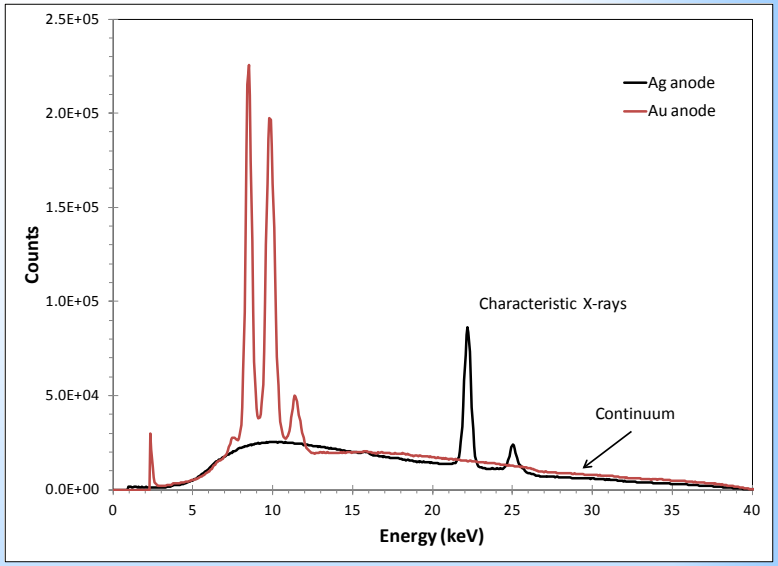
X-ray tubes for XRF

- Disadvantages
  - Most X-rays are in continuum → High background
  - Large, high power, heat dissipation
- Advantages
  - Can change HV and filters → Tune excitation for different elements
  - High intensity possible
  - Can turn it off, improves safety



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Radioisotopes

- Monoenergetic → Low background
- No power dissipation
- Fixed, limited emission energies
- Cannot turn them off
- Intensity falls with half life

- Modern tube technology has replaced sources in many but not all applications

	^{55}Fe	^{109}Cd	^{241}Am	^{57}Co
Energy (keV)	5.9	22.1 88	59.5	122
Elements (K lines)	Al - V	Fe - Mo	Ru - Er	Ba – U
Elements (L lines)	Br - I	Yb - Pu		

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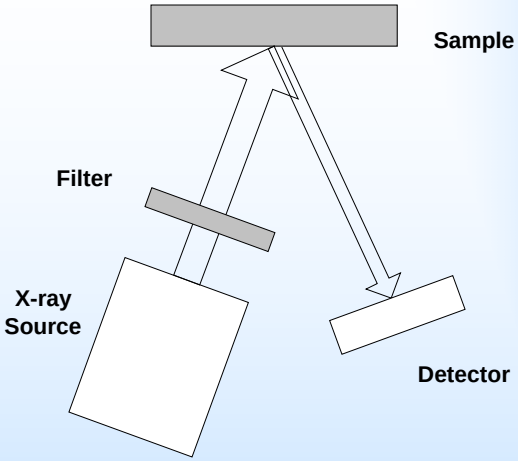
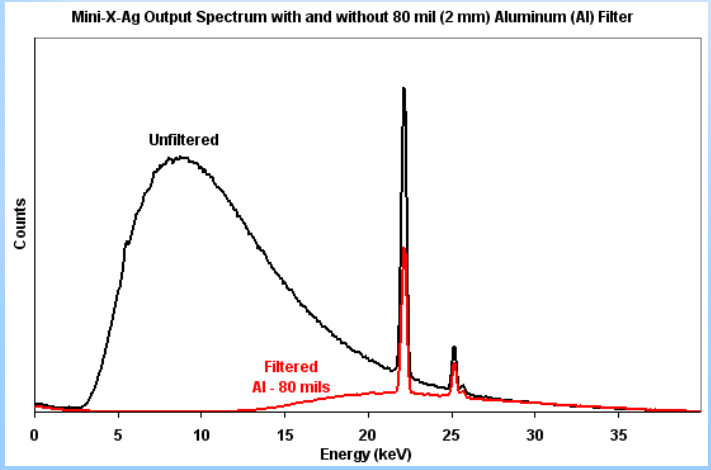
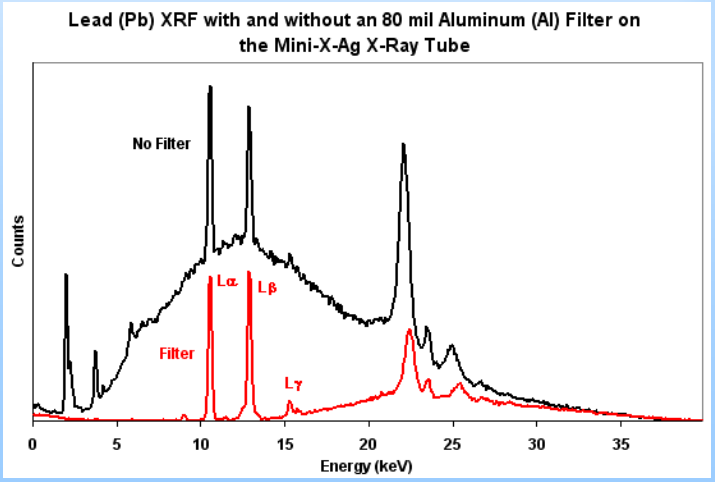
Source filter

- Attenuates incident X-ray below attenuation edge of filter material
- Reduces background
- Greatly enhances signal to background
- Optimum filter depends on energy of X-rays to be analyzed







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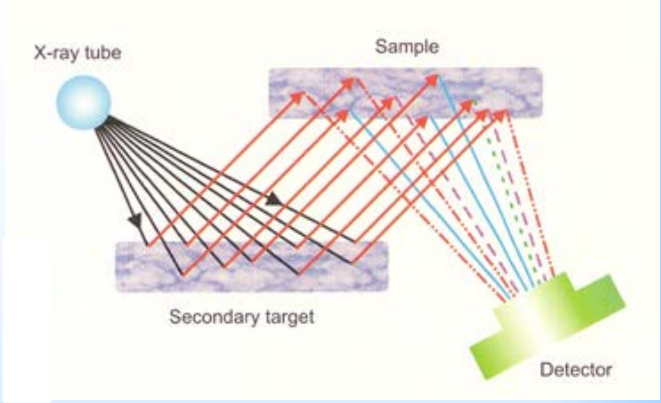
Secondary targets

- Primary beam excites K lines of the secondary target, which excite the sample.
- Excitation is at target K lines, so background is greatly reduced
- Inefficiency of the process requires much higher X-ray tube power.

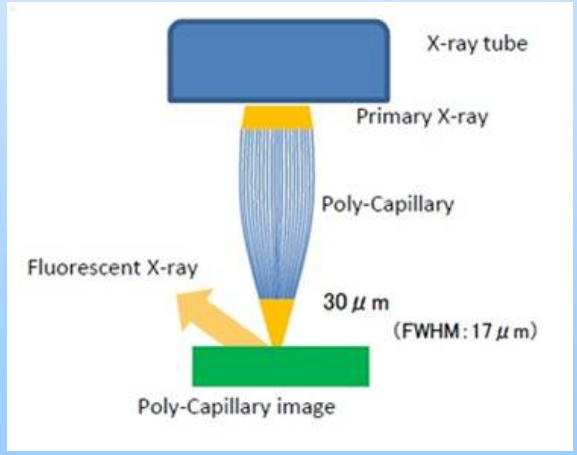


Capillary Optics

- X-rays cannot be reflected or refracted but capillary optics can be used to concentrate
- Increases signal intensity

Crystal Optics

- Crystals are used to diffract primary beam
- Only transmits in a narrow energy range so reduces background



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Electron beams

- Also excite characteristic X-rays. Used in "electron dispersive spectroscopy", related to XRF but different method
- Main advantage is that electrons have short range in sample so are very effective for measuring low energy X-rays from the lightest elements

Particle induced X-ray emissions (PIXE)

- Alpha particles, protons, and other energetic particles ionize materials which thus give off characteristic X-rays
- Can be used for X-ray spectroscopy (though not strictly XRF)

Synchrotrons

- Extremely bright source of X-rays so permit sophisticated analysis
- Only available at these large research facilities

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Mini-X and Mini-X OEM

- 50 kVp, 4W, small tube with USB control
- Au, Ag & Rh anodes, 10W tubes available
- Easy to attach custom collimator

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Additional Advice

Novice users often underestimate the important of optimizing the excitation source. They assume that, if they are exciting X-rays, the spectrometer is configured properly, and the analysis software has been set right, they will get good results.

One will, indeed, be able to see a signal. But the precision, accuracy, and detection limits will be much worse than they anticipate unless they are careful with the excitation conditions.

Rules of thumb:

- Excitation energy must be 2 keV above line you are measuring to see anything
- Best excitation is usually at a kV 2-3 times the energy of the line
- Best filter will use a K edge about 1.5 time energy of the line

40 kVp with a 10 mil Al filter is "jack of all trades, master of none". It will show all lines but will measure none of them very well.

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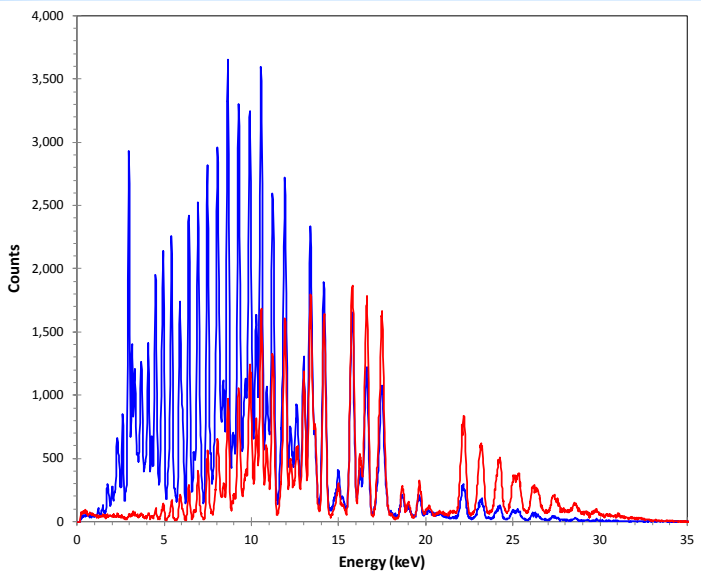
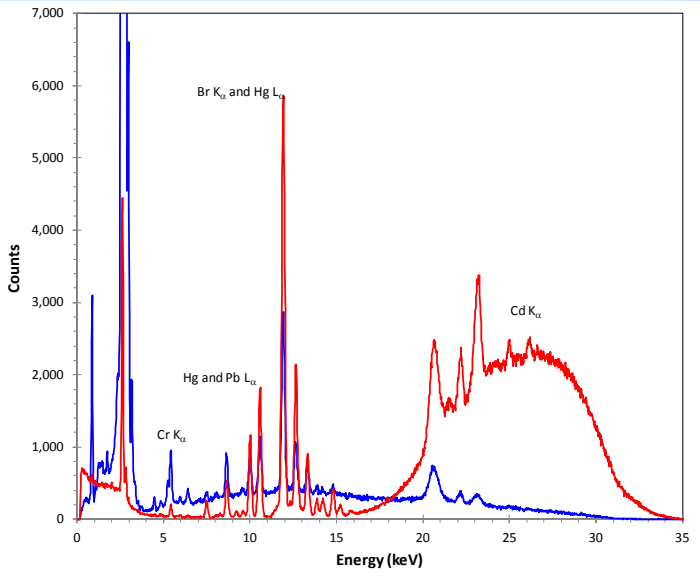
Filters and kVp are key

- Plots below show same targets with different filters
- Blue: No filter, good signal at low energy
- Red: Filter gives much better signal to background





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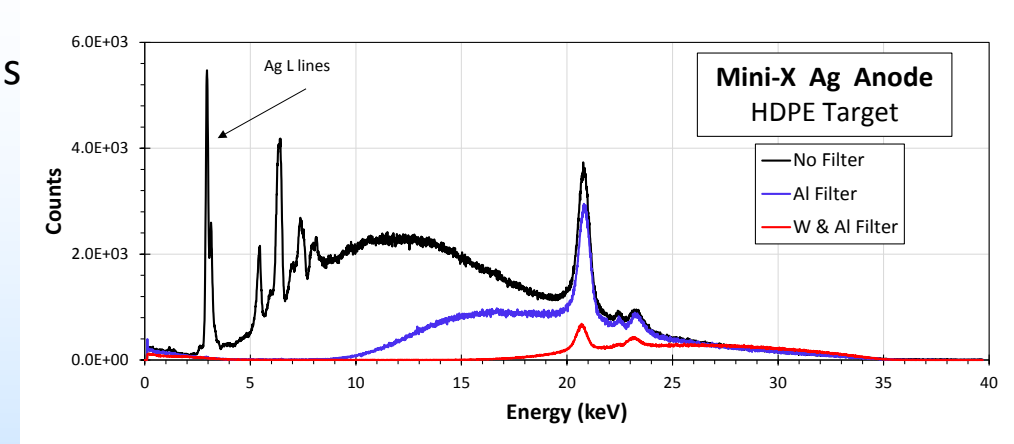
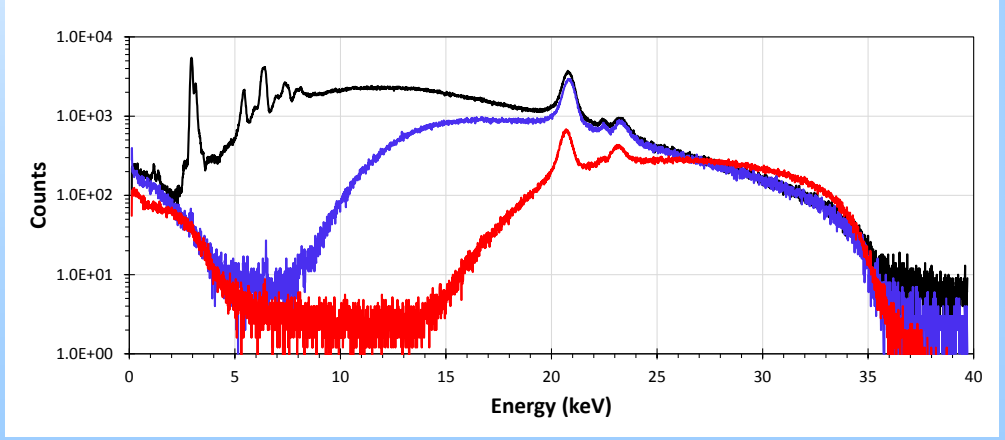
Filters and kVp are key

- Plots here show three options
- W/Al filter reduces 5 to 10 keV background x500
- X-rays penetrate deeply so little signal < 5 keV
- No filter only best at the lowest energies





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Filters and kVp are key

- Different filter and kVp setting gave best results for a different range of elements

	No Filter 15 kVp		No Filter 25 kVp		No Filter 35 kVp		No Filter 45 kVp		Filter 35 kVp		Filter 45 kVp	
	σ	S/B	σ	S/B	σ	S/B	σ	S/B	σ	S/B	σ	S/B
Ti	2.05%	2.80	2.44%	2.94	2.86%	2.77	2.82%	3.08	15.47%	1.56	21.71%	0.78
Fe	1.55%	5.48	1.68%	6.21	1.86%	6.31	1.98%	6.05	5.62%	6.24	6.18%	4.61
Ni	1.63%	4.97	1.58%	6.02	1.65%	6.69	1.76%	6.42	3.51%	8.32	3.68%	7.47
Zn	2.50%	1.98	8.12%	0.46	2.78%	1.73	5.11%	0.86	5.13%	1.97	4.47%	2.33
Ga	2.35%	2.16	1.73%	2.70	1.73%	2.79	1.82%	2.64	2.95%	2.91	3.02%	2.79
Ge	2.88%	1.92	1.83%	2.53	1.76%	2.61	1.78%	2.59	2.76%	2.65	2.81%	2.62
As	17.81%	0.28	1.86%	2.64	1.75%	2.76	1.73%	2.81	2.40%	3.17	2.40%	3.18
Se	6.93%	1.09	2.24%	3.27	2.87%	2.23	1.95%	3.84	2.79%	4.30	3.20%	3.39
Br	24.28%	0.30	1.93%	3.96	1.92%	3.59	1.72%	4.01	2.14%	4.76	2.31%	4.10
Rb			2.51%	3.19	1.99%	3.61	1.77%	4.06	1.90%	4.98	2.03%	4.36
Sr			2.35%	5.64	1.89%	6.94	1.52%	9.95	1.67%	13.53	1.97%	10.07
Zr			3.40%	3.21	2.44%	4.07	1.70%	6.42	1.64%	8.56	2.11%	5.58
Mo			4.12%	3.61	2.12%	7.78	1.65%	11.29	1.43%	19.42	1.66%	14.41
Ag					5.29%	3.13	4.16%	4.28	2.32%	6.16	2.52%	6.27
Sn					17.95%	0.81	4.40%	4.55	7.80%	0.91	4.17%	2.19