



# Handheld XRF Measurements of Fluorine

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# Agenda



- Introduction
- Conventional handheld XRF (HHXRF) technology
- Light element analysis HHXRF technology
  - Direct control of excitation conditions
  - Optional helium atmosphere
  - Optimized front-end geometry
  - Optimized detector
  - Live spectral analysis software
- Fluorine analysis HHXRF application examples
  - Semiconductor
  - Environmental
- Q & A



# Welcome

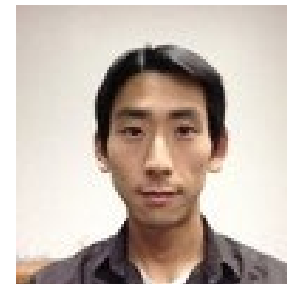


## Speakers

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## Handheld XRF Technology

- What does it do?
- How does it work?

# Handheld XRF Technology



## Periodic Table of Elements and X-ray Energies

[www.bruker.com/hhxf](http://www.bruker.com/hhxf)

1 H 1.01 0.0007 Hydrogen																	2 He 4.00 0.0002 Helium	
3 Li 6.94 0.53 Lithium	4 Be 9.01 1.85 Beryllium Ka 0.108																	10 Ne 20.18 0.0002 Neon Ka 0.849
11 Na 22.99 0.97 Sodium Ka 1.040	12 Mg 24.31 1.74 Magnesium Ka 1.254																	18 Ar 39.95 0.0002 Argon Ka 2.958
19 K 39.10 0.86 Potassium Ka 3.314	20 Ca 40.08 1.54 Calcium Ka 3.692 Lα 0.341	21 Sc 44.96 2.99 Scandium	22 Ti 47.87 4.54 Titanium Ka 4.512 Lα 0.452	23 V 50.94 6.11 Vanadium Ka 4.953 Lα 0.510	24 Cr 52.00 7.15 Chromium Ka 5.415 Lα 0.572	25 Mn 54.94 7.44 Manganese Ka 5.900 Lα 0.637	26 Fe 55.85 7.87 Iron Ka 6.405 Lα 0.705	27 Co 58.93 8.86 Cobalt Ka 6.931 Lα 0.775	28 Ni 58.69 8.91 Nickel Ka 7.480 Lα 0.849	29 Cu 63.55 8.93 Copper Ka 8.046 Lα 0.928	30 Zn 65.38 7.13 Zinc Ka 8.637 Lα 1.012	31 Ga 69.72 5.91 Gallium Ka 9.251 Lα 1.098	32 Ge 72.64 5.32 Germanium Ka 9.886 Lα 1.188	33 As 74.92 5.78 Arsenic Ka 10.543 Lα 1.282	34 Se 78.96 4.81 Selenium Ka 11.224 Lα 1.379	35 Br 79.90 3.12 Bromine Ka 11.924 Lα 1.481	36 Kr 83.80 0.0004 Krypton Ka 12.648 Lα 1.585	
37 Rb 85.47 1.53 Rubidium Ka 13.396 Lα 1.692	38 Sr 87.62 2.64 Strontium Ka 14.165 Lα 1.806	39 Y 88.91 4.47 Yttrium Ka 14.958 Lα 1.924	40 Zr 91.22 6.51 Zirconium Ka 15.775 Lα 2.044	41 Nb 92.91 8.57 Niobium Ka 16.615 Lα 2.169	42 Mo 95.94 10.22 Molybdenum Ka 17.480 Lα 2.292	43 Tc 98 11.50 Technetium	44 Ru 101.07 12.37 Ruthenium Ka 18.279 Lα 2.423	45 Rh 102.91 12.41 Rhodium Ka 19.216 Lα 2.558	46 Pd 106.42 12.02 Palladium Ka 20.177 Lα 2.838	47 Ag 107.87 10.50 Silver Ka 22.163 Lα 2.983	48 Cd 112.41 8.69 Cadmium Ka 23.173 Lα 3.133	49 In 114.82 7.31 Indium Ka 24.210 Lα 3.286	50 Sn 118.71 7.29 Tin Ka 25.271 Lα 3.444	51 Sb 121.76 6.69 Antimony Ka 26.359 Lα 3.604	52 Te 127.60 6.23 Tellurium Ka 27.473 Lα 3.768	53 I 126.90 4.93 Iodine Ka 28.612 Lα 3.938	54 Xe 131.29 0.0006 Xenon Ka 29.775 Lα 4.110	
55 Cs 132.91 1.87 Cesium Ka 30.973 Lα 4.285	56 Ba 137.33 3.59 Barium Ka 32.194 Lα 4.466	57 La 138.91 6.15 Lanthanum Ka 33.442 Lα 4.647	58 Ce 140.12 6.77 Cerium Ka 34.059 Lα 4.839 Ma 0.684	59 Pr 140.91 6.77 Praseodymium Ka 34.953 Lα 5.035 Ma 0.927	60 Nd 144.24 7.01 Neodymium Ka 35.228 Lα 5.228 Ma 0.979	61 Pm 144.91 7.26 Promethium Ka 35.432 Lα 5.432 Ma 1.023	62 Sm 150.36 7.52 Samarium Ka 35.633 Lα 5.633 Ma 1.078	63 Eu 151.96 5.24 Europium Ka 35.849 Lα 5.849 Ma 1.131	64 Gd 157.25 7.90 Gadolinium Ka 36.053 Lα 6.053 Ma 1.181	65 Tb 158.93 8.23 Terbium Ka 36.273 Lα 6.273 Ma 1.240	66 Dy 162.50 8.55 Dysprosium Ka 36.498 Lα 6.498 Ma 1.293	67 Ho 164.93 8.80 Holmium Ka 36.720 Lα 6.720 Ma 1.349	68 Er 167.26 9.07 Erbium Ka 36.949 Lα 6.949 Ma 1.404	69 Tm 168.93 9.32 Thulium Ka 37.180 Lα 7.180 Ma 1.462	70 Yb 173.04 6.97 Ytterbium Ka 37.416 Lα 7.416 Ma 1.526	71 Lu 174.47 9.84 Lutetium Ka 37.655 Lα 7.655 Ma 1.580		
87 Fr (223) 1.87 Francium Lα 12.031 Ma 2.732	88 Ra (226) 5.50 Radium Lα 12.339 Ma 2.806	89 Ac (227) 10.07 Actinium Lα 12.652 Ma 2.900	90 Th 232.04 11.72 Thorium Lα 12.968 Ma 2.996	91 Pa 231.04 15.37 Protactinium Lα 13.291 Ma 3.082	92 U 238.03 18.95 Uranium Lα 13.614 Ma 3.171	93 Np (237) 20.45 Neptunium Lα 13.946 Ma 3.250	94 Pu (244) 19.84 Plutonium Lα 14.282 Ma 3.339	95 Am (243) 13.69 Americium Lα 14.620 Ma 3.438	96 Cm (247) 13.51 Curium Lα 14.779 Ma 3.531	97 Bk (247) 14.79 Berkelium	98 Cf (251) 15.1 Californium	99 Es (252) 13.5 Einsteinium	100 Fm (257) 15.1 Fermium	101 Md (258) 15.1 Mendelevium	102 No (259) 15.1 Nobelium	103 Lr (262) 15.1 Lawrencium		

Atomic number	Atomic weight
35	79.90
Br	3.12
Bromine	Density (g/cm <sup>3</sup> )
	Symbol
	Element name
	Energy (keV)
	Spectral line



Conventional Handheld XRF Technology

Innovation with Integrity

Handheld XRF



# Handheld XRF Applications



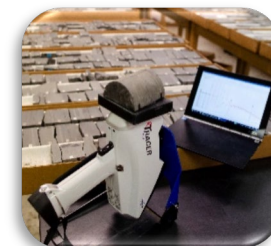
Alloy PMI and quality control



Scrap metal recycling



Precious metals



Geology, mining & exploration



Applications & product development



Environmental



Research & education



Food safety & agriculture



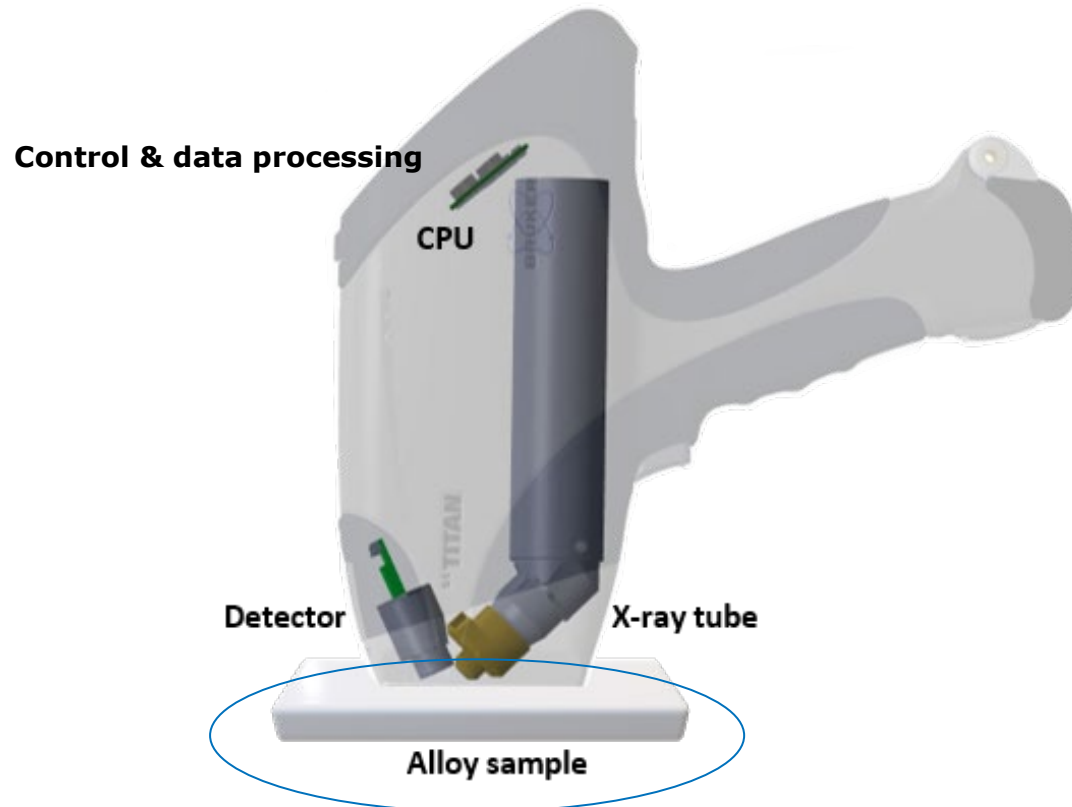
Conservation & archaeology



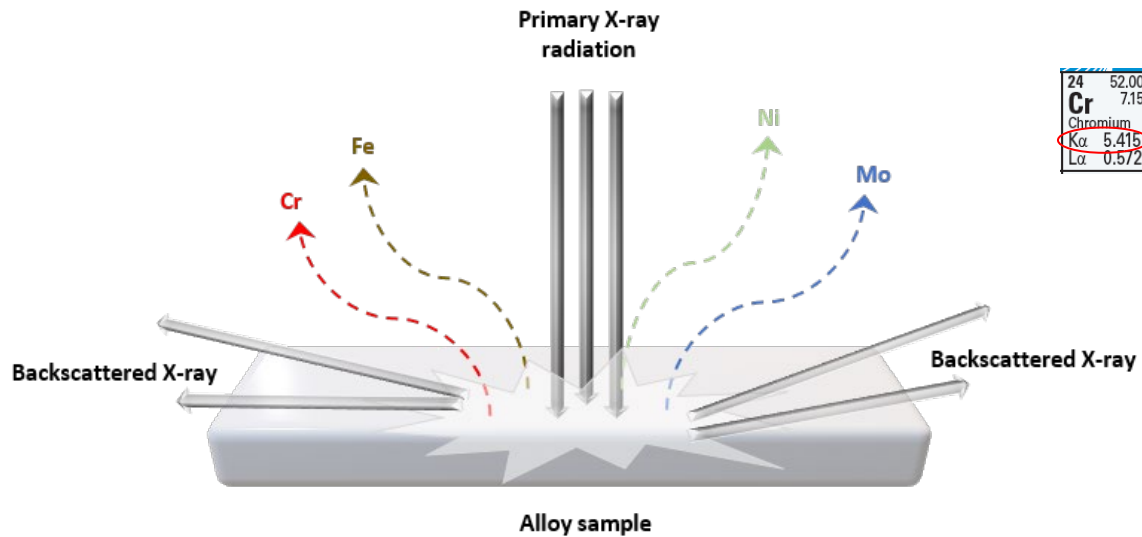
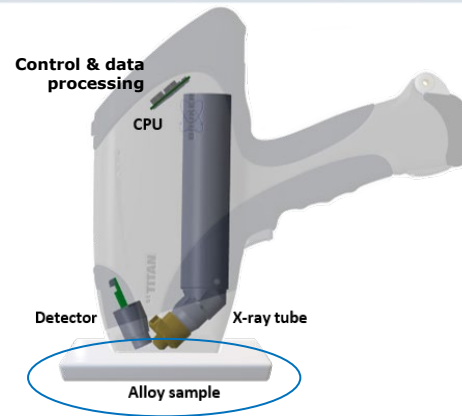
RoHS & consumer safety

Multiple HXRF Applications

# HHXRF Technology



# HHXRF Technology



24	52.00	26	55.85	28	58.69	42	95.94
<b>Cr</b>	7.15	<b>Fe</b>	7.87	<b>Ni</b>	8.91	<b>Mo</b>	10.22
Chromium		Iron		Nickel		Molybdenum	
K $\alpha$ 5.415		K $\alpha$ 6.405		K $\alpha$ 7.480		K $\alpha$ 17.480	
L $\alpha$ 0.572		L $\alpha$ 0.705		L $\alpha$ 0.849		L $\alpha$ 2.292	



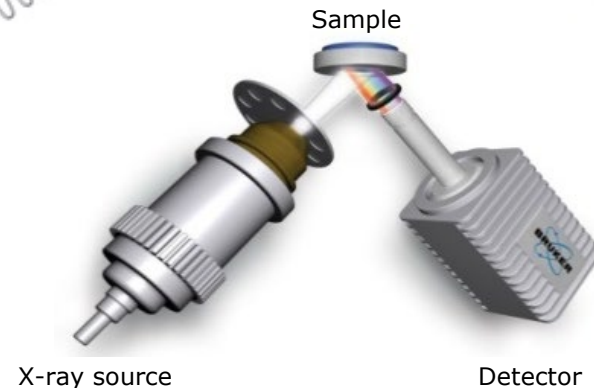
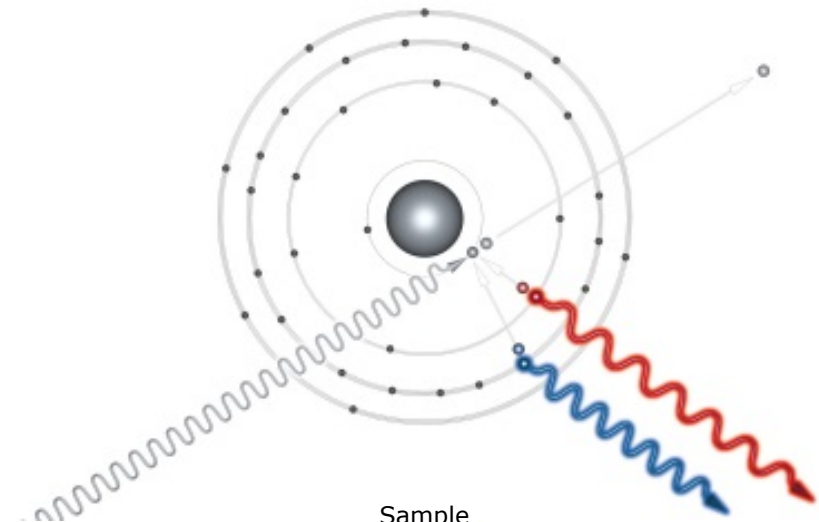
# HHXRF Technology

## Energy Dispersive (ED) X-ray Fluorescence (XRF) Spectroscopy

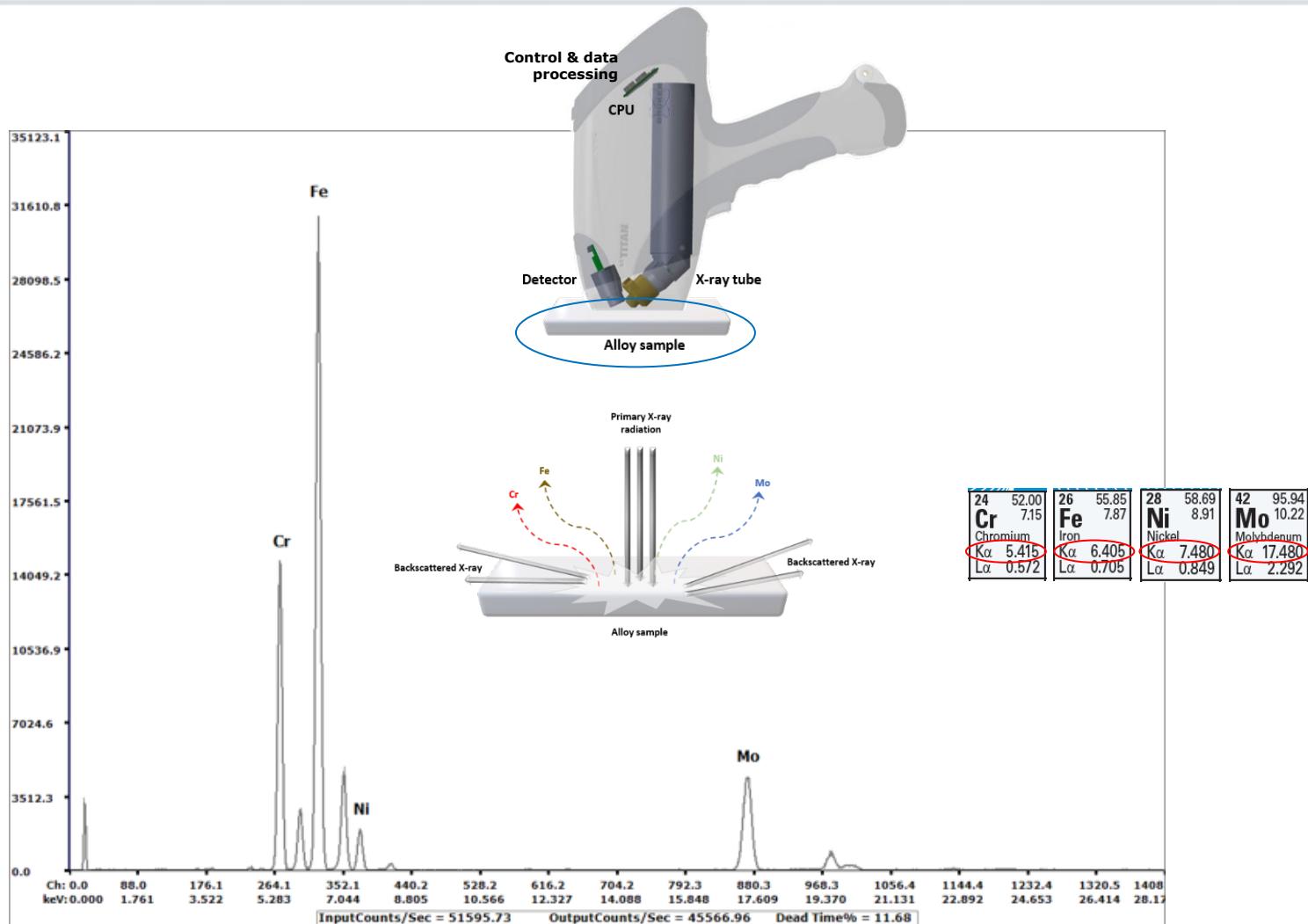
- Energy from an EDXRF source aimed at a sample can eject the elemental atoms' inner orbital electrons
- Outer electrons move into the voids to regain stability
- While moving in, the outer electrons generate energy characteristic of the element
- These characteristic energies are the fluorescent X-rays of the element

Atomic number	35	79.90	Atomic weight
Symbol	Br	3.12	Density (g/cm <sup>3</sup> )
Element name	Bromine		
Energy (keV)	K <sub>α</sub> 11.924		
Spectral line	L <sub>α</sub> 1.481		

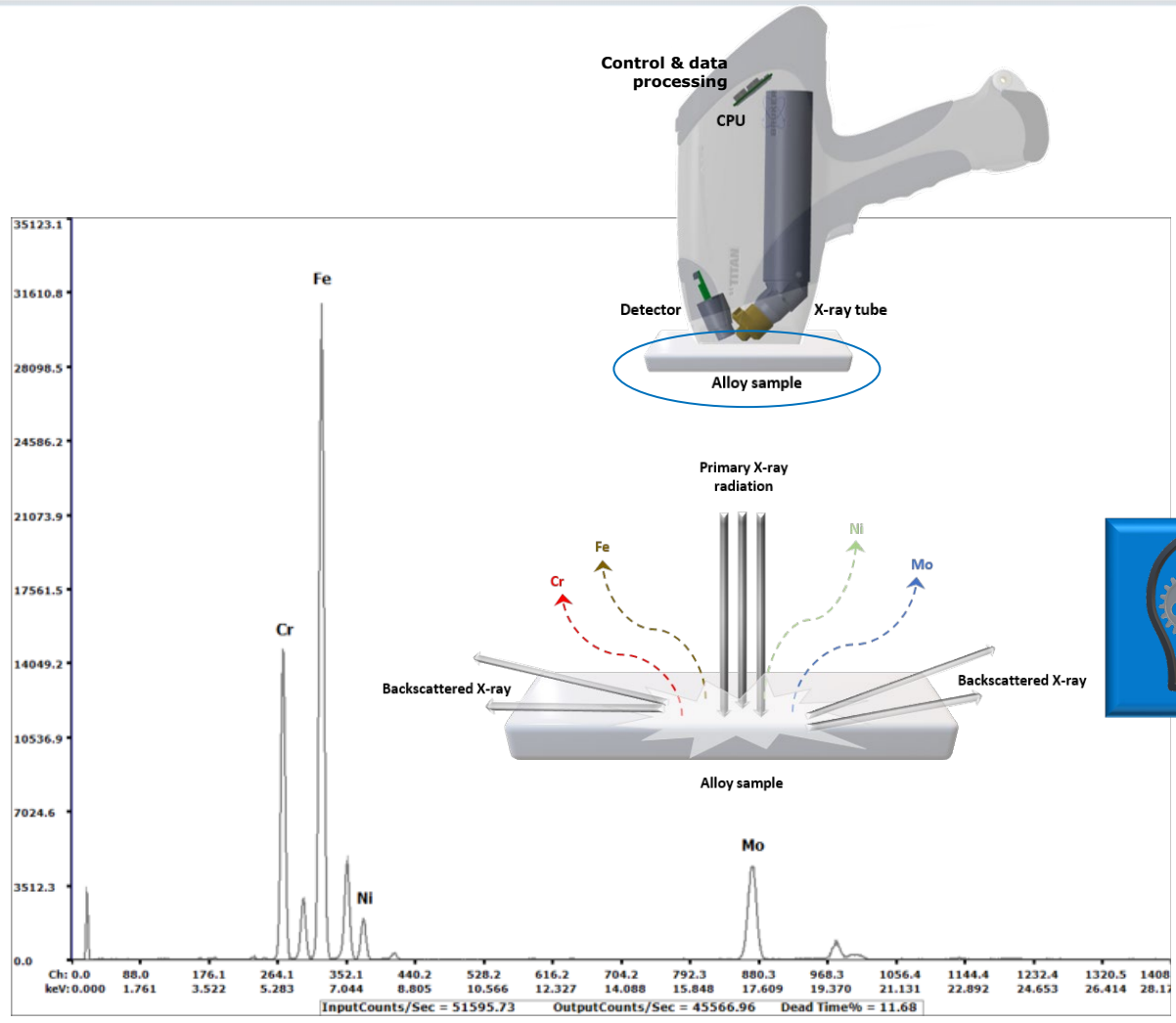
Electron orbitals of element's atom



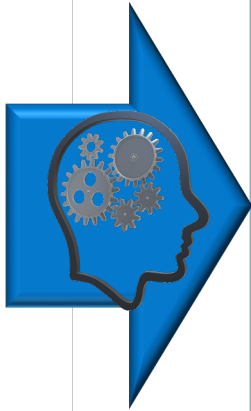
# HHXRF Technology



# HHXRF Technology



24	52.00	26	55.85	28	58.69	42	95.94
<b>Cr</b>	7.15	<b>Fe</b>	7.87	<b>Ni</b>	8.91	<b>Mo</b>	10.22
Chromium		Iron		Nickel		Molybdenum	
K $\alpha$ 5.415		K $\alpha$ 6.405		K $\alpha$ 7.480		K $\alpha$ 17.480	
L $\alpha$ 0.572		L $\alpha$ 0.705		L $\alpha$ 0.849		L $\alpha$ 2.292	



<b>316SS</b>				
124 Match 9.8 11-24 17:26				
Time 46.0   HiZ SS Fe				
El	Min	%	Max	+/- [*2]
Fe	60.00	70.40	75.00	1.07
Cr	16.00	16.25	18.00	0.45
Ni	10.00	11.40	14.00	0.50
Mo	2.00	1.63	3.00	0.10
Sn		0.21		0.09
Nb		0.11		0.06
Ti	0.00	< LOD	0.00	0.14
Mn	0.00	< LOD	2.00	0.24

Spectrum

## HHXRF light element analysis technology

- How is it different?

# HHXRF light element analysis technology



## Periodic Table of Elements and X-ray Energies

1 H 1.01 Hydrogen 0.0007	2 He 4.00 Helium 0.0002																													
3 Li 6.94 Lithium 0.53	4 Be 9.01 Beryllium 1.85	www.bruker.com														9 F 19.00 Fluorine 0.001	10 Ne 20.18 Neon 0.0002													
11 Na 22.99 Sodium 0.97	12 Mg 24.31 Magnesium 1.74															17 Cl 35.45 Chlorine 2.622	18 Ar 39.95 Argon 0.0002													
13 Al 26.98 Aluminum 1.486	14 Si 28.09 Silicon 2.33	15 P 30.97 Phosphorus 2.010	16 S 32.07 Sulfur 2.309	31 Ga 69.72 Gallium 5.91	32 Ge 72.64 Germanium 5.32	33 As 74.92 Arsenic 5.78	34 Se 78.96 Selenium 4.81	35 Br 79.90 Bromine 3.12	36 Kr 83.80 Krypton 0.0004	49 In 114.82 Indium 7.31	50 Sn 118.71 Tin 7.29	51 Sb 121.76 Antimony 6.69	52 Te 127.60 Tellurium 6.23	53 I 126.90 Iodine 4.93	54 Xe 131.29 Xenon 0.0006															
19 K 39.10 Potassium 0.86	20 Ca 40.08 Calcium 1.54	21 Sc 44.96 Scandium 2.99	22 Ti 47.87 Titanium 4.54	23 V 50.94 Vanadium 6.11	24 Cr 52.00 Chromium 7.15	25 Mn 54.94 Manganese 7.44	26 Fe 55.85 Iron 7.87	27 Co 58.93 Cobalt 8.86	28 Ni 58.69 Nickel 8.91	29 Cu 63.55 Copper 8.93	30 Zn 65.38 Zinc 7.13	41 Nb 92.91 Niobium 10.22	42 Mo 95.94 Molybdenum 10.22	43 Tc 115.00 Technetium 11.50	44 Ru 101.07 Ruthenium 12.37	45 Rh 102.91 Rhodium 12.41	46 Pd 106.42 Palladium 12.02	47 Ag 107.87 Silver 10.50	48 Cd 112.41 Cadmium 8.69	61 Pm 144.24 Promethium 7.01	62 Sm 150.36 Samarium 7.52	63 Eu 151.96 Europium 5.24	64 Gd 157.25 Gadolinium 7.90	65 Tb 158.93 Terbium 8.23	66 Dy 162.50 Dysprosium 8.55	67 Ho 164.93 Holmium 8.80	68 Er 167.26 Erbium 9.07	69 Tm 168.93 Thulium 9.32	70 Yb 173.04 Ytterbium 6.97	71 Lu 174.47 Lutetium 9.84
37 Rb 85.47 Rubidium 1.53	38 Sr 87.62 Strontium 2.64	39 Y 88.91 Yttrium 4.47	40 Zr 91.22 Zirconium 6.51	41 Nb 92.91 Niobium 8.57	42 Mo 95.94 Molybdenum 10.22	43 Tc 115.00 Technetium 11.50	44 Ru 101.07 Ruthenium 12.37	45 Rh 102.91 Rhodium 12.41	46 Pd 106.42 Palladium 12.02	47 Ag 107.87 Silver 10.50	48 Cd 112.41 Cadmium 8.69	72 Hf 178.49 Hafnium 6.15	73 Ta 180.95 Tantalum 6.65	74 W 183.84 Tungsten 19.25	75 Re 186.21 Rhenium 21.02	76 Os 190.23 Osmium 22.65	77 Ir 192.22 Iridium 21.46	78 Pt 195.08 Platinum 21.46	79 Au 196.97 Gold 19.28	80 Hg 200.59 Mercury 13.53	81 Tl 204.37 Thallium 11.85	82 Pb 207.20 Lead 10.55	83 Bi 208.98 Bismuth 9.81	84 Po (209) Polonium 9.32	85 At (210) Astatine 7.00	86 Rn (222) Radon 0.01				
55 Cs 132.91 Cesium 1.87	56 Ba 137.33 Barium 3.59	57 La 138.91 Lanthanum 6.15	58 Ce 140.12 Cerium 6.77	59 Pr 140.91 Praseodymium 6.77	60 Nd 144.24 Neodymium 7.01	61 Pm (145) Promethium 7.26	62 Sm 150.36 Samarium 7.52	63 Eu 151.96 Europium 5.24	64 Gd 157.25 Gadolinium 7.90	65 Tb 158.93 Terbium 8.23	66 Dy 162.50 Dysprosium 8.55	87 Fr (223) Francium 1.87	88 Ra (226) Radium 5.50	89 Ac (227) Actinium 10.07	90 Th 232.04 Thorium 11.72	91 Pa 231.04 Protactinium 15.37	92 U 238.03 Uranium 18.95	93 Np (237) Neptunium 20.45	94 Pu (244) Plutonium 19.84	95 Am (243) Americium 13.69	96 Cm (247) Curium 13.51	97 Bk (247) Berkelium 14.79	98 Cf (251) Californium 15.1	99 Es (252) Einsteinium 13.5	100 Fm (257) Fermium 10.2	101 Md (258) Mendelevium 10.2	102 No (259) Nobelium 10.2	103 Lr (262) Lawrencium 10.2		
Atomic number 35 79.90 Atomic weight Br 3.12 Density (g/cm <sup>3</sup> ) Bromine Symbol Kα 11.924 Element name Lα 1.481 Energy (keV) Spectral line																														



Light Element Handheld XRF Technology

Innovation with Integrity

Handheld XRF

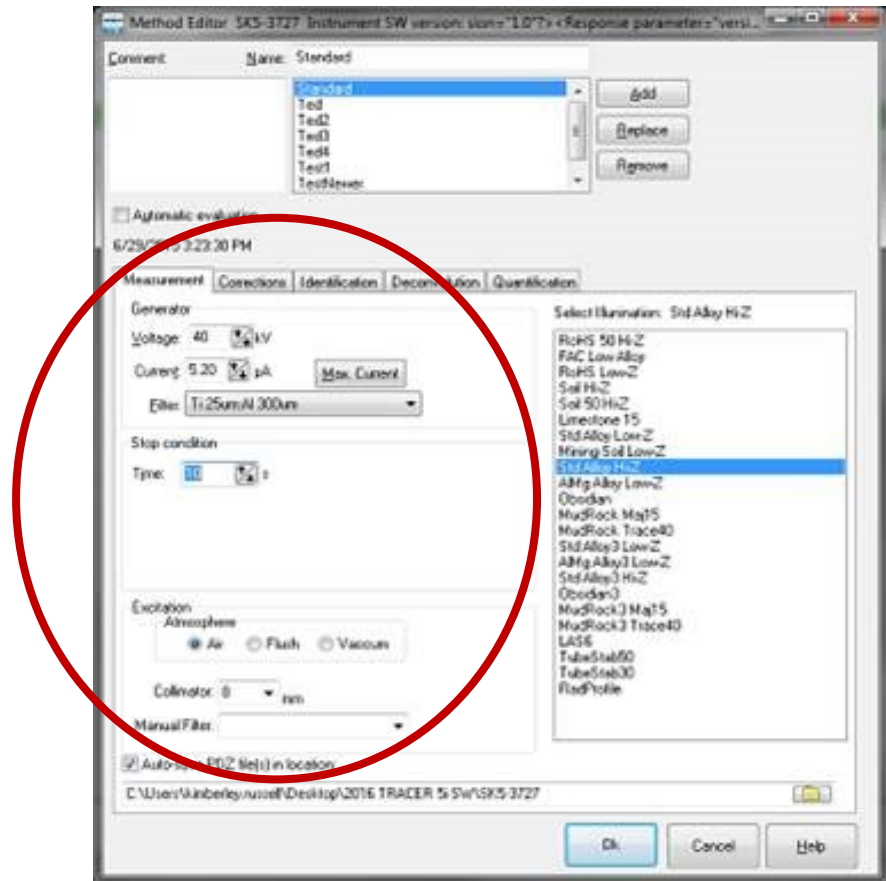


# HHXRF light element analysis technology

Direct user control of settings to optimize HHXRF measurements



HH Screen for user control of measurement settings



PC screen for user control of measurement settings

# HHXRF light element analysis technology

## Optional helium atmosphere for light element detection capability

- Fluorine is a low atomic number element with an XRF measurement depth of about  $1\mu\text{m}$ , a very weak signal at  $0.677\text{ keV}$ , and is absorbed by all materials
- To get a strong enough XRF signal, air and other materials absorbing the fluorine signal between the sample and detector need to be removed
- The instrument nose needs to be purged with helium which absorbs the fluorine signal much less than air ( $\text{N}_2$ ,  $\text{O}_2$ ,  $\text{Ar}$ ) does
- In addition to purging the nose, its protective window needs to be removed to provide a non-obstructive path for the F signal



# HHXRF light element analysis technology

## Optional helium atmosphere for light element detection

**TRACER 5g**

	LOD (PPM)	Sens (counts/PPM)
Na	N/A	N/A
Mg	932	0.12
Al	462	0.59
Ca	25	13.13
Fe	46	29.36

Air atmosphere and  
3 µm prolene window

**TRACER 5g**

	LOD (PPM)	Sens (counts/PPM)
Na	548	0.31
Mg	155	1.44
Al	165	3.36
Ca	27	14.00
Fe	50	26.40

Lab helium atmosphere and  
3 µm prolene window

**TRACER 5g**

	LOD (PPM)	Sens (counts/PPM)
Na	312	0.62
Mg	122	2.13
Al	134	4.41
Ca	24	14.04
Fe	50	25.87

Lab helium atmosphere and  
no window

**TRACER 5g**

	LOD (PPM)	Sens (counts/PPM)
Na	356	0.52
Mg	127	1.95
Al	143	4.07
Ca	25	14.15
Fe	50	26.36

Balloon helium atmosphere and  
no window



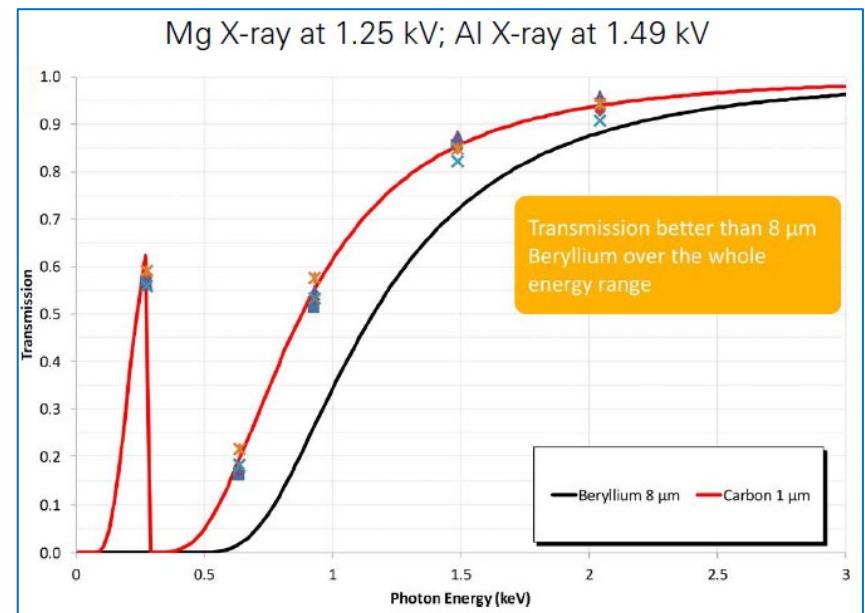
# HHXRF light element analysis technology

## 1 $\mu\text{m}$ Graphene window high resolution SDD for sensitive, clean spectra

- The TRACER 5g incorporates a large area Silicon Drift Detector (SDD). Conventional HHXRF 8  $\mu\text{m}$  beryllium windows have been replaced with 1  $\mu\text{m}$  graphene windows to provide extra sensitivity and clean spectra for light element HHXRF analysis



Large area SDD with graphene window



Transmission of beryllium and graphene windows

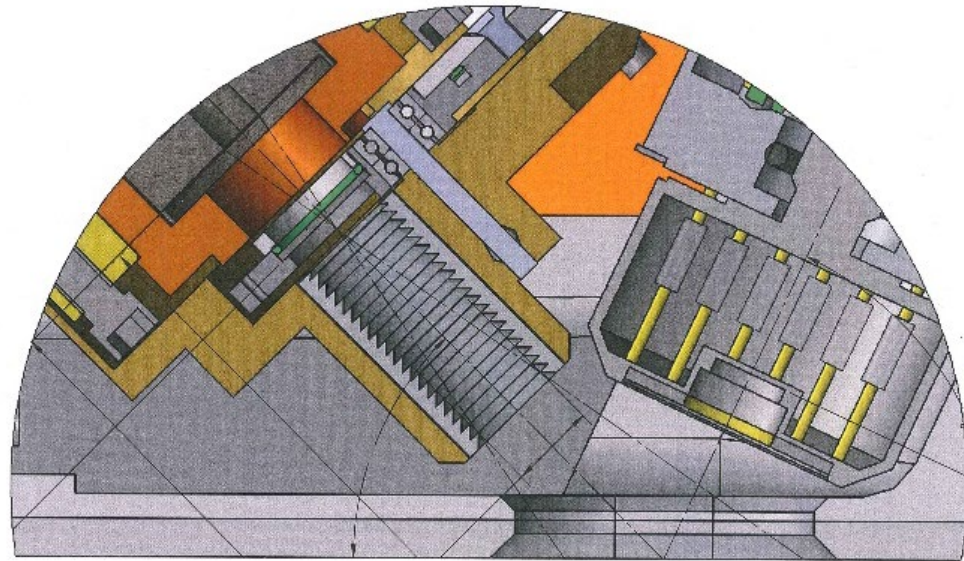


# HHXRF light element analysis technology

## SharpBeam™ front end geometry increases light element sensitivity



- SharpBeam™ front-end geometry maximizes count rate, increases light element sensitivity, provides a well-defined measurement spot, and minimizes detection of X-ray scatter

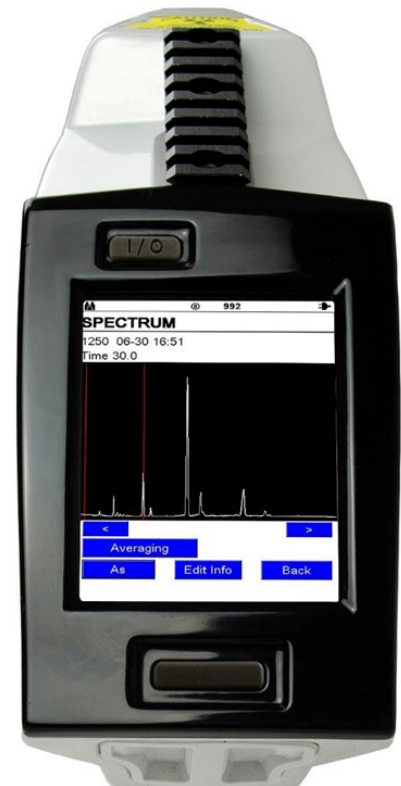
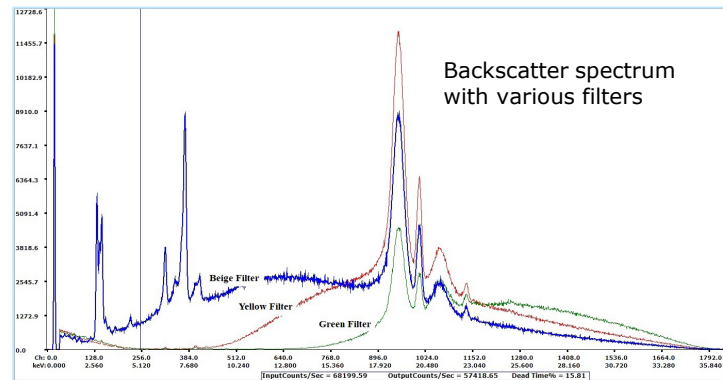
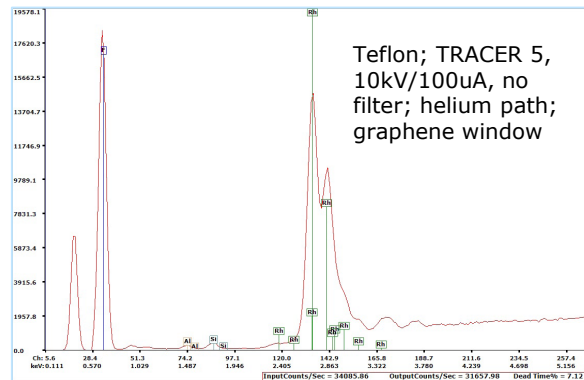
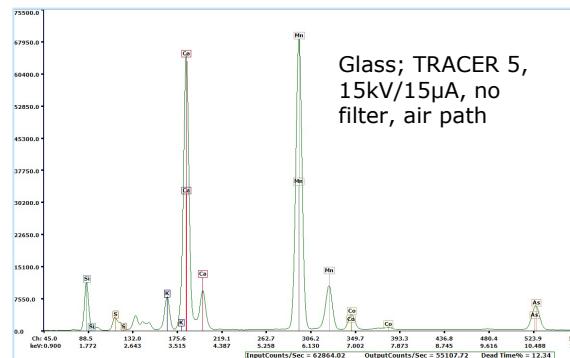
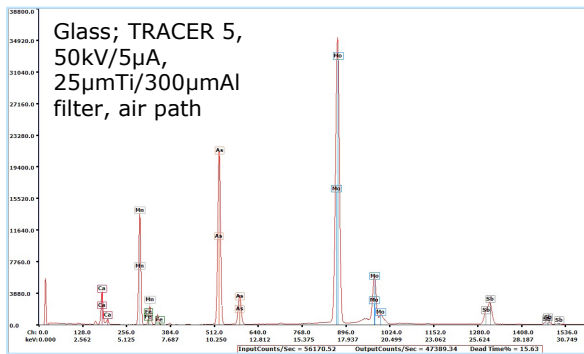




# HHXRF light element analysis technology

## Live spectra analysis software guides measurement optimization of analysis and provides immediate information on sample composition

- Live spectra analysis software helps guide optimum measurement conditions (e.g., kV/ $\mu$ A, filter, path, time), especially important when developing new applications



# HHXRF light element analysis technology



## The TRACER 5g platform enables light element HHXRF analysis of fluorine with:

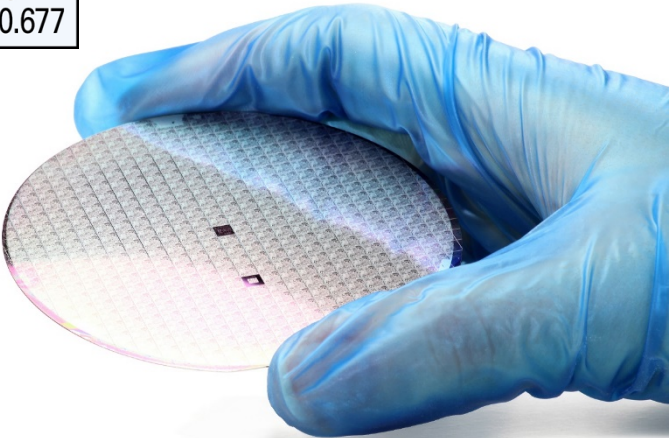
- Direct control of excitation conditions (e.g., kV/ $\mu$ A, filter, atmosphere, time)
- Optional helium atmosphere to minimize absorption of the light element signal
- SmartBeam™ front-end geometry to maximize the count rate
- Large area SDD with a graphene window for extra sensitivity and clean spectra
- Live spectra analysis via handheld and PC software to optimize measurement conditions



# Fluorine analysis HHXRF application examples

# Semiconductor application

9	19.00
F	0.001
Fluorine	
K $\alpha$	0.677



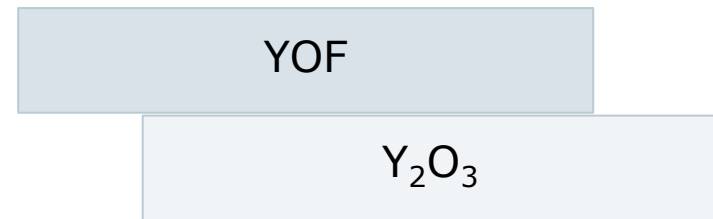
- Fluorine is the most reactive chemical element and the lightest of the halogen group
- Fluorine compounds are used for a variety of semiconductor manufacturing applications including chemical vapor deposition, plasma etching, and cleaning
- A challenge in using fluorine (F) compounds for applications like these is the ability to measure it during manufacture, especially as a coating or residual
- The semiconductor industry needed to find a solution. Handheld XRF was proposed for its portability, in-situ measurement capability, ability to provide fast results, and its nondestructive nature

# Semiconductor application



## Fluorine in semiconductor manufacturing

- Yttrium oxyfluoride (YOF), a plasma-resistant material, is replacing yttrium oxide ( $Y_2O_3$ ) as the protective material in plasma process chambers for the manufacture of 3-D stacking circuits
- It has become critical to detect F to differentiate which Y compound is being used

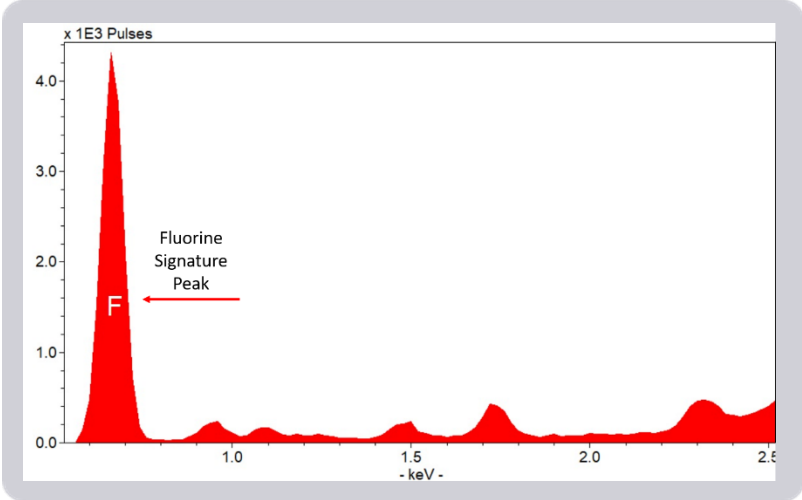




# Semiconductor application

## Feasibility

- Feasibility of measuring the low energy fluorine signature peak with a TRACER 5g was first established using polytetrafluoroethylene (PTFE) tape
- TRACER 5g settings were 10kV/100 $\mu$ A, helium purge, 8 mm spot size, no protective window, 30 seconds measurement time
- A clear fluorine peak at 0.677 keV in PTFE tape was detected by the TRACER 5g

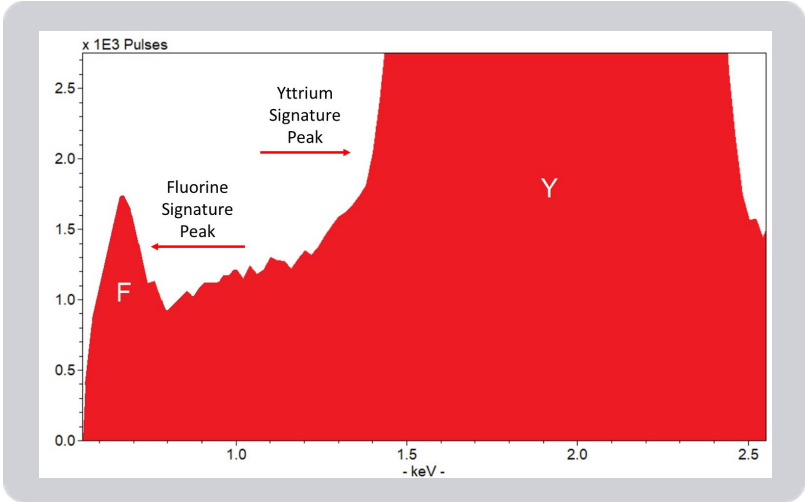
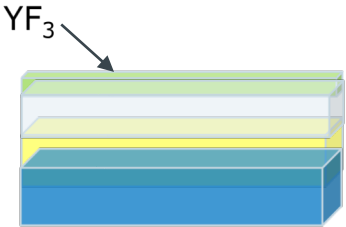


Polytetrafluoroethylene (PTFE) tape measurement with TRACER 5g

# Semiconductor application

## Data

- An yttrium fluoride ( $YF_3$ ) coating used in the semiconductor industry was tested
- TRACER 5g settings were 10kV/100 $\mu$ A, helium purge, 8 mm spot size, no protective window, 30 seconds measurement time
- A clear F peak at 0.677 keV in  $YF_3$  coating was detected by the TRACER 5g making it possible not only to identify the presence of F, but also to distinguish Y-compounds containing F to those without F



Yttrium fluoride ( $YF_3$ ) coating measurement with TRACER 5g

# Environmental application

## Fluorine in ski wax



9	19.00
<b>F</b>	0.001
Fluorine	
K $\alpha$	0.677

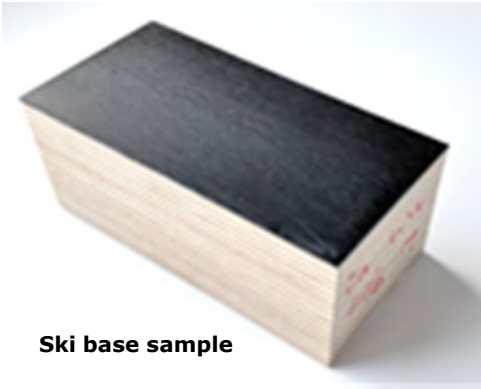


- Persistent Organic Pollutants, or POPs, are referred to as “forever chemicals” because they are almost impossible to get rid of when they get into our water sources and soil
- PFOS (perfluoro-octane-sulphonate) and PFOA (perfluoro-octanoic acid), referred to as fluorine-based PFAS, are in the Stockholm Convention’s POPs list of harmful substances
- PTFE (Polytetrafluoroethylene), a particular type of PFAS, is a common ingredient in ski waxes to help reduce professional skiers race times, even if by seconds
- A challenge in promoting the phasing out of PFAS for ski and snowboard waxes is the ability to ensure fluorinated waxes are not sold at retail stores or used on the slopes

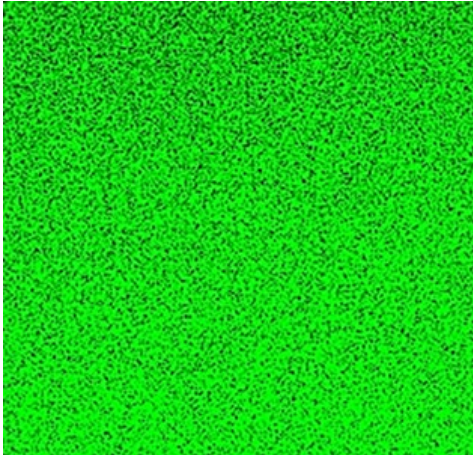
# Environmental application

## Sample presentation

- Five samples with ski bases and different wax compounds were measured to test if fluorinated ski wax samples can be reliably identified from fluorine-free ski wax samples
- All ski base samples were identical, but four of them had different types of fluorinated ski wax and one of the ski base samples had fluorine-free ski wax
- A ski base sample was analyzed with a micro-XRF system to confirm uniformity of sample surface via a F distribution map of a 1 x 1 cm area with 50  $\mu\text{m}$  pixel size



**Ski base sample**



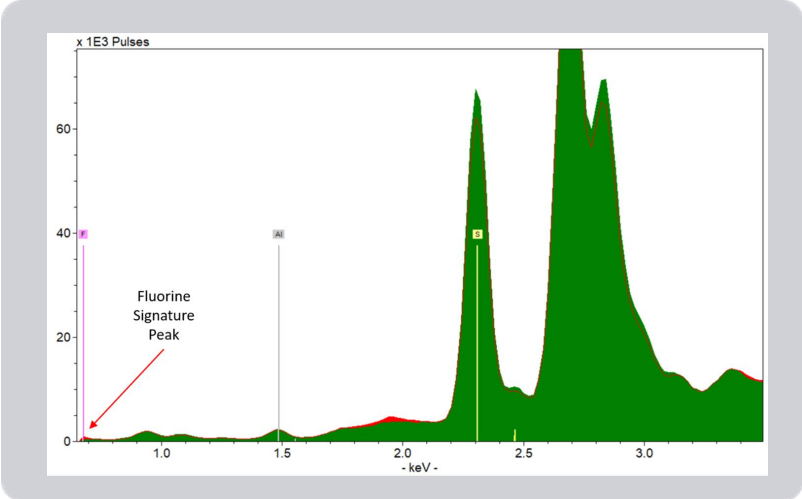
**Micro-XRF was used to measure and map the distribution of F on the ski base to confirm uniformity of sample surface**

# Environmental application

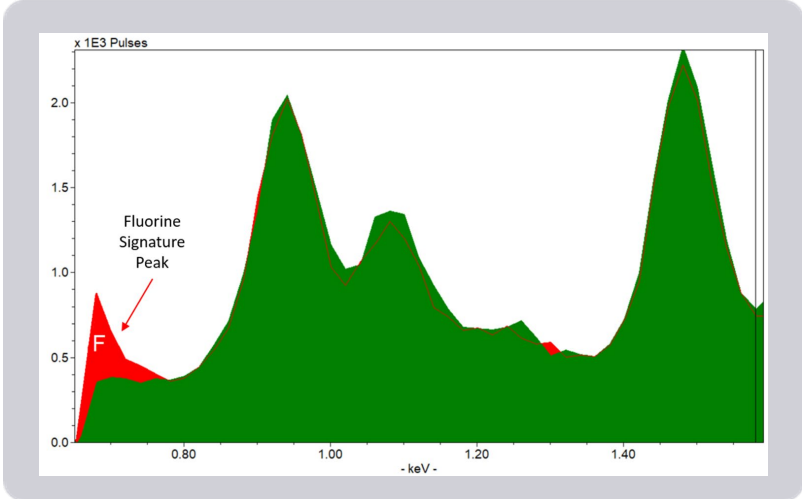
## Data



- TRACER 5g settings were 10kV/180 $\mu$ A, helium purge, 8 mm spot size, no protective window, 180 seconds measurement time
- Test spectra from the TRACER 5g showed a small, but clear F signal in all ski base samples containing fluorine-based ski wax
- In comparison to other elements, such as aluminum (Al), sulfur (S), calcium (Ca) and iron (Fe), the fluorine signal was small
- Magnification showed a clear F peak in all fluorinated ski wax samples which made it possible to clearly distinguish them from fluorine-free ski wax



Fluorine peaks are small compared to other elements present, but when the Y axis is magnified, the peaks become clear.



Tracer 5g measurement can successfully distinguish fluorinated ski wax from fluorine-free ski wax.



# Summary

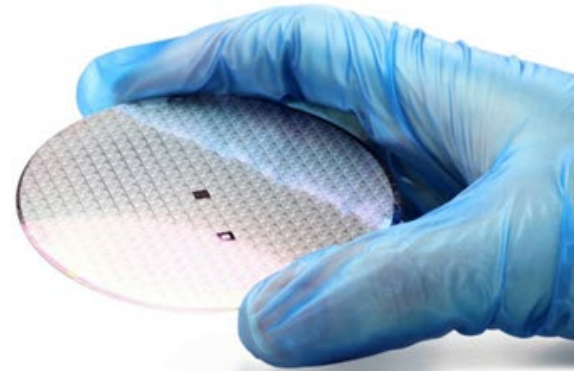


- Fluorine analysis is a challenge for conventional handheld XRF
- The TRACER 5g's combination of direct user control of settings, optional helium atmosphere, SmartBeam™ geometry, large-area SDD with graphene window, and live spectral analysis software enable fluorine analysis with HHXRF
- The TRACER 5g's F signal at 0.677 keV in PTFE tape is clearly visible
- The TRACER 5g's F signal at 0.677 in YF<sub>3</sub> semiconductor coatings is clearly visible which makes it possible to distinguish from fluorine-free coatings
- Magnification of TRACER 5g data shows a clear F signal in fluorinated ski wax samples which makes it possible to distinguish them from fluorine-free ski wax
- The TRACER 5g's fluorine limit of detection (LOD) is highly dependent on the application; it is typically between 1% to 10% depending on measured material and measurement conditions

# Q & A



9	19.00
<b>F</b>	0.001
Fluorine	
K $\alpha$	0.677

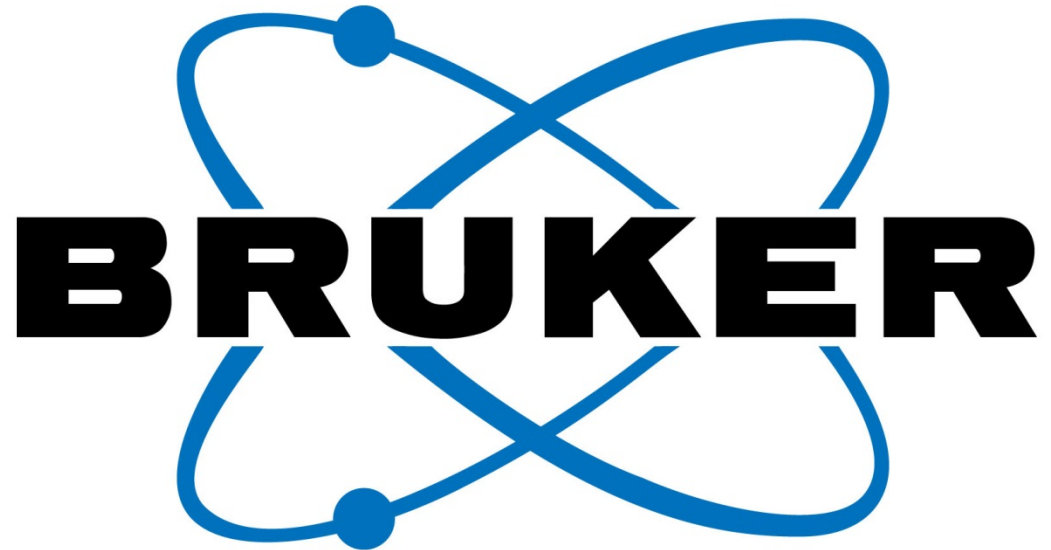


Any Questions?

Please **type in** your questions for our speakers in the **Questions Box** and click **Submit**.

Thank You!





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