Quantification avec EDS et WDS: aspects pratiques

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Sommaire

→ Présentation du système: installation et performance

→ Tests une fois le système installé

Pourquoi WDS?

- Meilleure résolution
- Détection de traces d'élements

Note: typical values, not specifications!

Resolution EDS (MnKa op 124 eV)

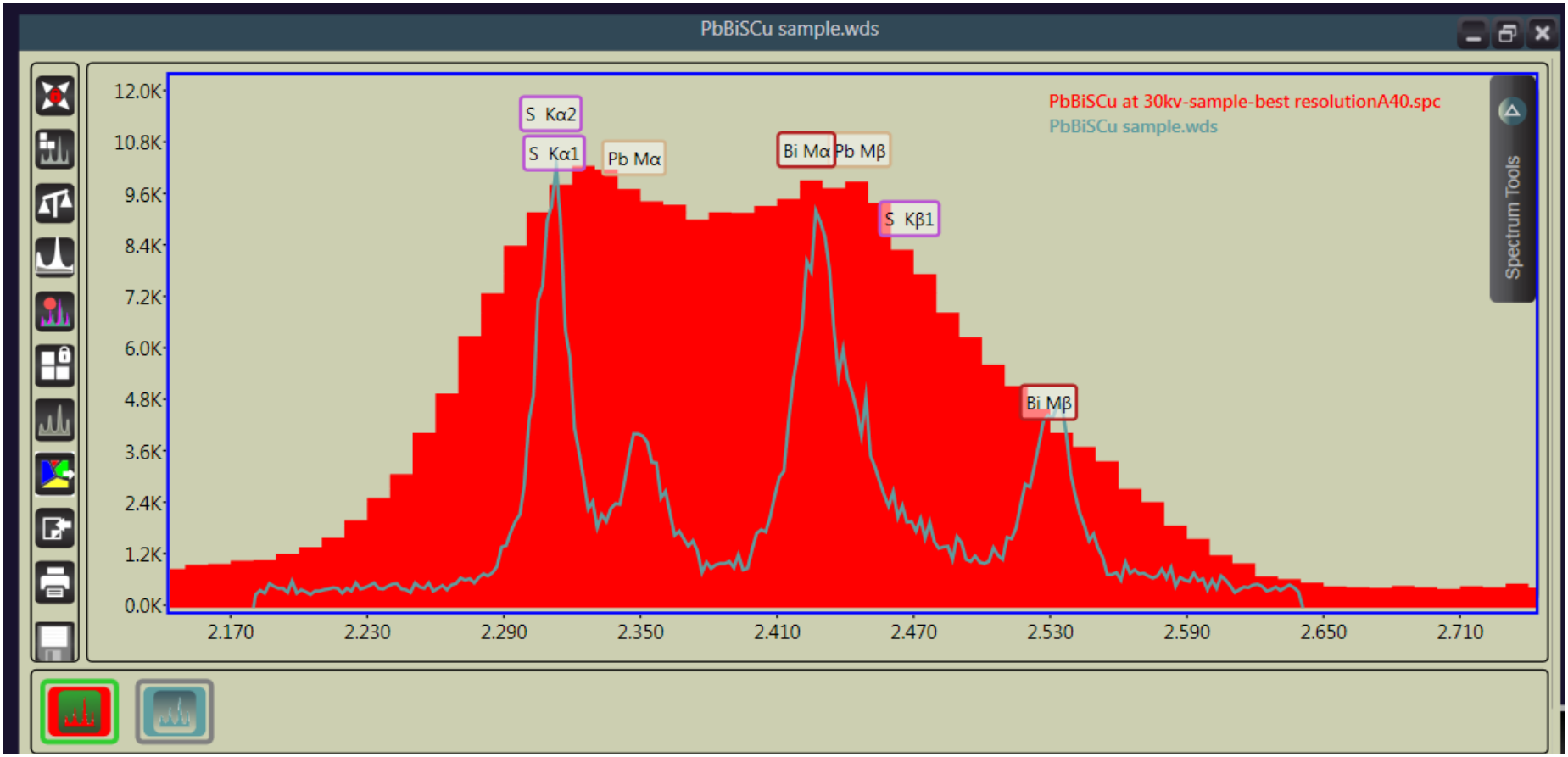
Values in eV

Element	Resolution WDS	Resolution EDS
Bka	15	41
CKa	12	43
NKa	14	45
OKa	15	50
CuLa	26	75
AlKa	24	80
SiKa	5	85
CuKa (TEXS)	58	150

Source: EDAX

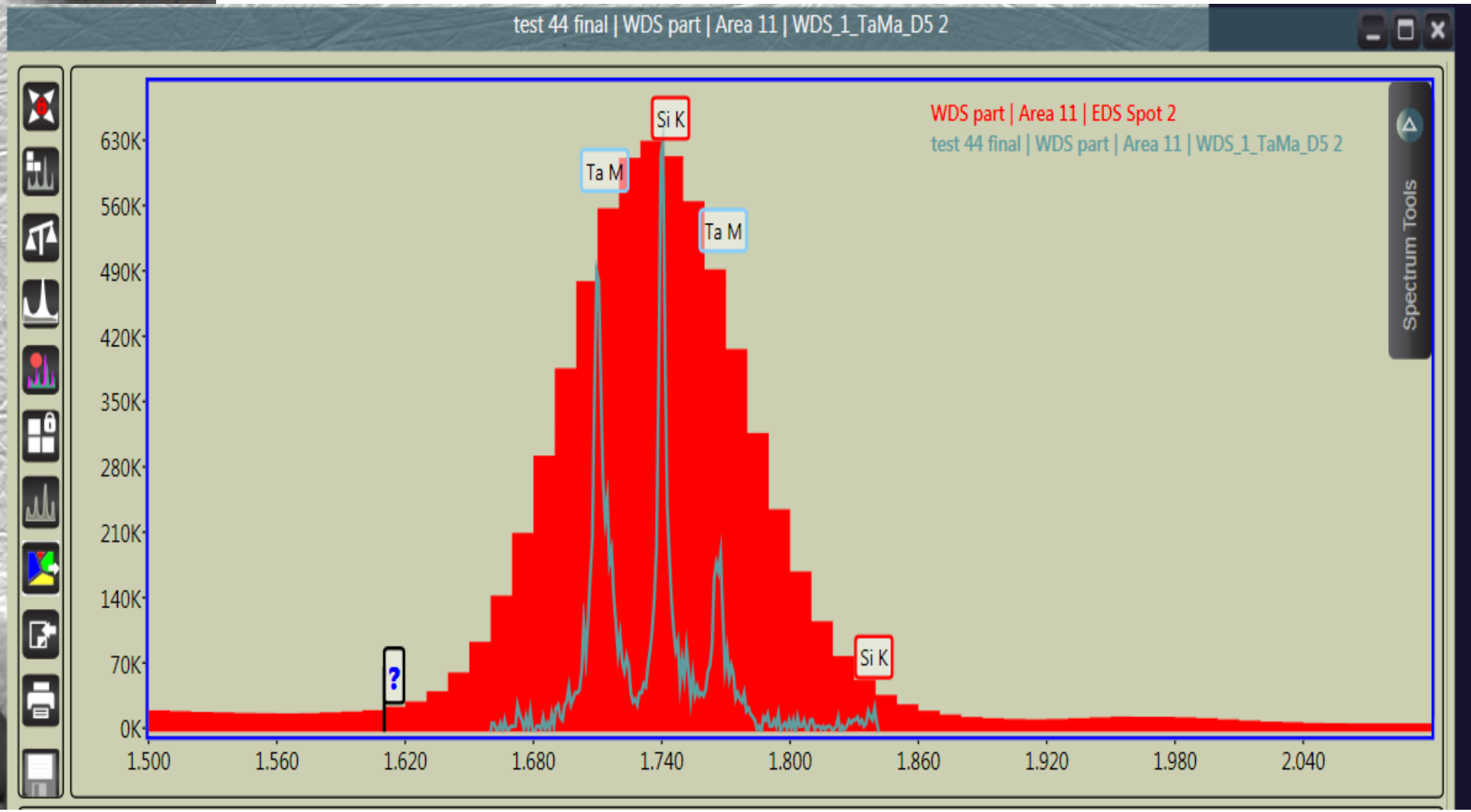
Meilleure résolution

→ Solution pour les chevauchements des pics



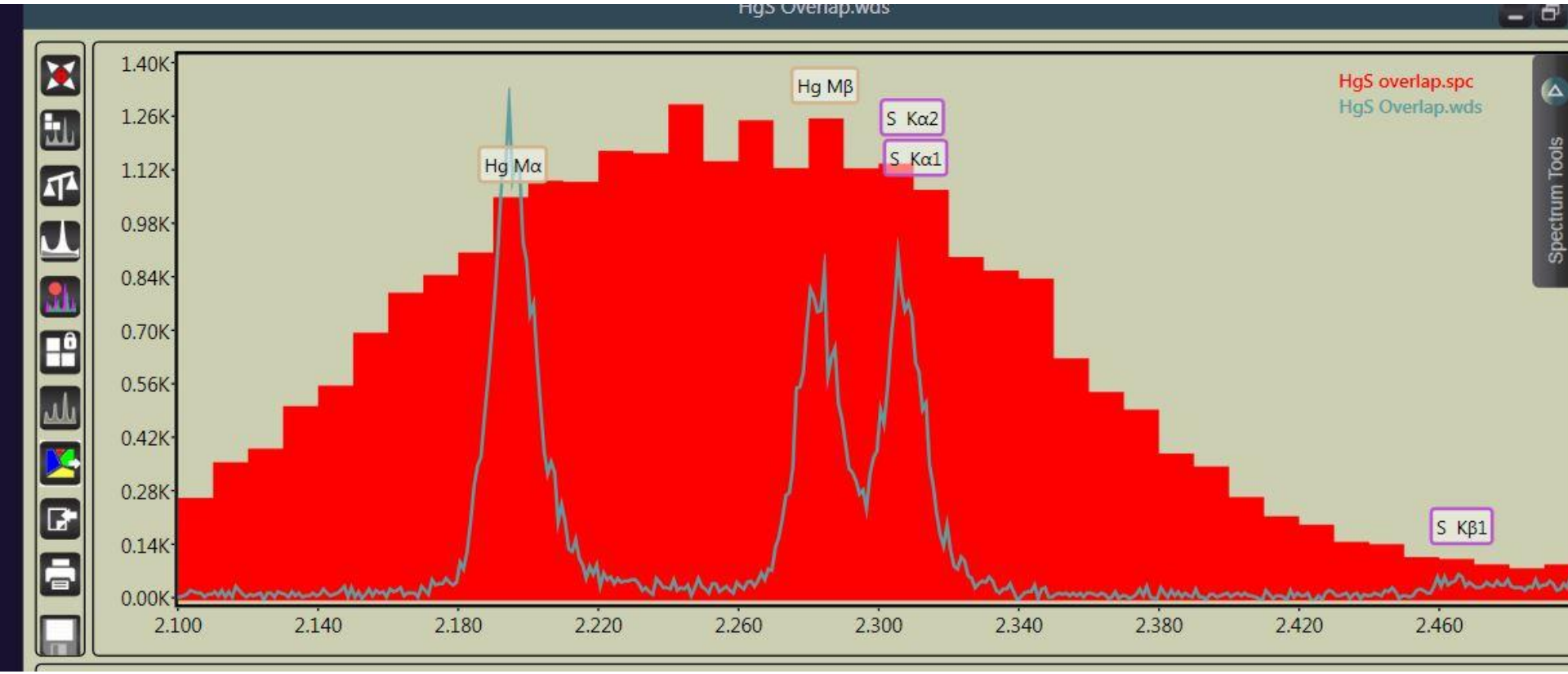
Chevauchement Pb-Bi-S

Source: EDAX –Harry Verhulst



Chevauchement Ta-Si

Source: EDAX –Harry Verhulst



Chevauchement Hg-S

Source: EDAX –Harry Verhulst

Détection de traces d'élements

TEXS Minimum Detection Limits (MDL)

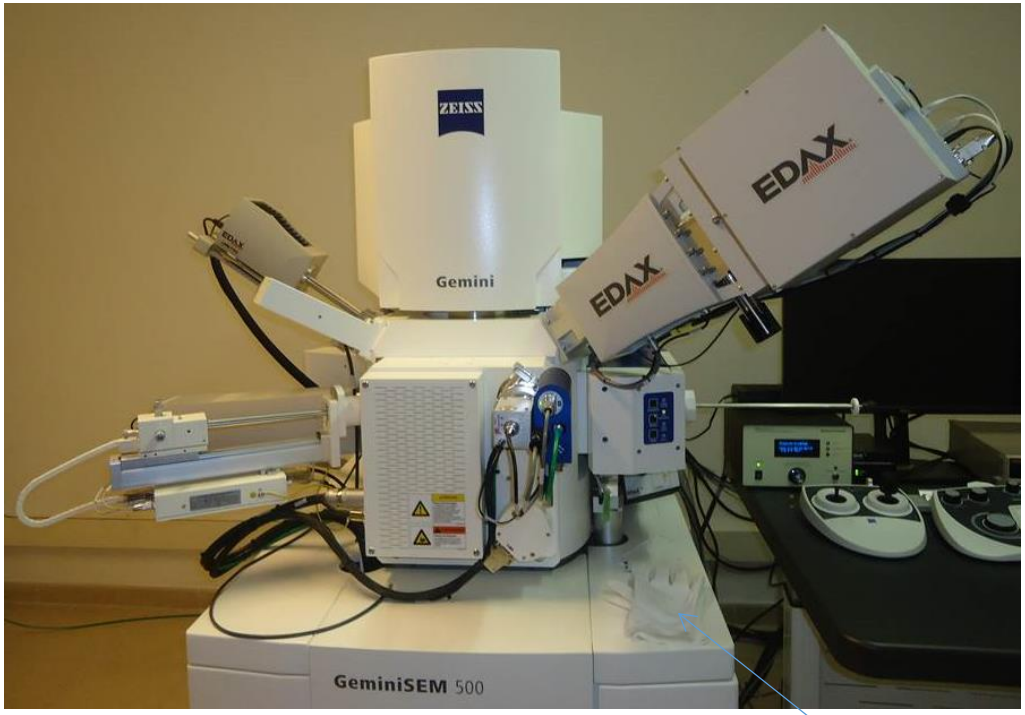
Element	Crystal	kV	Current, nA	Time, sec	2 sigma MDL, wt%	in ppm
CrKa	LiF	20	24.2	5	0.029	288
NiKa	LiF	20	24.2	5	0.078	780
SiKa	PET	20	24.2	5	0.024	241
AlKa	2d=30	20	38.4	5	0.040	401
ZrLa	PET	20	23.8	5	0.068	680
CeLa	LiF	25	22.9	5	0.056	560
MoLa	PET	25	22.9	5	0.034	340
TiKa	LiF	25	22.9	5	0.022	220
Vka	LiF	25	22.9	5	0.037	370
CrKa	LiF	25	22.9	5	0.011	110
MnKa	LiF	25	22.9	5	0.015	150
FeKa	LiF	25	22.9	5	0.029	290
NiKa	LiF	25	22.9	5	0.027	270
CuKa	LiF	25	22.9	5	0.043	430

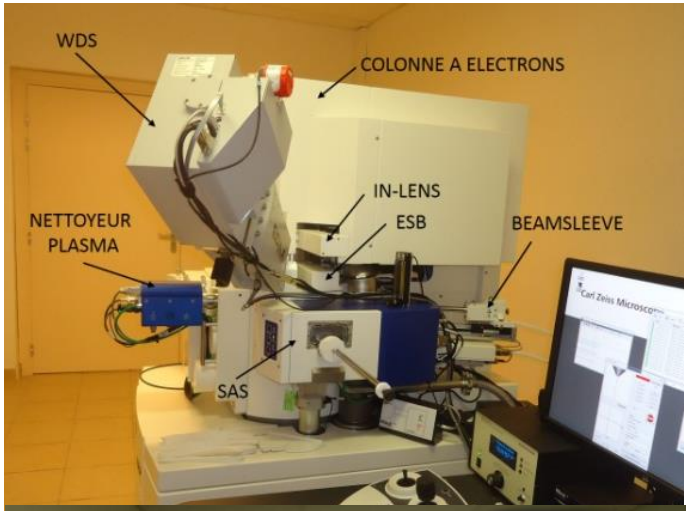
Element	Crystal	kV	Current, nA	Time, sec	2 sigma MDL, wt%	in ppm
C Ka	2d=80	10	18.6	5	0.063	632
N Ka	2d=80	10	18.6	1	0.180	1799
O Ka	2d=80	10	18.6	5	0.040	400
Na Ka	2d=61	10	18.6	5	0.021	214
Mg Ka	2d=30	10	18.6	1	0.070	704
CaKa	PET	10	18.6	1	0.656	6564
CaKa	LiF	20	8.1	5	0.020	200
ZrLa	PET	10	16.9	5	0.150	1500
Yla	PET	10	16.9	5	0.140	1400
CeLa	LiF	10	16.9	5	0.270	2700

Note: limites sous conditions normales d`utilisation du microscope, i.e. courant 10-20 nA et temps d`acquisition de 5s

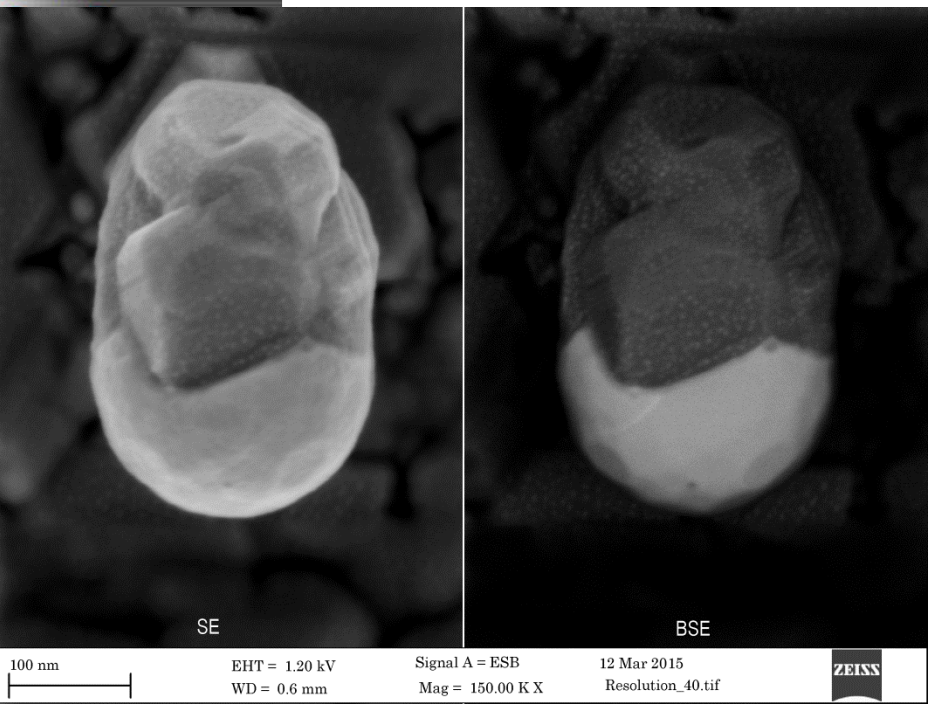
Source: EDAX

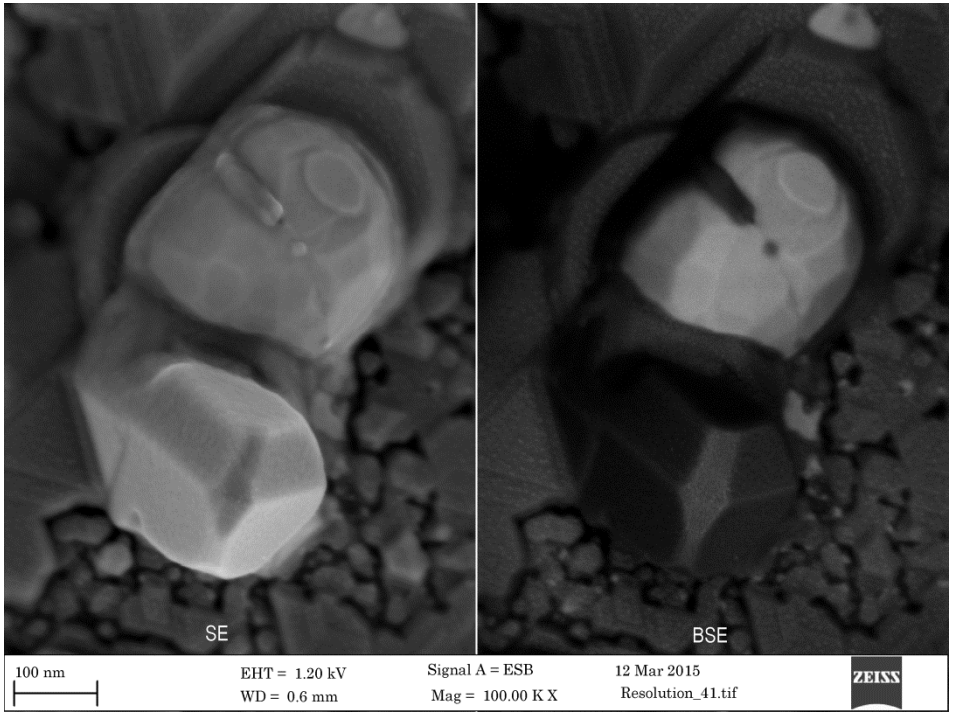
2015 premier MEB ZEISS Gemini 500 20 nA

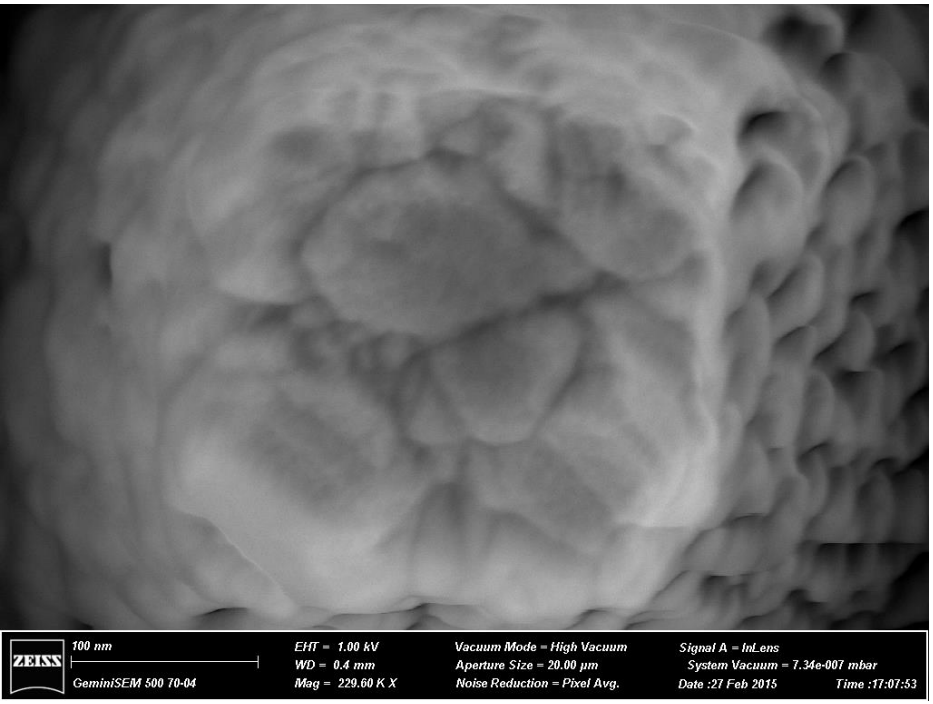


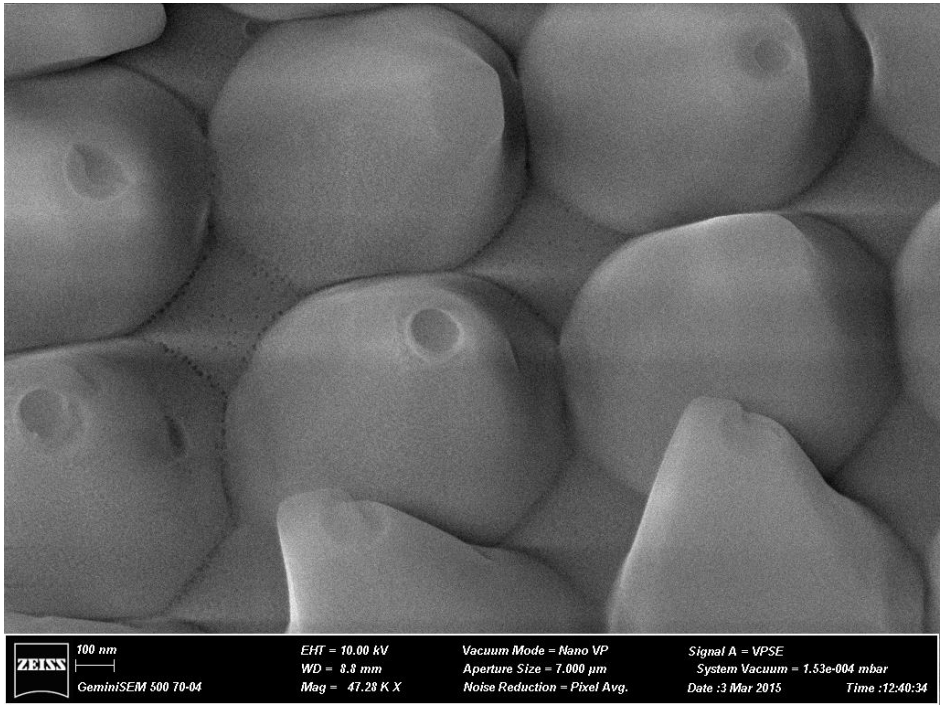


Resolution	1.2 nm (500 V) 1.1 nm (1 kV) 0.6 nm (15 kV)	1.8 nm (3kV, 50 Pa VP) 0.9 nm (1kV, Sample Bias)
Beam Current	3 pA (1 kV) - 20 nA (30kV), 2 Gun Modes, 14 Steps	
Variable Pressure	5 – 500 Pa high resolution configuration 5 – 150 Pa large field of view configuration	


SE
BSE
100 nm
EHT = 1.20 kV
WD = 0.6 mm
Signal A = ESB
Mag = 150.00 K X
12 Mar 2015
Resolution_40.tif
ZEISS

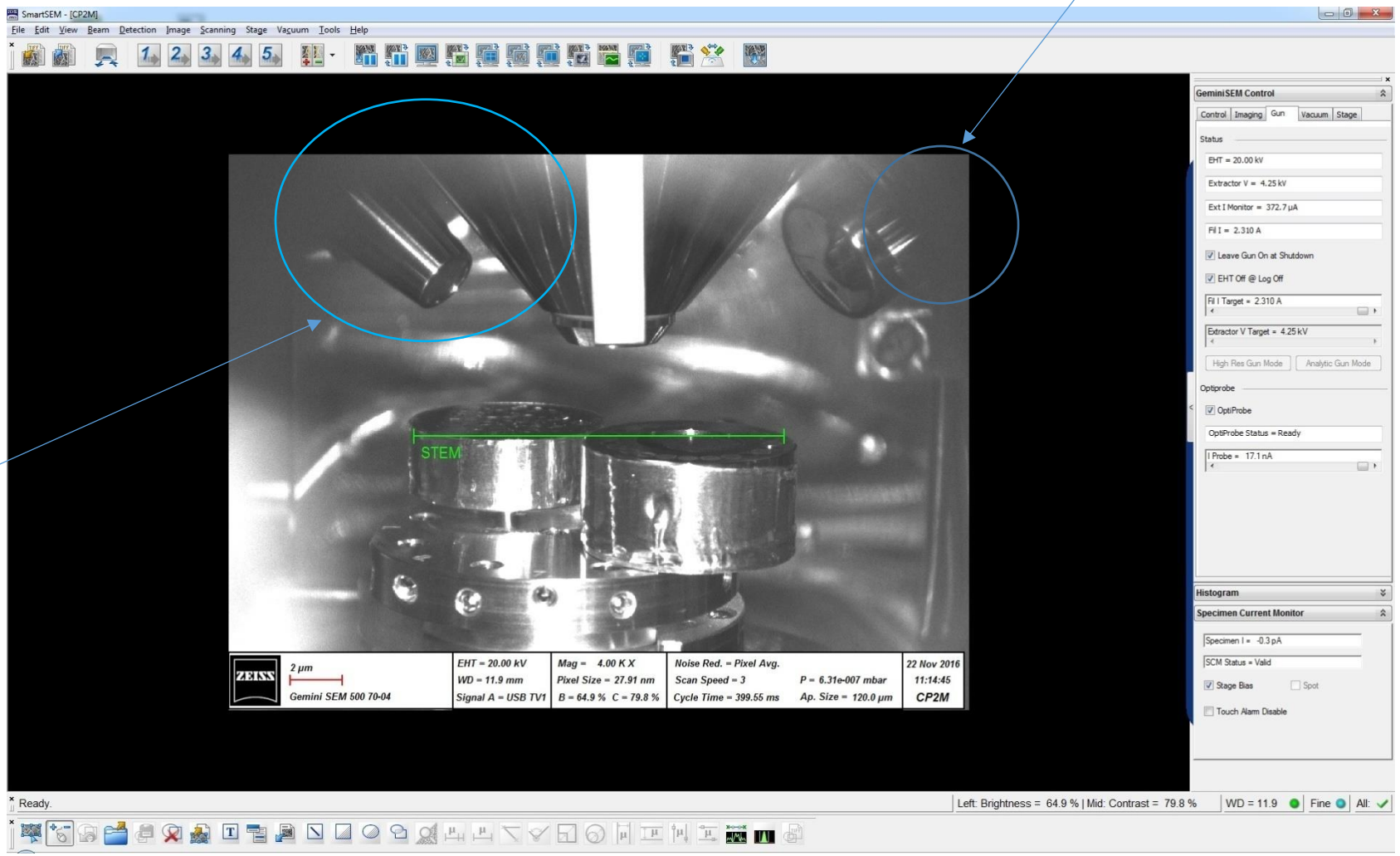

SE
BSE
100 nm
EHT = 1.20 kV
WD = 0.6 mm
Signal A = ESB
Mag = 100.00 K X
12 Mar 2015
Resolution_41.tif
ZEISS


ZEISS
100 nm
GeminiSEM 500 70-04
EHT = 1.00 kV
WD = 0.4 mm
Mag = 229.60 K X
Vacuum Mode = High Vacuum
Aperture Size = 20.00 µm
Noise Reduction = Pixel Avg.
Signal A = InLens
System Vacuum = 7.34e-007 mbar
Date :27 Feb 2015
Time :17:07:53


ZEISS
100 nm
GeminiSEM 500 70-04
EHT = 10.00 kV
WD = 8.8 mm
Mag = 47.28 K X
Vacuum Mode = Nano VP
Aperture Size = 7.000 µm
Noise Reduction = Pixel Avg.
Signal A = VPSE
System Vacuum = 1.53e-004 mbar
Date :3 Mar 2015
Time :12:40:34

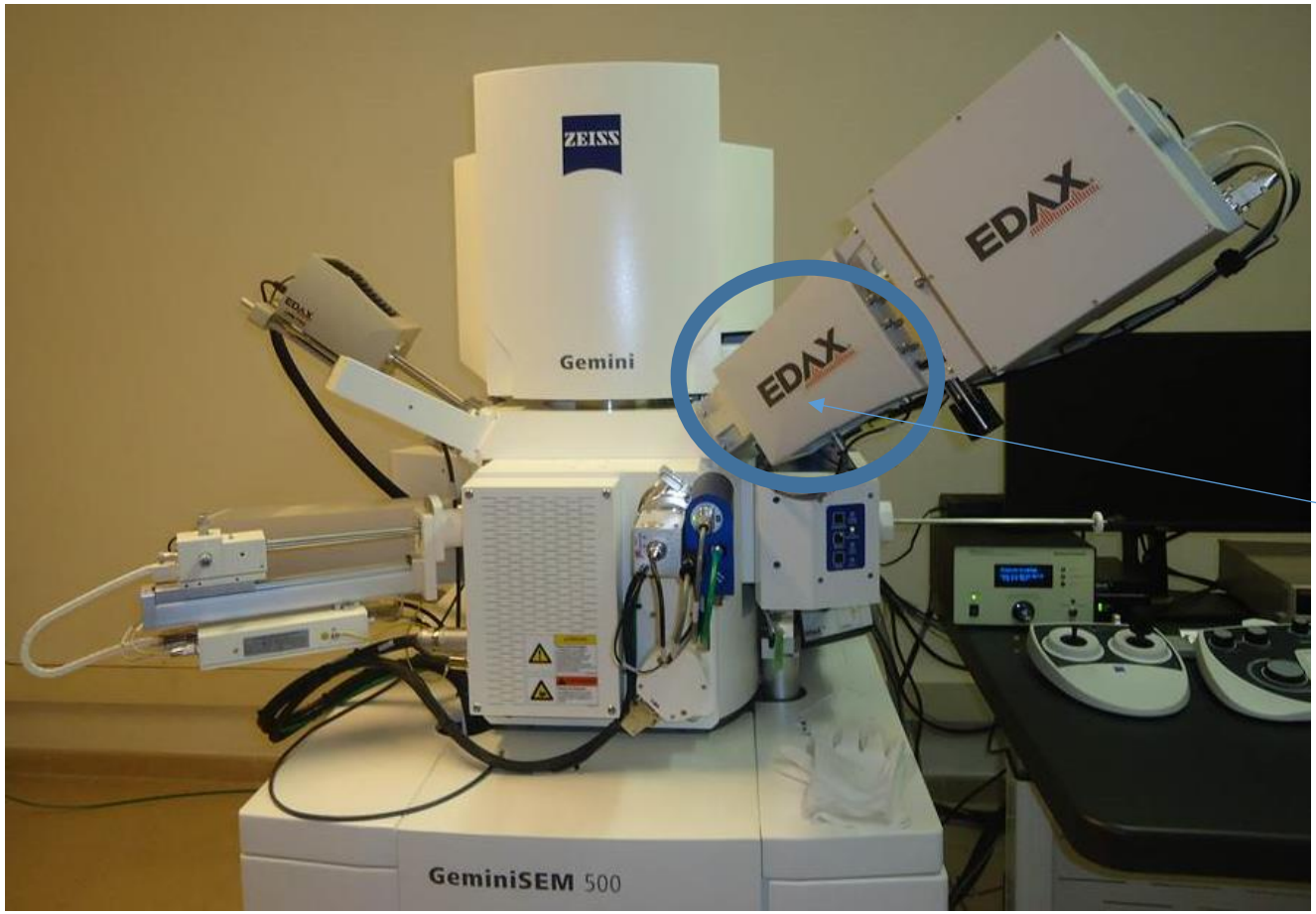
WDS à faisceau parallele (PBS –Parallel Beam SPectrometer), comment ça marche?

EDS


SmartSEM - [CP2M]
File Edit View Beam Detection Image Scanning Stage Vacuum Tools Help
GeminiSEM Control
Control Imaging Gun Vacuum Stage
Status
EHT = 20.00 kV
Extractor V = 4.25 kV
Ext I Monitor = 372.7 µA
Fil I = 2.310 A
Leave Gun On at Shutdown
EHT Off @ Log Off
Fil I Target = 2.310 A
Extractor V Target = 4.25 kV
High Res Gun Mode
Analytic Gun Mode
Optiprobe
OptiProbe
OptiProbe Status = Ready
I Probe = 17.1 nA
STEM
2 µm
Gemini SEM 500 70-04
EHT = 20.00 kV
WD = 11.9 mm
Signal A = USB TV1
Mag = 4.00 K X
Pixel Size = 27.91 nm
B = 64.9 % C = 79.8 %
Noise Red. = Pixel Avg.
Scan Speed = 3
Cycle Time = 399.55 ms
P = 6.31e-007 mbar
Ap. Size = 120.0 µm
22 Nov 2016
11:14:45
CP2M
Histogram
Specimen Current Monitor
Specimen I = -0.3 pA
SCM Status = Valid
Stage Bias
Spot
Touch Alarm Disable
Ready.
Left: Brightness = 64.9 % | Mid: Contrast = 79.8 %
WD = 11.9
Fine
All:

WDS

WDS à faisceau parallele (PBS –Parallel Beam Spectrometer), comment ça marche?



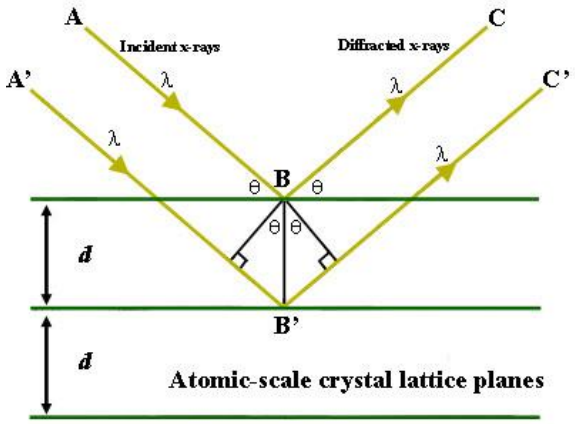
Focalisation

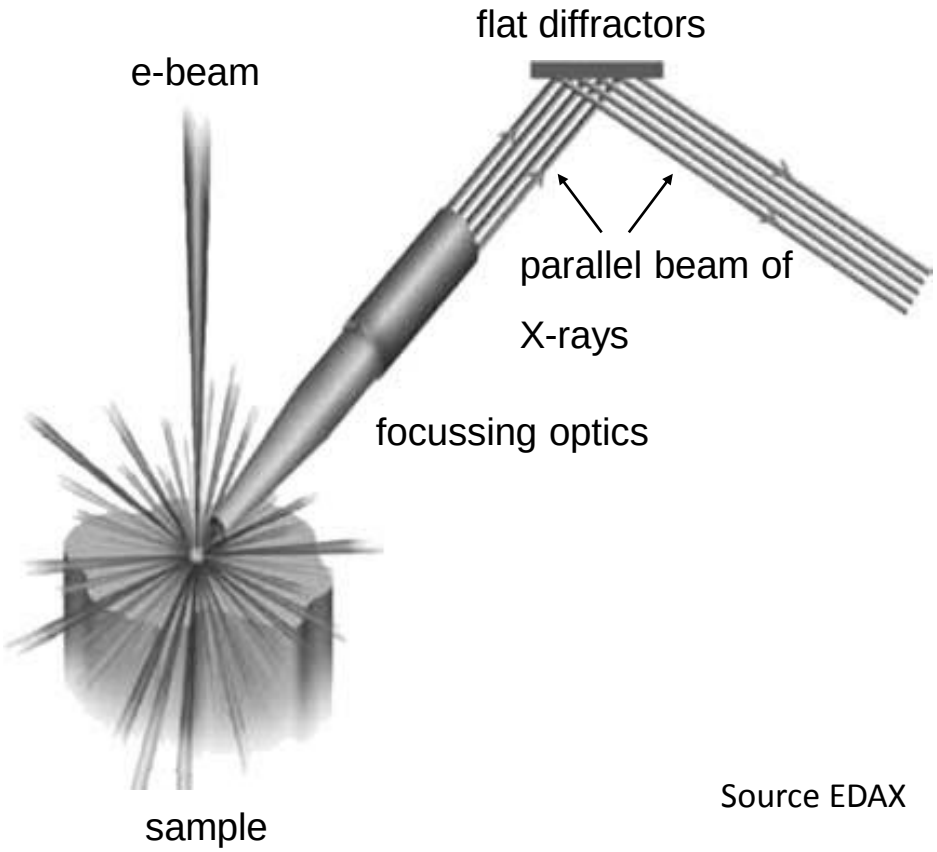
Parallel Beam WDS

Bragg's Law of x-ray diffraction

$$n \cdot \lambda = 2d \cdot \sin\theta$$

λ = wavelength of interest
d = interplanar spacing of the diffractor
θ = angle between the incident ray and the scattering planes
n = integer





Source EDAX

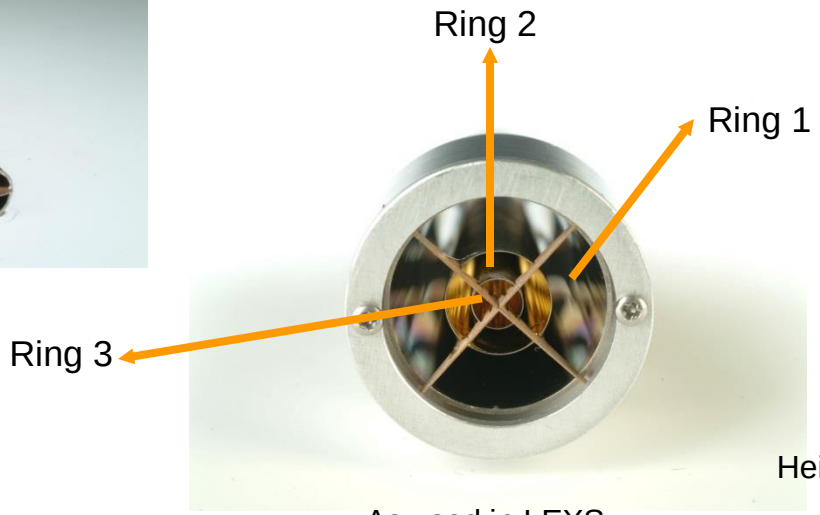
How to produce a Parallel Beam of X-Rays

- X-Ray Mirrors
  - Nested paraboloids to efficiently produce as parallel beam
- Polycapillary Optics
  - Multi fiber and monolithic optics used to produce a parallel beam

Source EDAX

X-Ray Mirrors (High Collection Optics)

Pour les basses energies
WDS LEXS (Low Energy X-ray Spectrometer)



As used in LEXS

Ring 1 – < 500 eV

Ring 2 – 500-1500 eV

Ring 3 – 1500-2400eV

Height tolerance:

200 µm (BeKa : 0.108 keV)

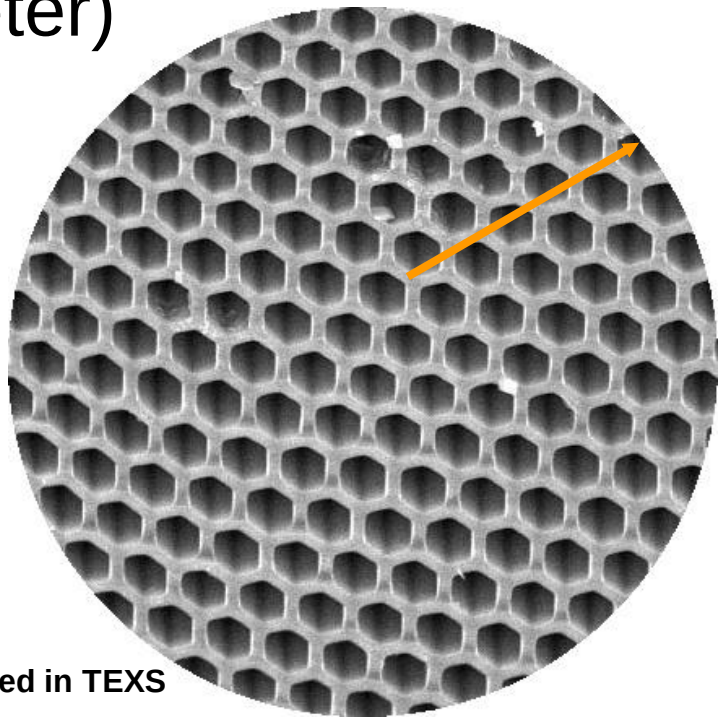
25 µm (SiKa : 1.74 keV)

70 eV to 2450 eV

Source EDAX

Polycapillary optics

LambdaSpec TEXS
(Transition Element
X-ray Spectrometer)



As used in TEXS

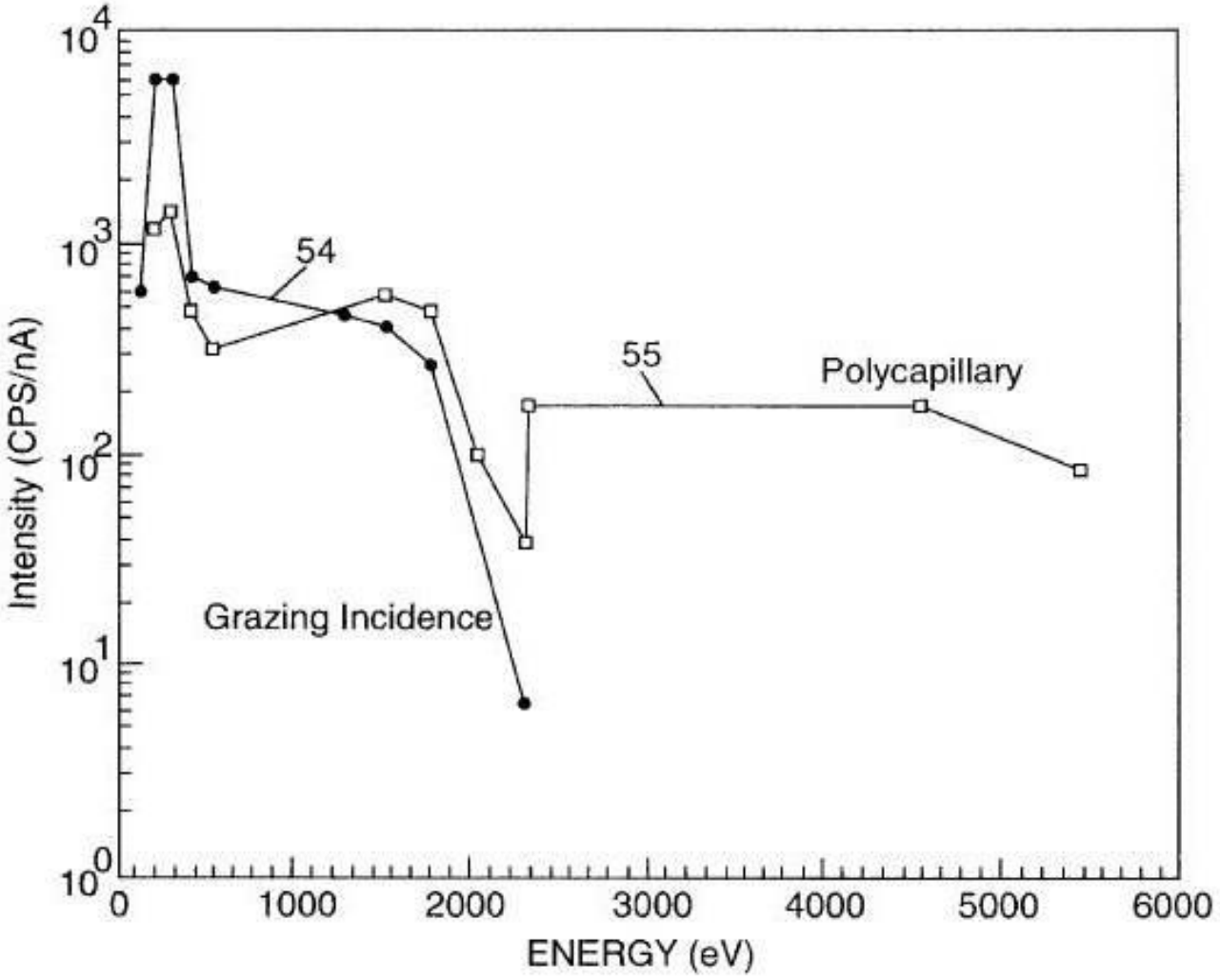
About 500.000 capillaries used

(channels: 5-10 µm)

170 eV to 10 keV

Source EDAX

HCO versus Polycapillary optics



The concentric mirrors have higher reflection efficiency than the poly-capillary optic for lower energies, whereas the poly-capillary optic is a more effective optic for higher energies

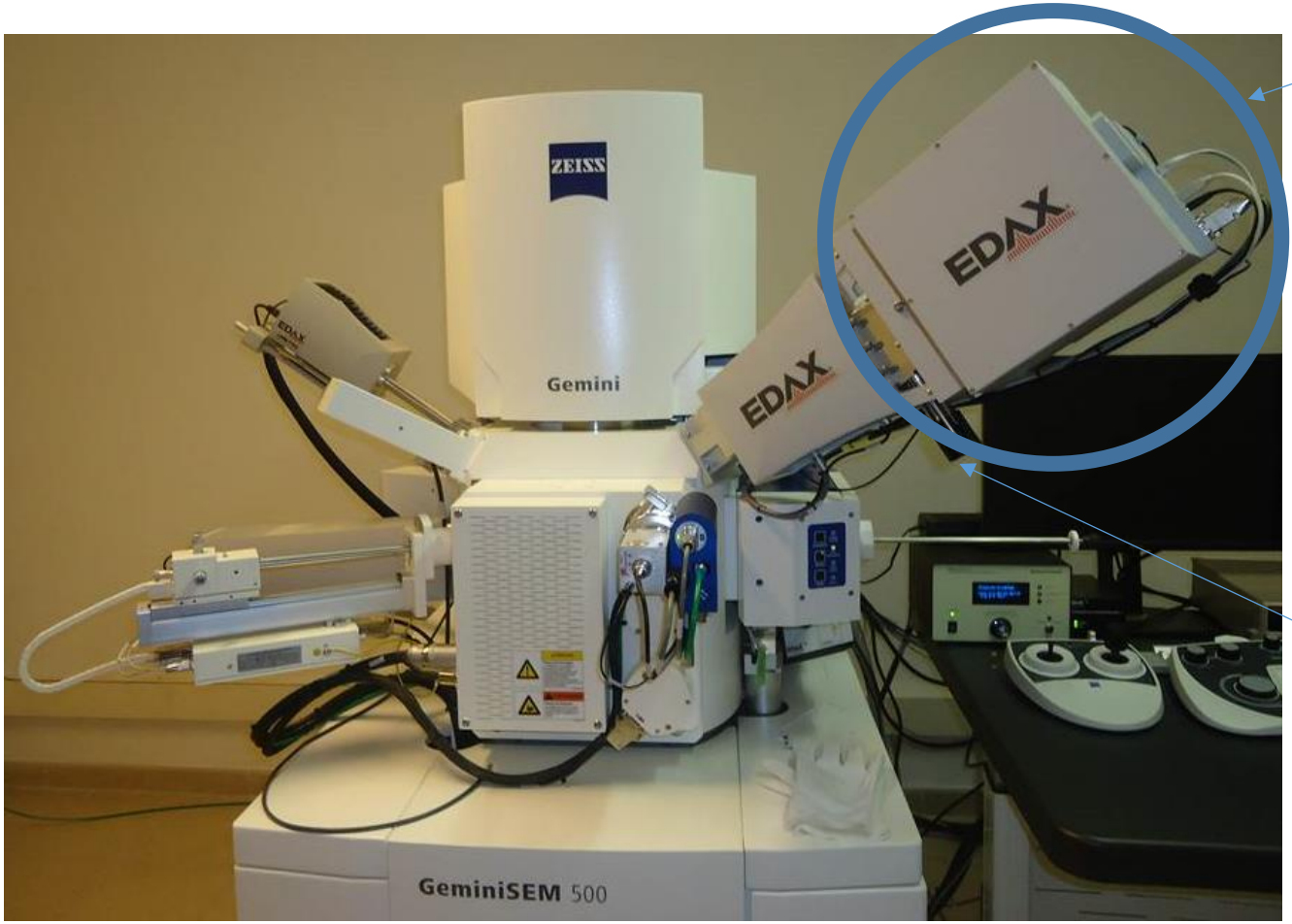
Source EDAX

Advantages of PBS

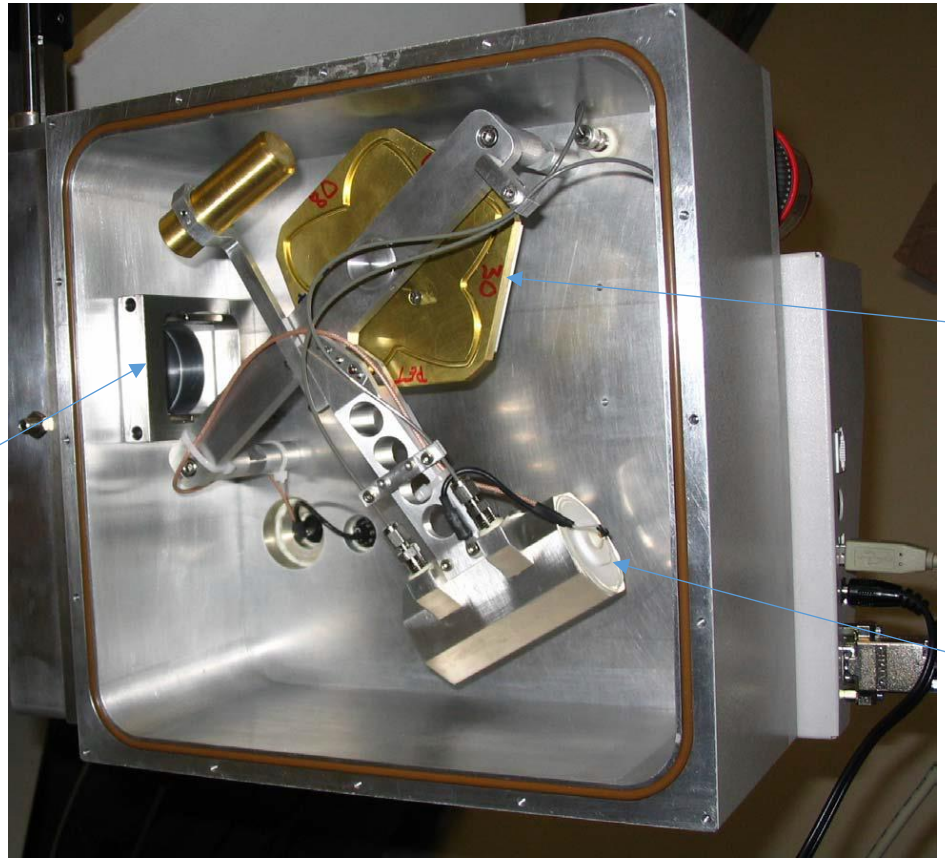
- Compact design (dimensions: 25x25x15 cm)
- Higher sensitivity
  - X-Ray path shorter
  - HCO or capillary optics are efficient X-Ray collection devices
- High beam current not required to achieve sensitivity
- Flat crystals are more stable/easier to make
- Can be mounted on “all” SEM chambers without modifications (port selection thus less critical than curved crystal spectrometers)
- Less complex movements

Source EDAX

Spectromètre



Vanne



Entrée du faisceau

Tourelle à
5 cristaux

Compteur
Proportionnel

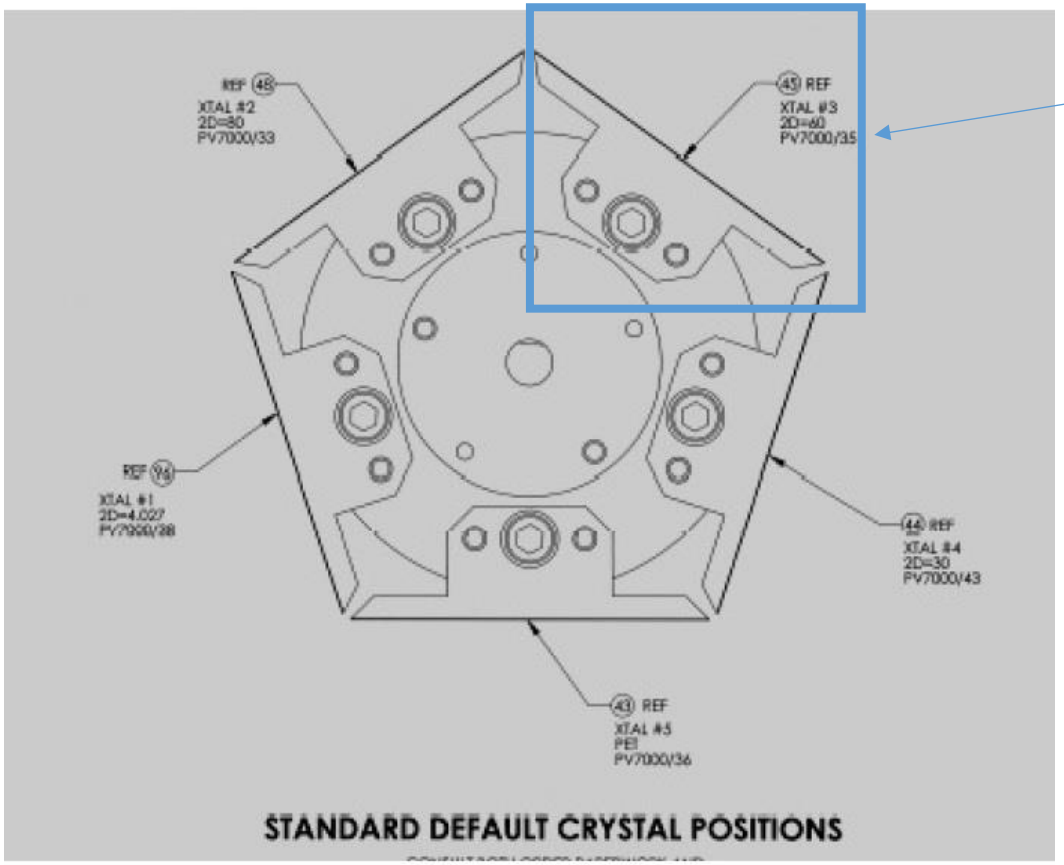
Pas de fente!!!

Source EDAX

	Crystal	2d spacing [Å]	Element coverage	eV range optimal	TEXS/ LEXS
	MoB_4C	197	Be, B, C	100 – 360	**L**
	MoB_4C	120	C	220 – 320	**L**
	Ni/C	100	B, C	180 – 400	**T**
2	**CrSc**	80	C, N	200 – 420	**T/L**
	CrTi	80	TiL	400 – 500	**L**
5	**WSi**	60	O – N (K-Lines) Ti- Ga (L-Lines)	420 – 1100	**T/L**
3	**OVO**	30	Na – Si Ga – Rb	1100 – 1700	**T/L**
4	**PET**	8.74	Si – S Rb – Mo	1700 - 2400	**T/L**
1	**LiF(200)**	4.027	K – Ge	3300 – 10.8	**T**
	LiF(220)	2.847	V – Y	4700 – 15.3	**T**

Diffractor Table, showing the ones used in TEXS in purple color.

Source: EDAX –Harry Verhulst

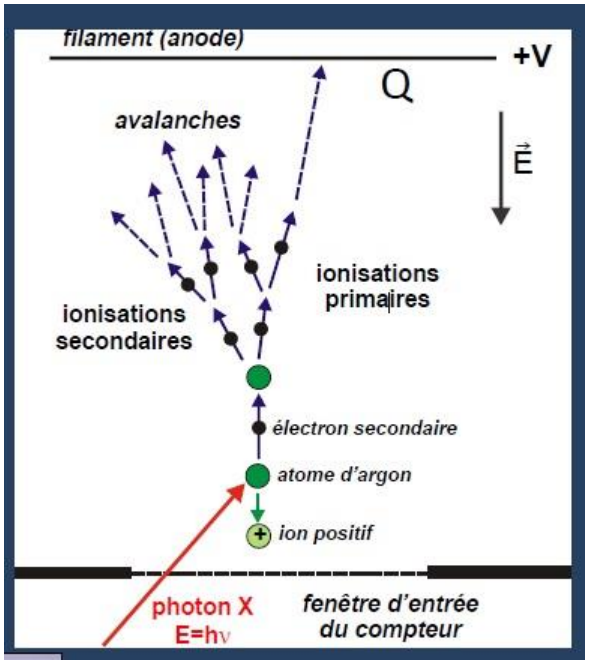


Cristal

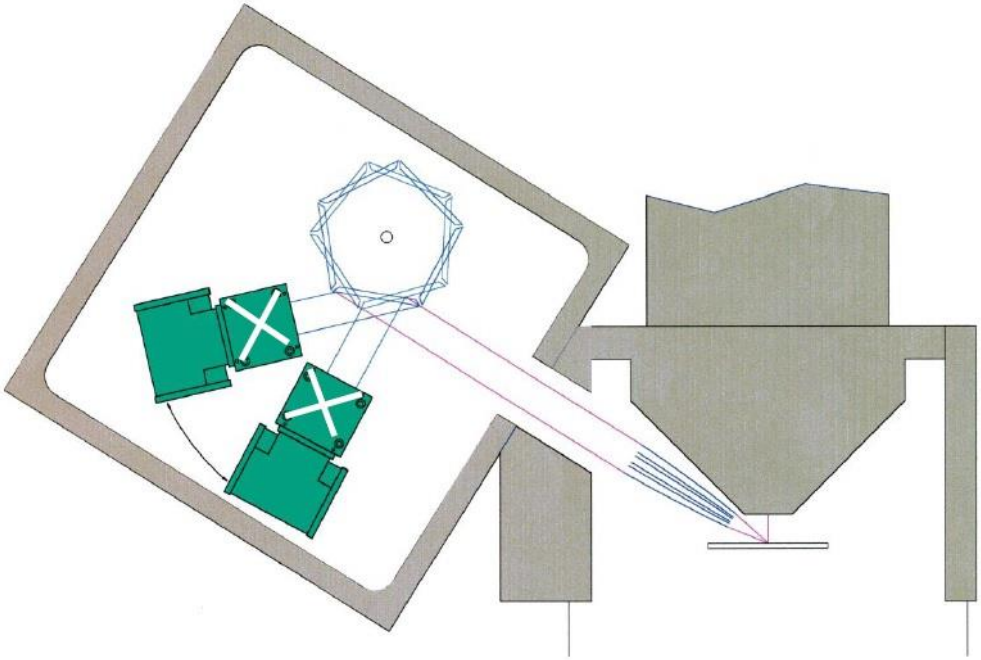
Source EDAX

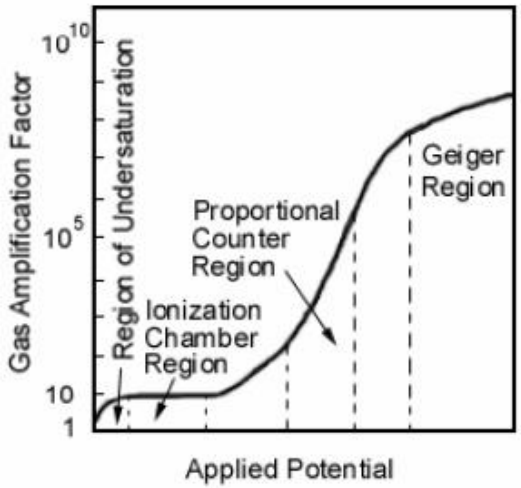
Merci Tomas Vikstrom - EDAX

Compteur proportionnel a flux gazeux
P10 gas (90% Argon / 10% Methane Ultra Purity)



Source Jacky Ruste





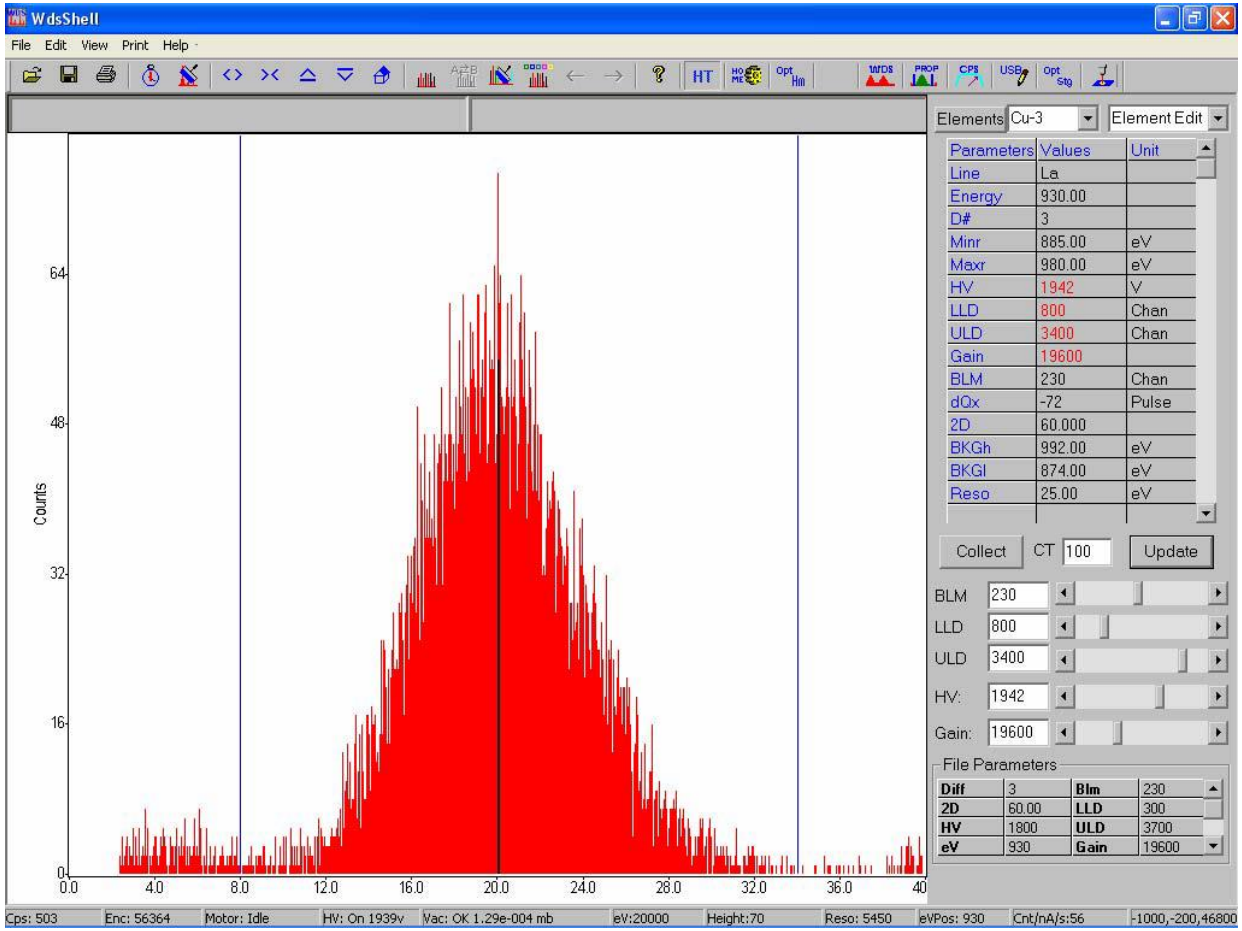
Source EDAX

Alignement du spectromètre
avec le programme WdsShell - HT compteur, hauteur, la position du pic

- Definition de coups max et meilleures resolution avec des standards

- Calibration de raies avec 2 standards par cristal

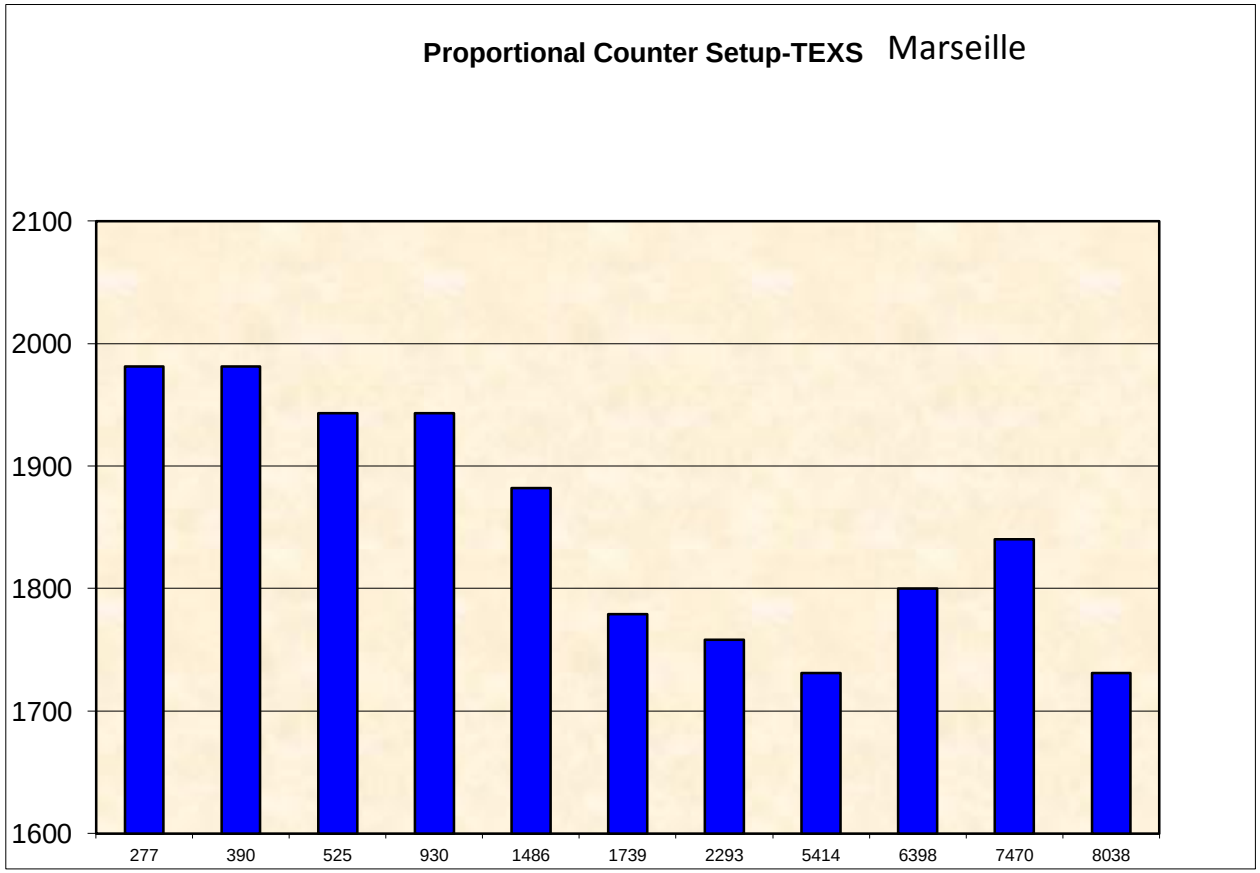
Référence pour cette etape:
Le test d`acceptance-comparaison avec les mesures réalisées à l`usine



D1-2d dQx calc CrK-CuK.xls
D2-2d dQx calc C-N.xls
D3-2d dQx calc O-Cu.xls
D4-2d dQx calc Al-Cu.xls
D5-2d dQx calc Si-Mo.xls

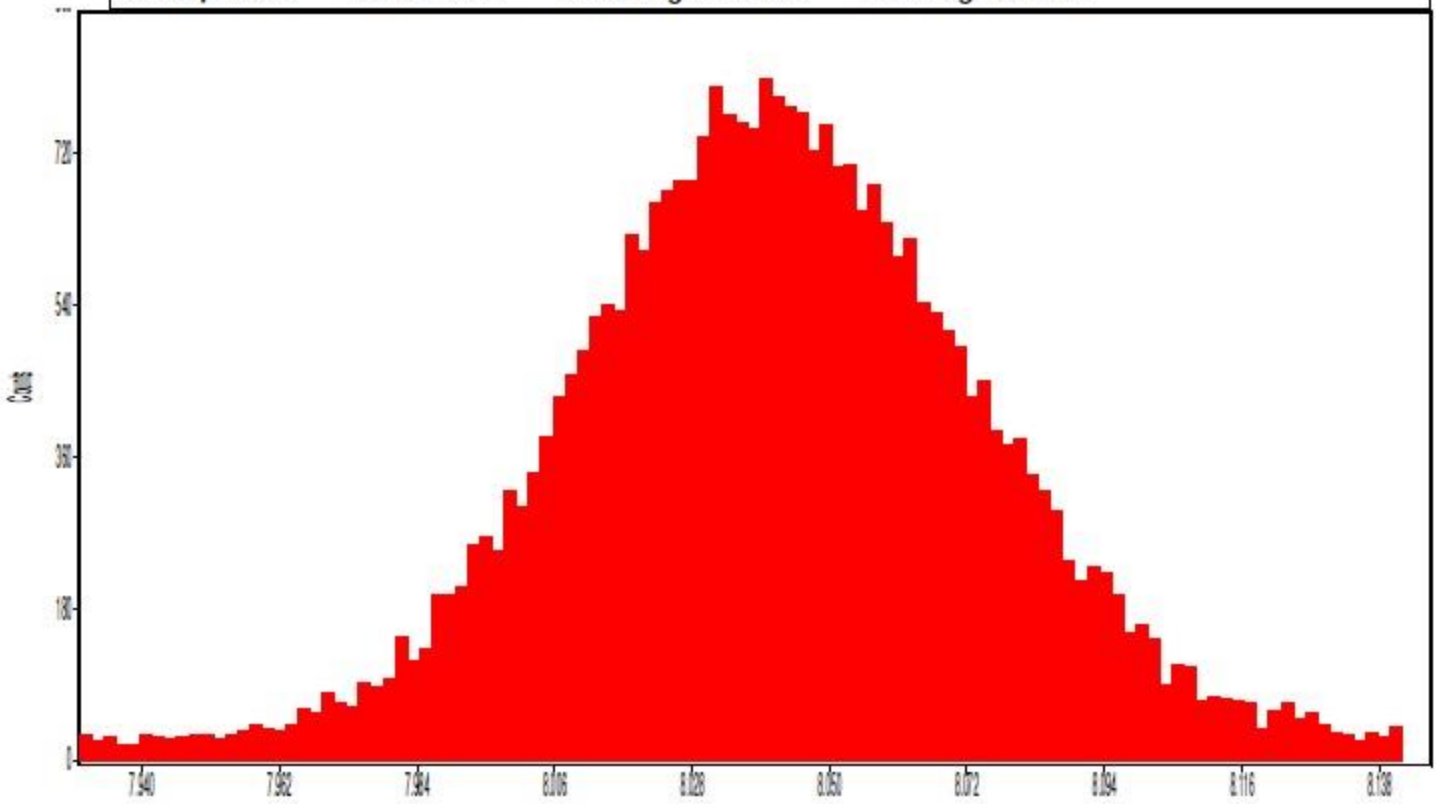
C	Ka	6	277	1981
N	Ka	7	390	1981
O	Ka	8	525	1943
Cu	La	29	930	1943
Al	Ka	13	1486	1882
Si	Ka	14	1739	1779
Mo	La	42	2293	1758
Cr	Ka	24	5414	1731
Fe	Ka	26	6398	1800
Ni	Ka	28	7470	1840
Cu	Ka	29	8038	1731

Alignement du HT du compteur pour chaque raie



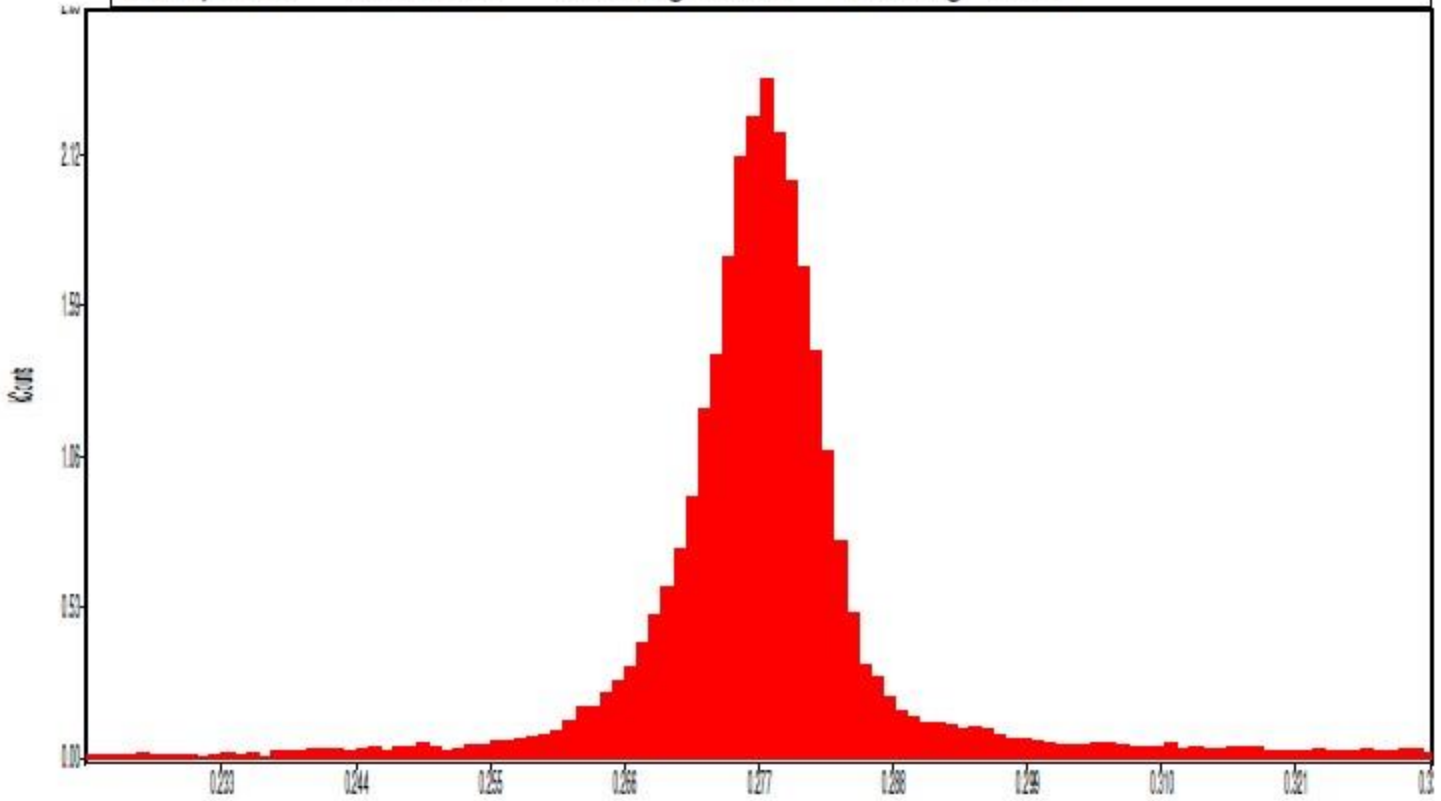
CuKα

Label: C:\EDAX SERVICE\Installation\WDS\Service visit June 2015\CuK.Wds				Jun-03-2015 _TIME:18:59:32
kv: 20.00	Cnts/nA/s: 109.95	MaxCnts: 807	BKG: 23.6	P/B: 34.2
Mag: 4102	WD: 10.96	Reso: 73	Beam Current: 3.67	
Diffractor: 1.0(--)	Prop HV: 1731	LLD: 400	ULD: 3200	Det Type: WDS_TEXS
Calibration (dQx) 217	2d: 4.015	Gain: 19675	BLM: 110	
ev Step: 2.00	Dwell: 2.00	Start Eng: 7930.00	End Eng: 8146.00	



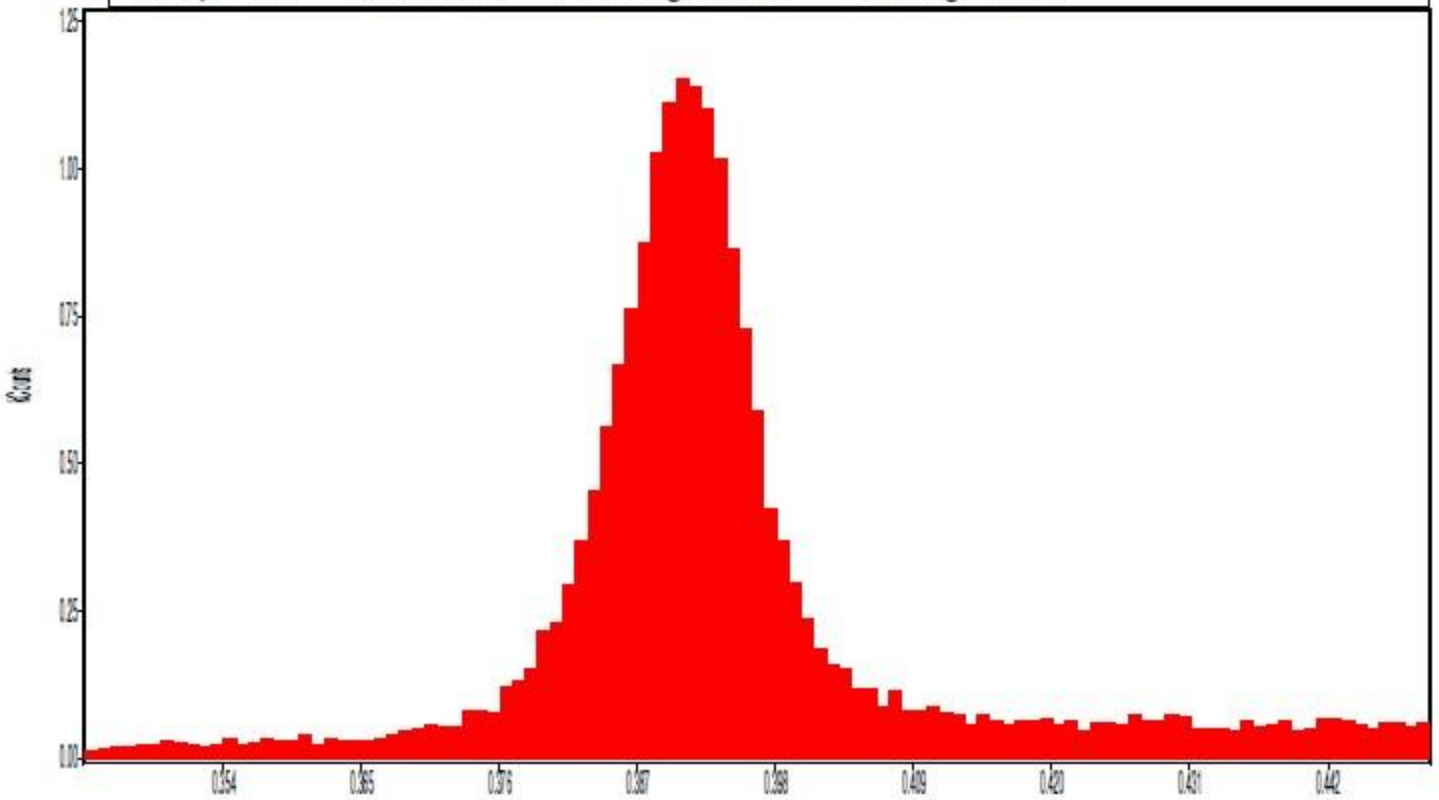
CKα

Label: Spc-0	Jun-04-2015	_TIME:13:8:2		
kv: 20.00	Cnts/nA/s: 446.72	MaxCnts: 2390	BKG: 11.6	P/B: 206.0
Mag: 3434	WD: 12.05	Reso: 10	Beam Current: 5.35	
Diffractor: 2.0(--)	Prop HV: 1981	LLD: 120	ULD: 3600	Det Type: WDS_TEXS
Calibration (dQx) 484	2d: 75.470	Gain: 19675	BLM: 110	
ev Step: 1.00	Dwell: 1.00	Start Eng: 222.00	End Eng: 332.00	

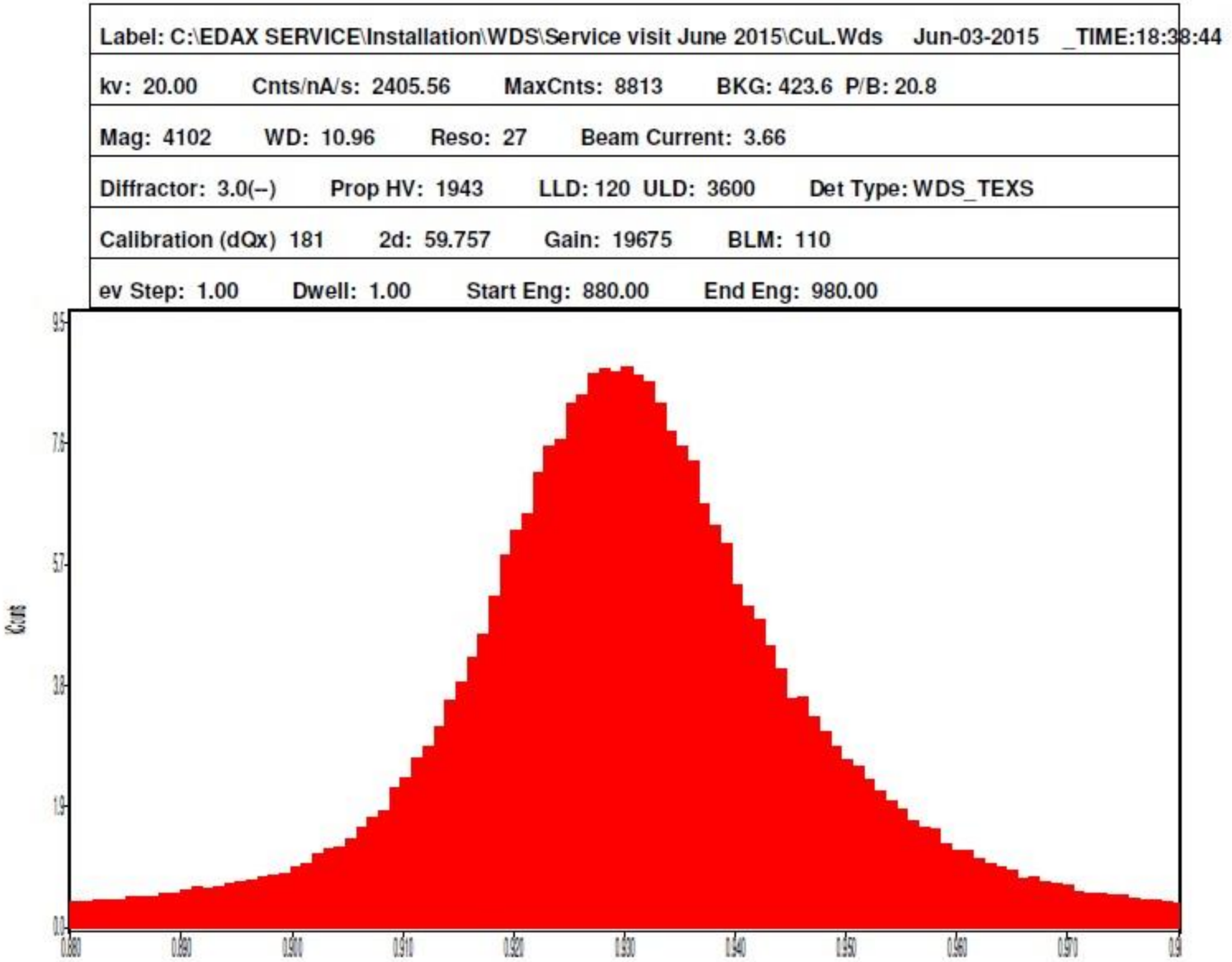


NKα

Label: C:\EDAX SERVICE\Installation\WDS\Service visit June 2015\N.Wds			Jun-03-2015	_TIME:18:27:33
kv: 20.00	Cnts/nA/s: 223.56	MaxCnts: 1154	BKG: 17.8	P/B: 64.8
Mag: 4102	WD: 10.96	Reso: 12	Beam Current: 5.16	
Diffractor: 2.0(–)	Prop HV: 1981	LLD: 120	ULD: 3600	Det Type: WDS_TEXS
Calibration (dQx) 484	2d: 75.470	Gain: 19675	BLM: 110	
ev Step: 1.00	Dwell: 1.00	Start Eng: 343.00	End Eng: 450.00	

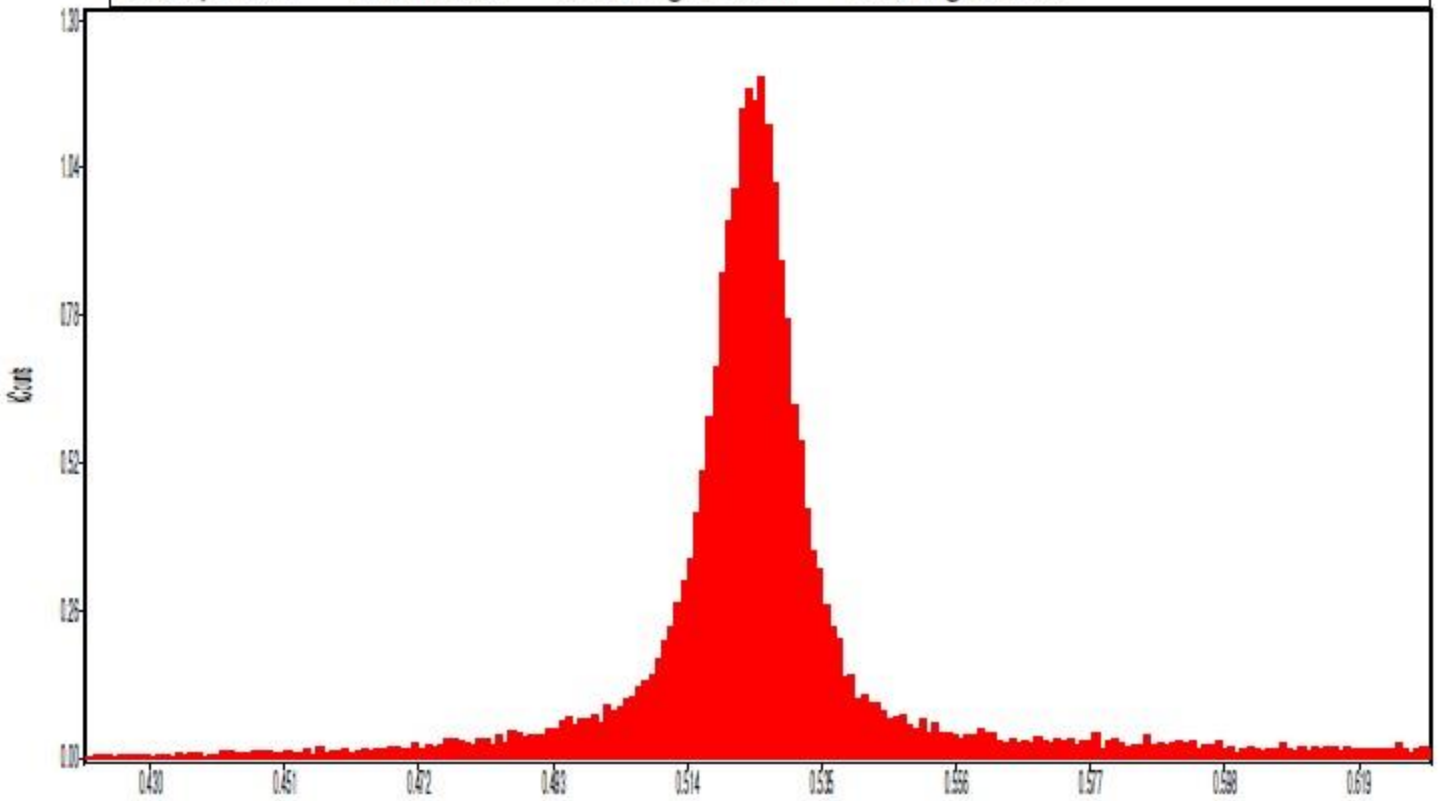


CuLα (diffractor 3, 2d=60Å)



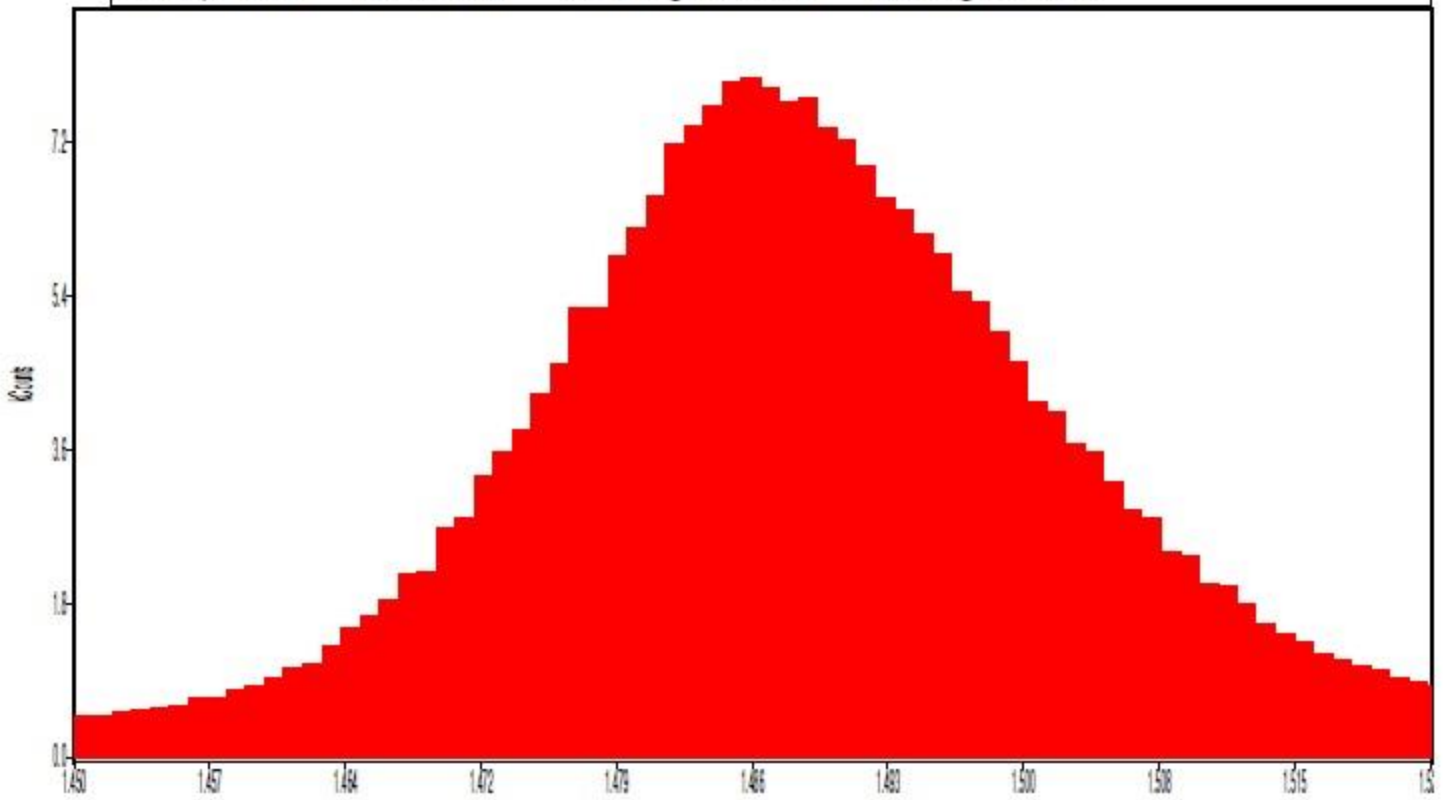
OKα

Label: C:\EDAX SERVICE\Installation\WDS\Service visit June 2015\OK.Wds				Jun-03-2015 _TIME:17:25:45
kv: 20.00	Cnts/nA/s: 266.96	MaxCnts: 1199	BKG: 3.2	P/B: 374.7
Mag: 4102	WD: 10.96	Reso: 14	Beam Current: 4.49	
Diffractor: 3.0(--)	Prop HV: 1943	LLD: 160	ULD: 3200	Det Type: WDS_TEXS
Calibration (dQx) 200	2d: 59.757	Gain: 19675	BLM: 110	
ev Step: 1.00	Dwell: 1.00	Start Eng: 420.00	End Eng: 630.00	

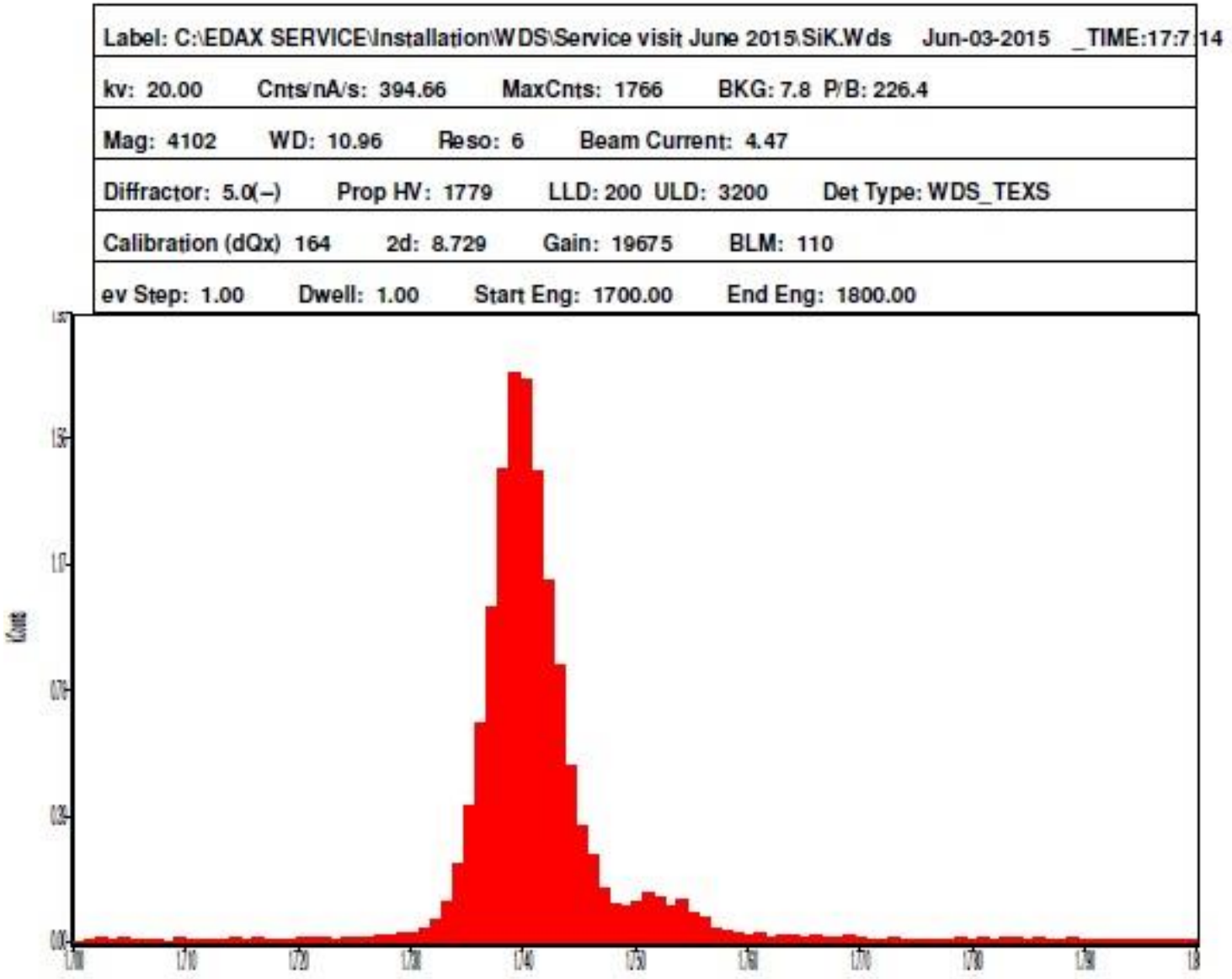


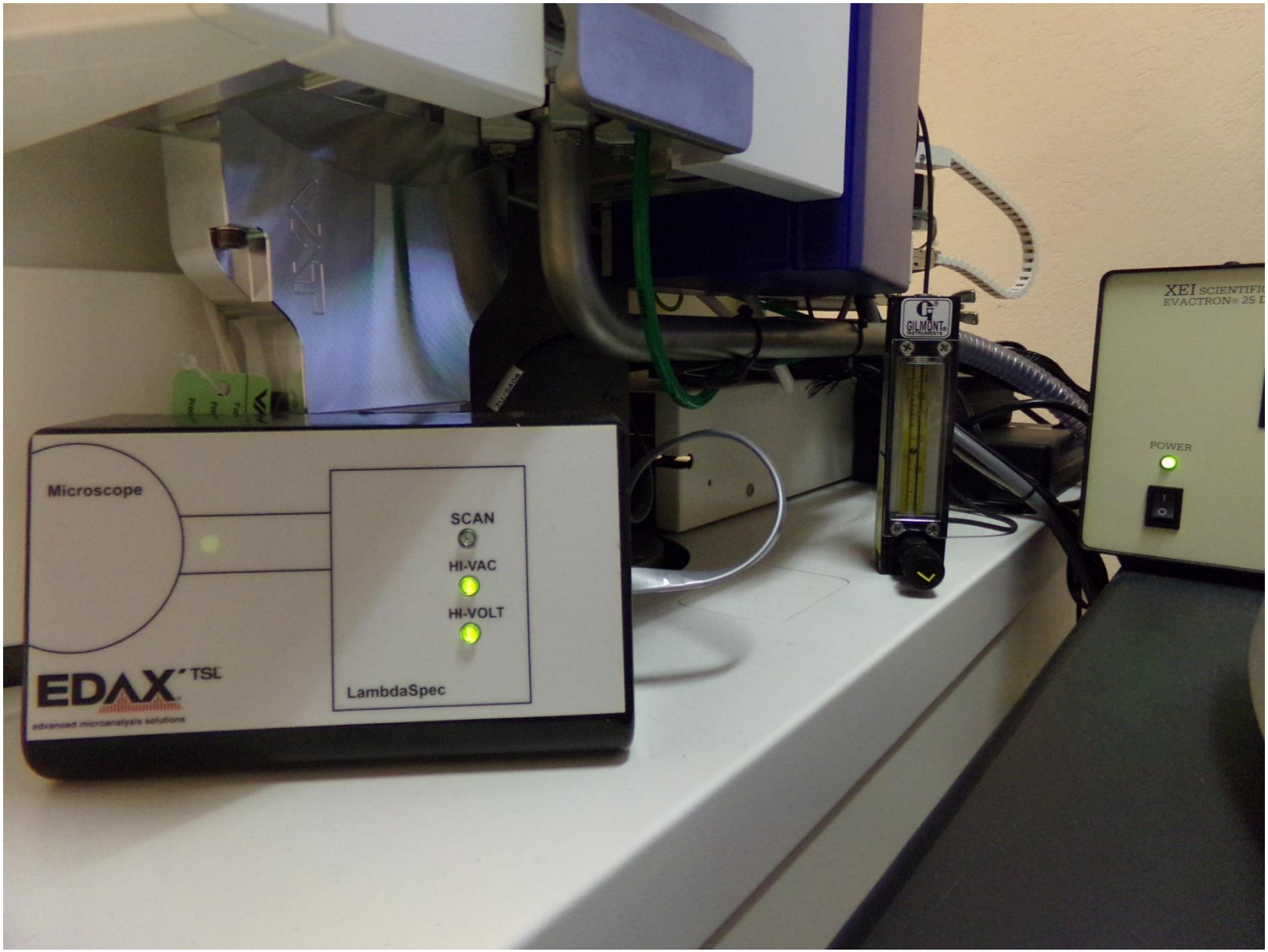
AlKα

Label: C:\EDAX SERVICE\Installation\WDS\Service visit June 2015\Al.Wds			Jun-03-2015	_TIME:18:14:28
kv: 20.00	Cnts/nA/s: 1678.58	MaxCnts: 7967	BKG: 530.8	P/B: 15.0
Mag: 4102	WD: 10.96	Reso: 28	Beam Current: 4.75	
Diffractor: 4.0(–)	Prop HV: 1882	LLD: 300	ULD: 3600	Det Type: WDS_TEXS
Calibration (dQx) 192	2d: 29.828	Gain: 19675	BLM: 110	
ev Step: 1.00	Dwell: 1.00	Start Eng: 1450.00	End Eng: 1522.00	

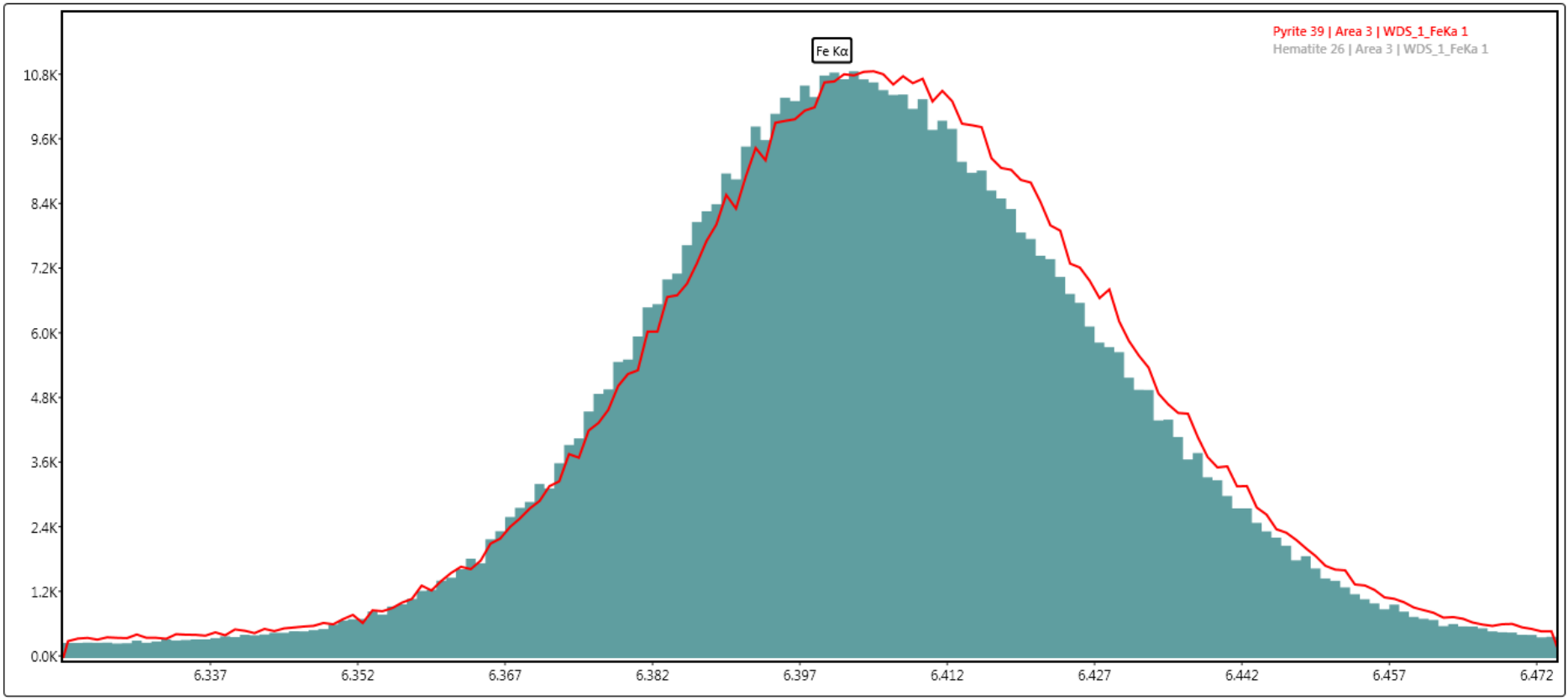


SiKα


Label: C:\EDAX SERVICE\Installation\WDS\Service visit June 2015\SiK.Wds Jun-03-2015 _TIME:17:7:14
kv: 20.00 Cnts/nA/s: 394.66 MaxCnts: 1766 BKG: 7.8 P/B: 226.4
Mag: 4102 WD: 10.96 Reso: 6 Beam Current: 4.47
Diffractor: 5.0(–) Prop HV: 1779 LLD: 200 ULD: 3200 Det Type: WDS_TEXS
Calibration (dQx) 164 2d: 8.729 Gain: 19675 BLM: 110
ev Step: 1.00 Dwell: 1.00 Start Eng: 1700.00 End Eng: 1800.00
KCounts

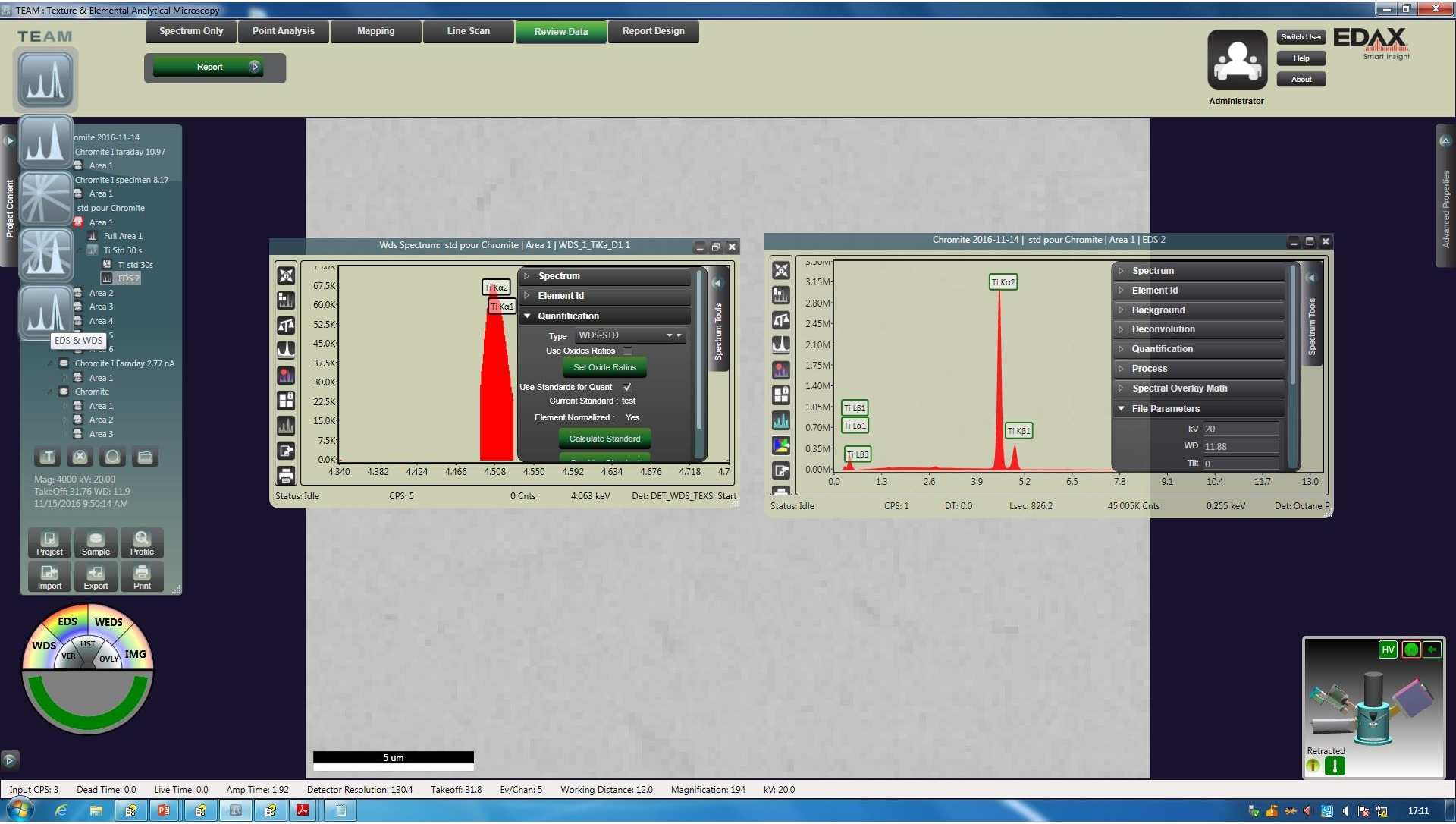

Microscope
SCAN
HI-VAC
HI-VOLT
EDAX TSL
advanced microanalysis solutions
LambdaSpec
GILMONT
XEI SCIENTIFIC
EVACTRON 25
POWER

Hematite (Fe 3+)
Pyrite (Fe 2+)

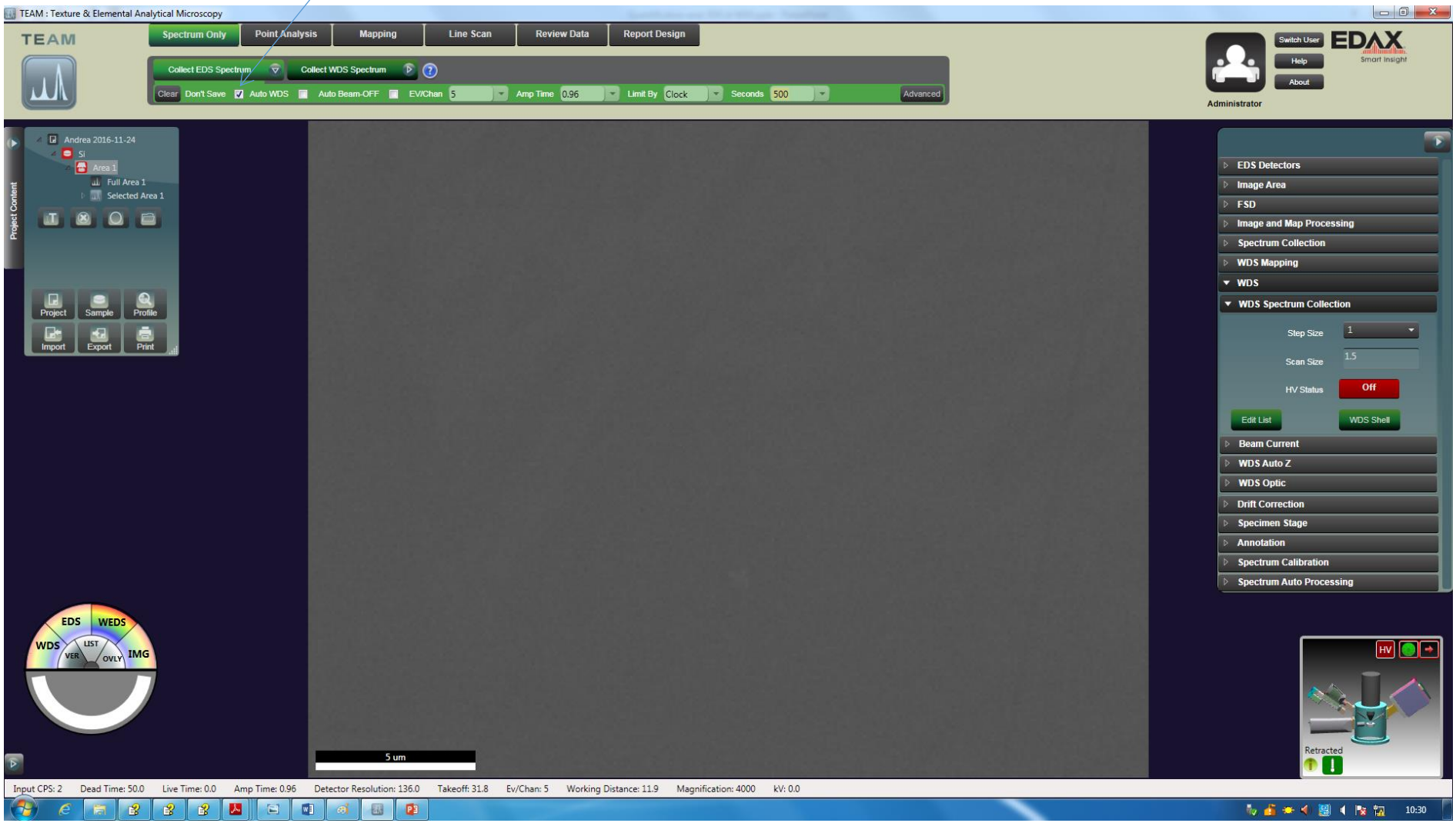


Status: Idle CPS: 0 DT 10.873K Cnts 6.400 keV Det: Apollo XF Start: 6322eV End: 6475eV PeakFraction: 1.5 Scan: FS Resolution: 52.5eV Step: 1

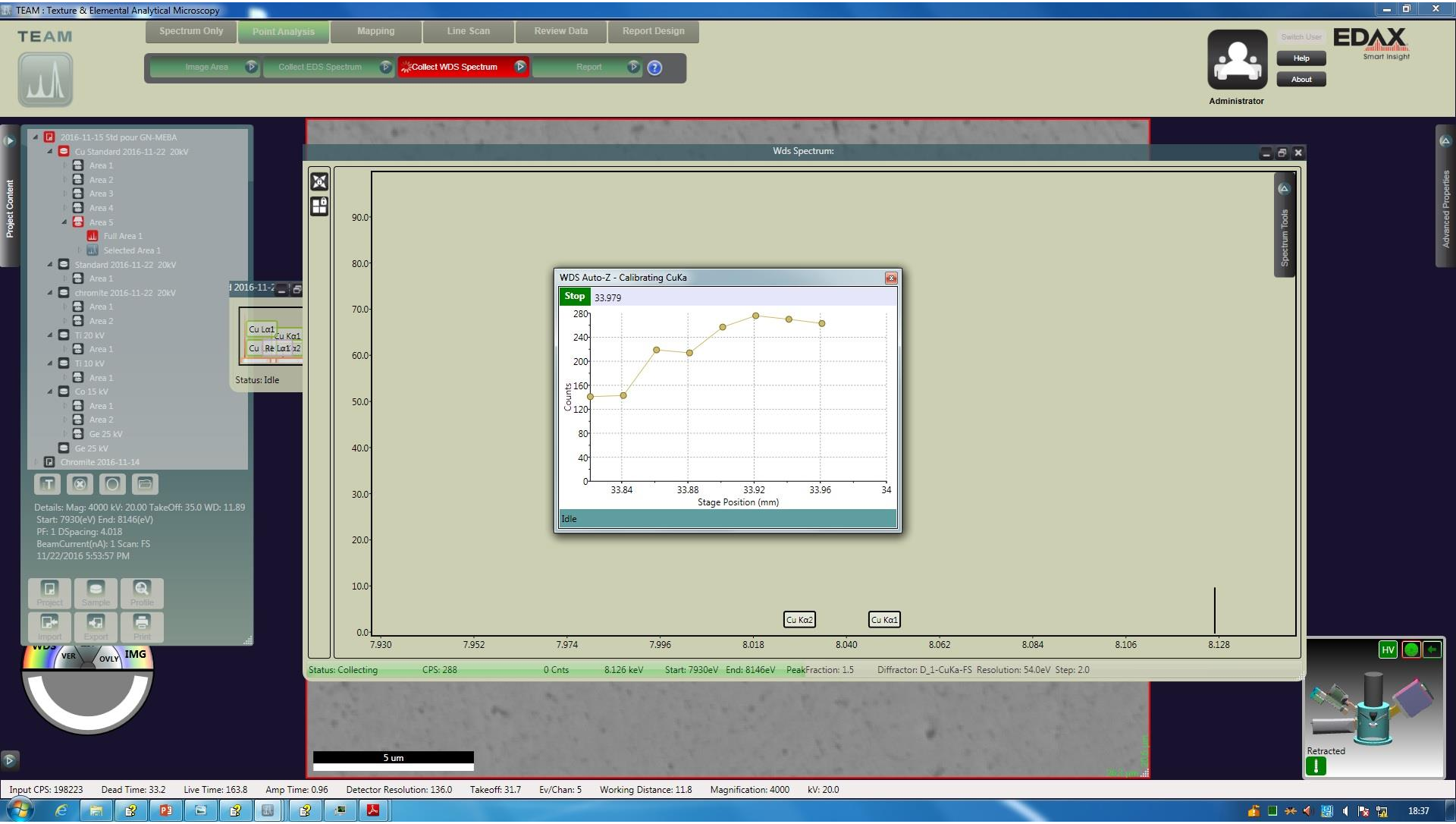
Software TEAM – EDS + WDS



Auto WDS: tout automatique ...



Check Height Cu Ka



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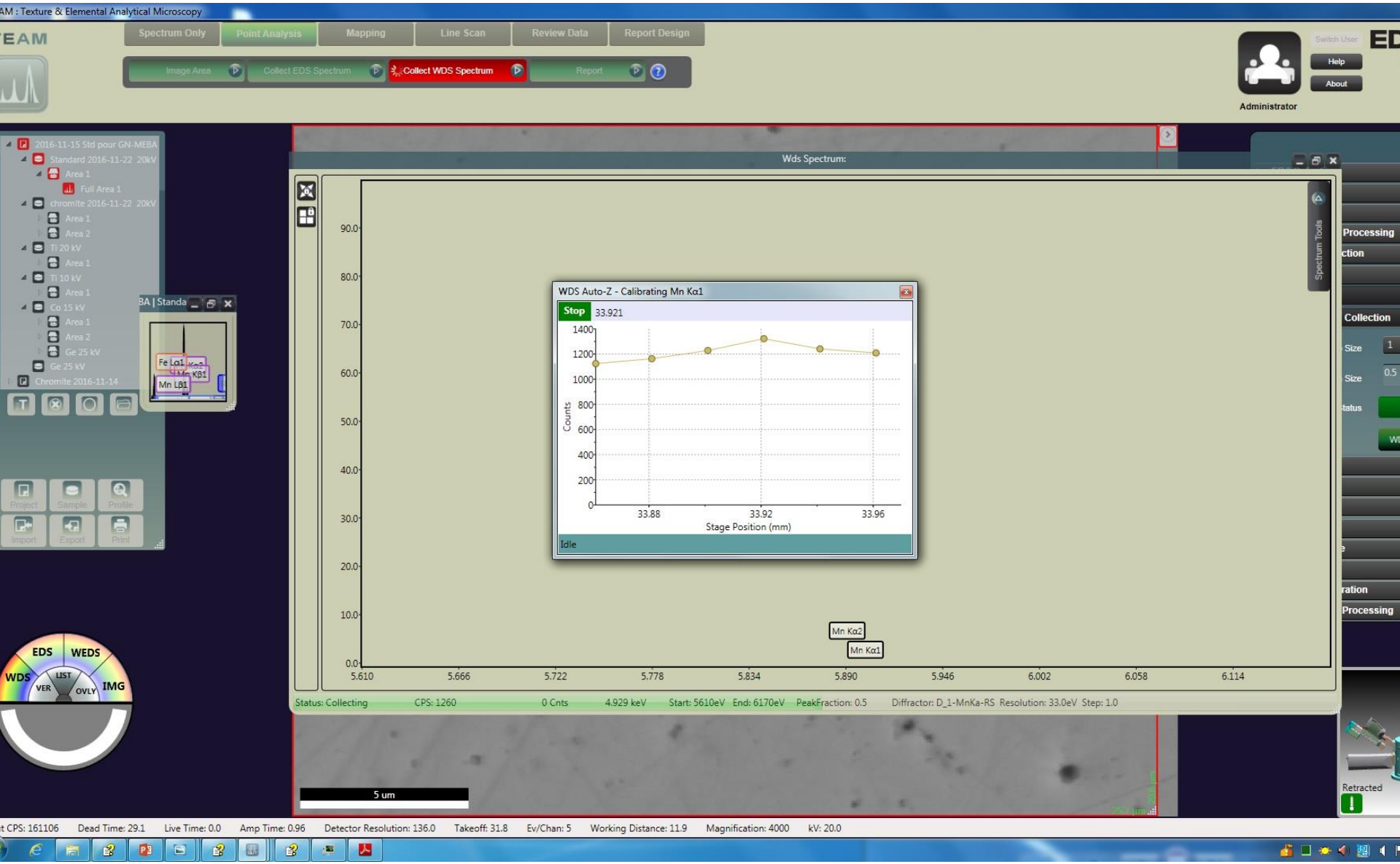
Check Height Mn Ka



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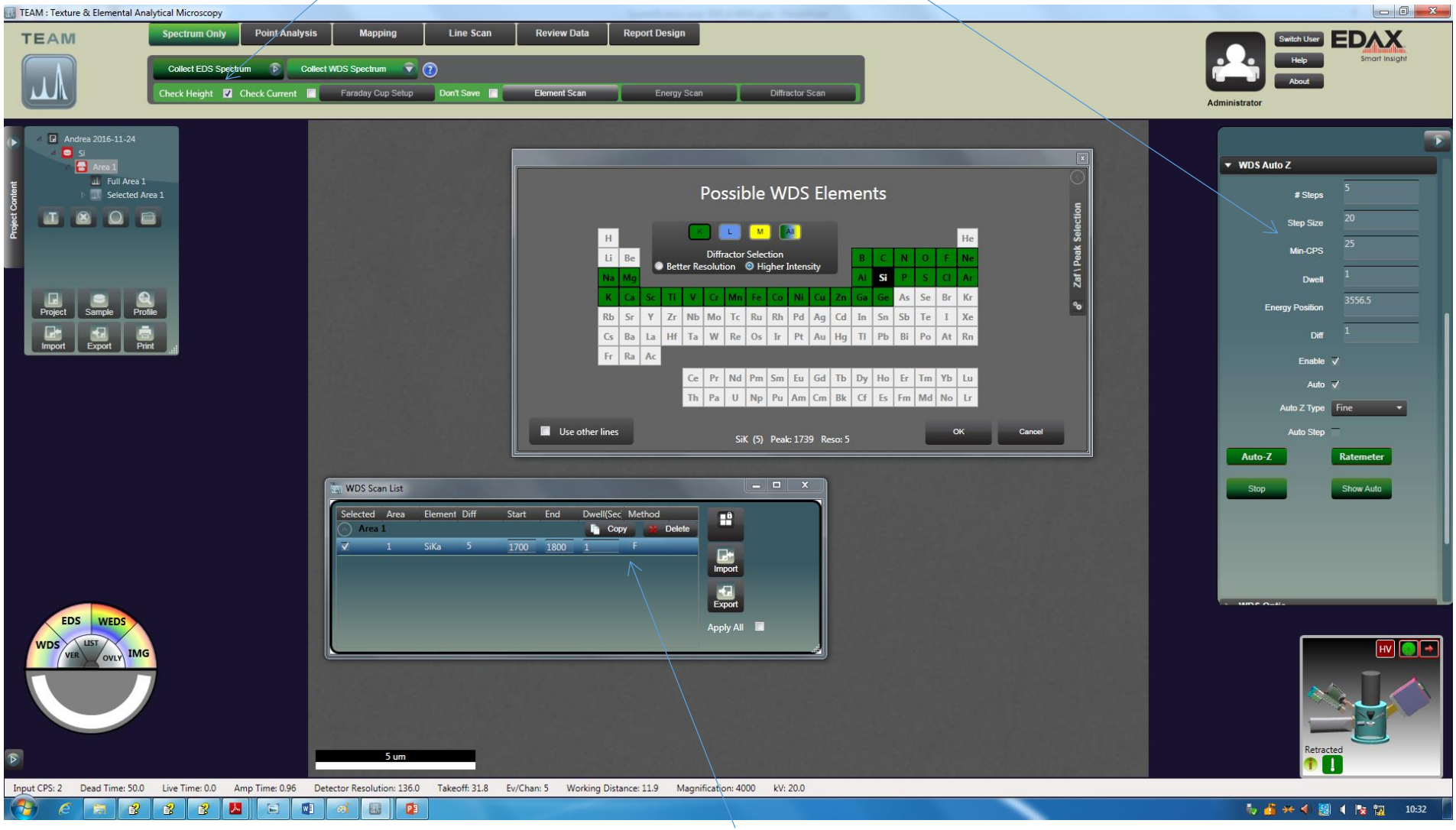
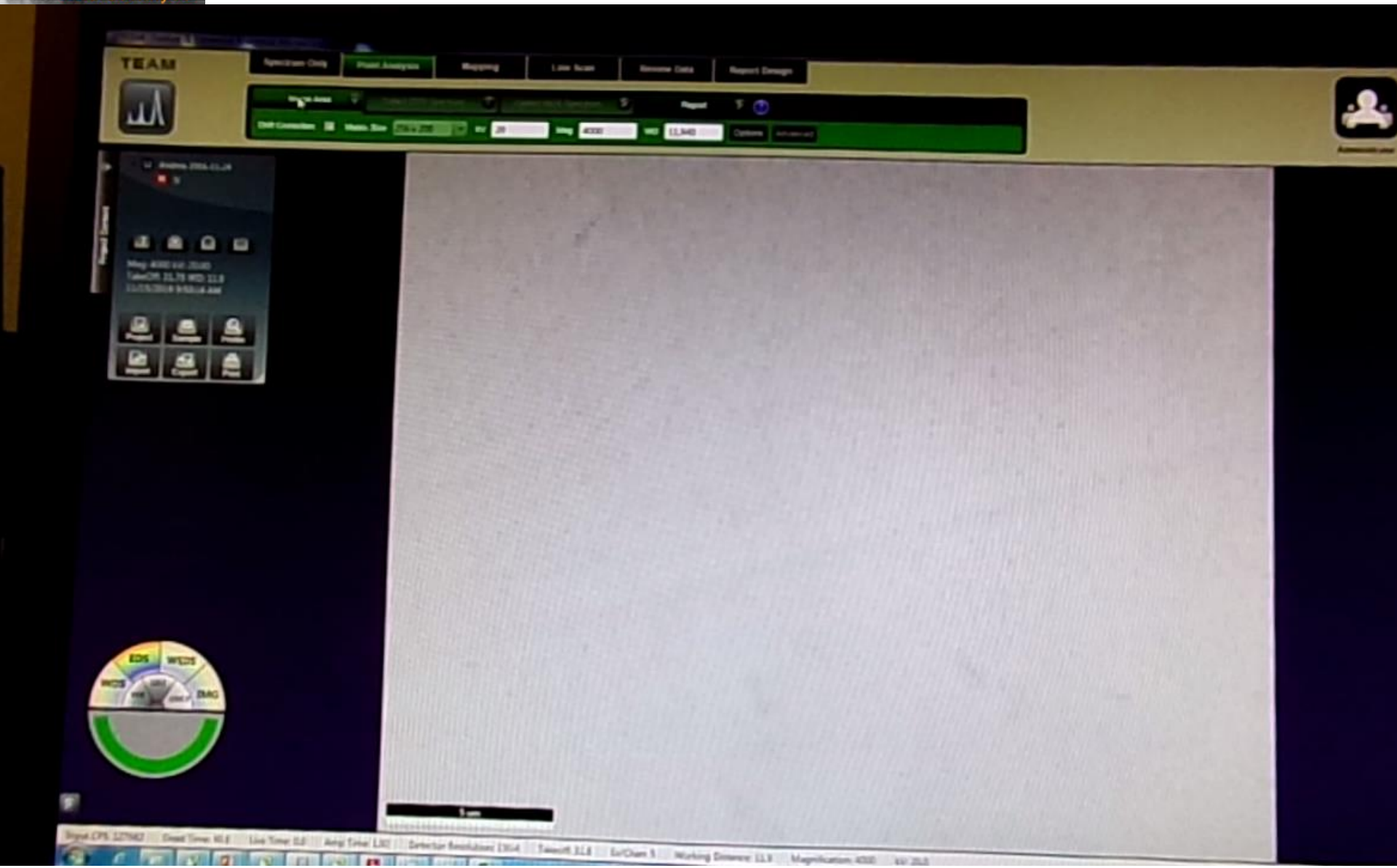
Check height (pas de 20 µm en Z par défaut)



Mode FULL



Film

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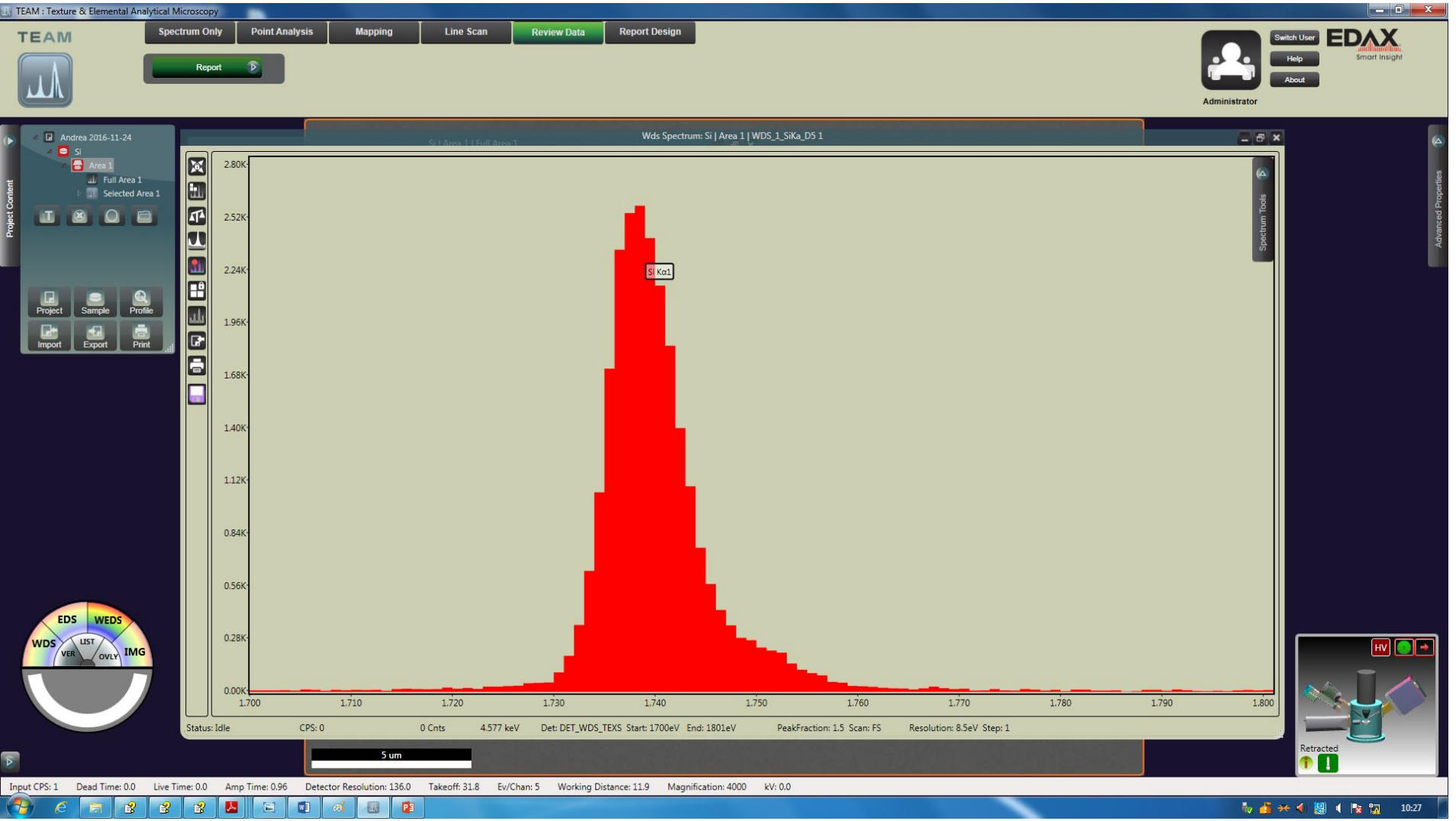
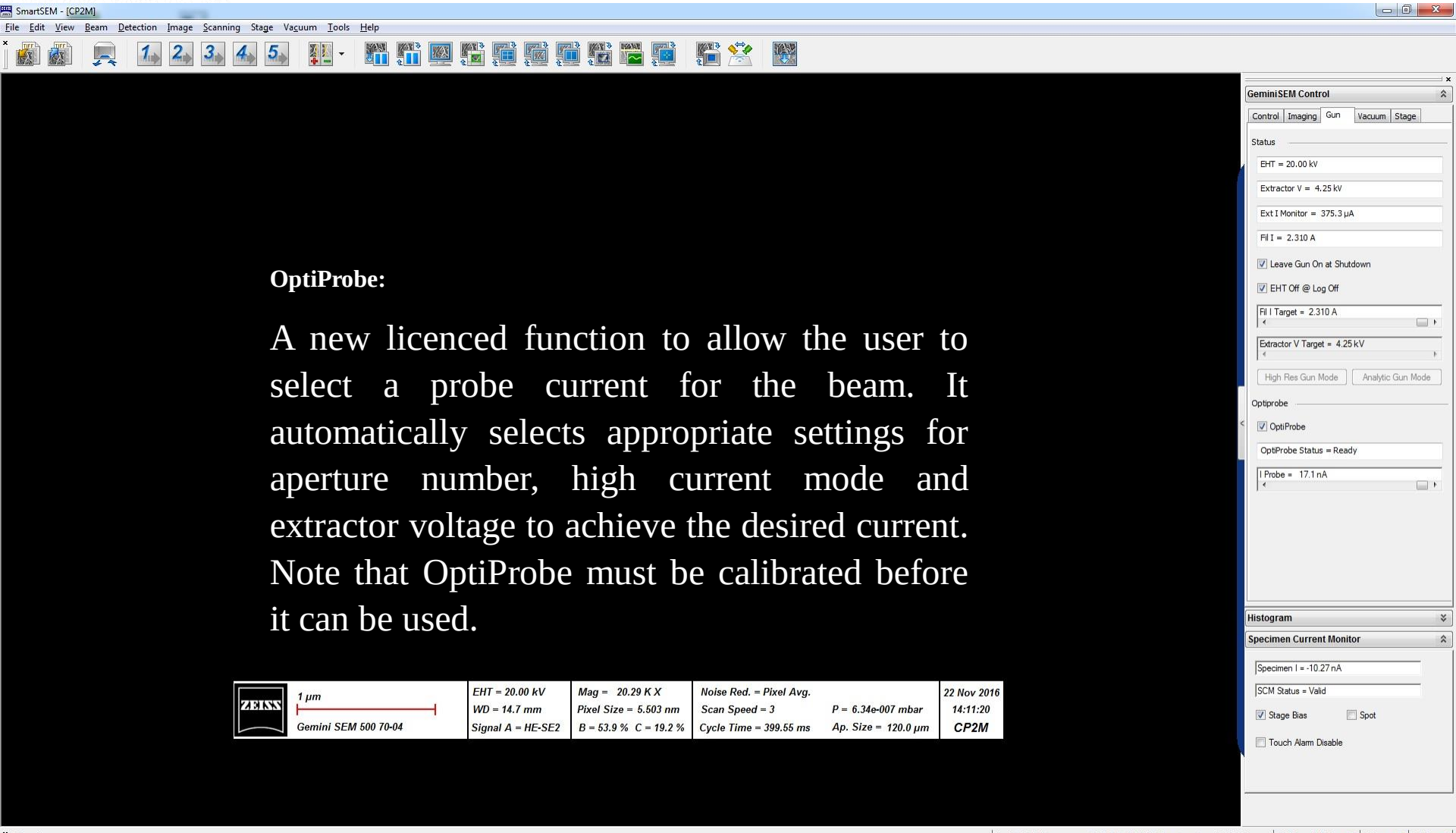
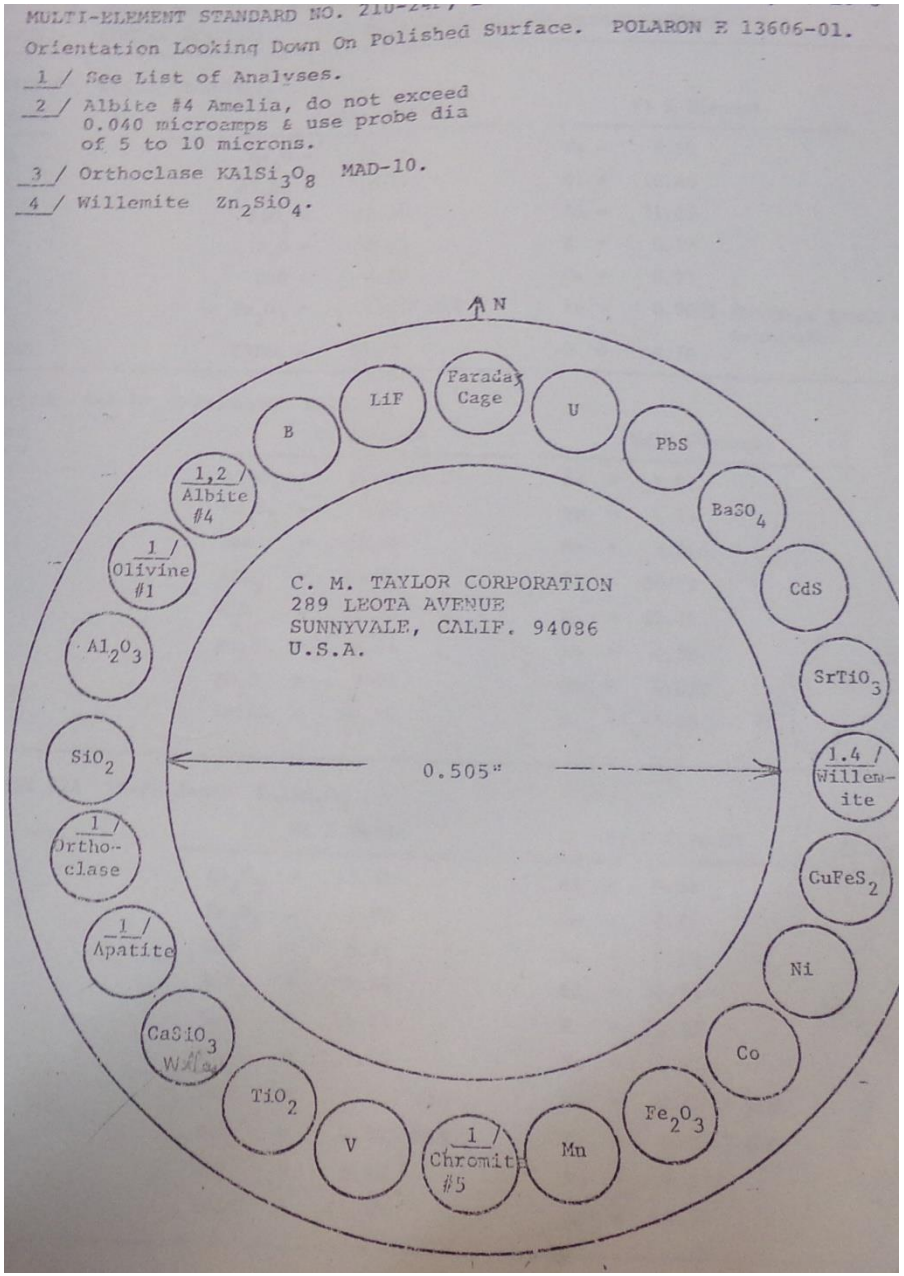


QUANTIFICATION



STANDARDS PURS ET MULTI-ELEMENTS



Choix: Chromite= (Fe, Mg, Al)Cr2O4

CHROMITE #5 AUSTRALIA

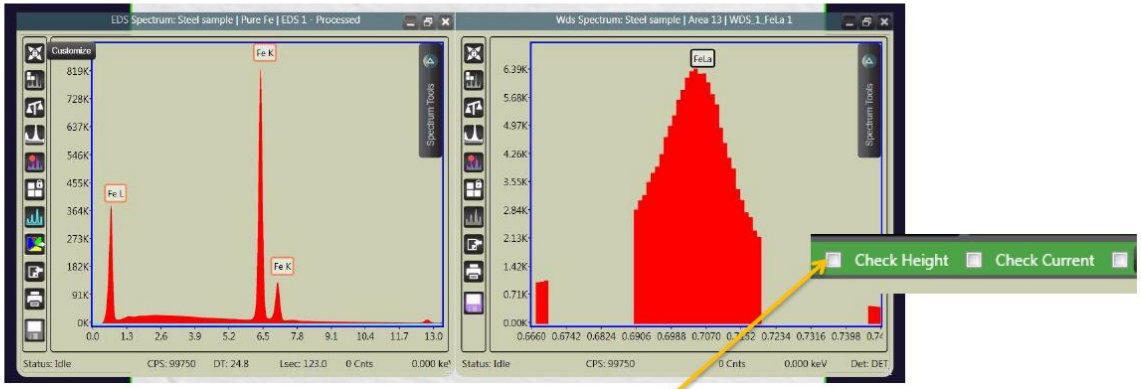
Element	Wt % Oxide			Wt % Element		
Al	Al_2O_3	=	23.91	Al	=	12.65
Cr	Cr_2O_3	=	45.65	Cr	=	31.23
Fe	as FeO	=	12.72	Fe	=	9.89
Ti	TiO_2	=	0.12	→ Ti	=	0.072
Zn	ZnO	=	0.01? NLD	Zn	=	0.008? NLD
Mg	MgO	=	17.26	Mg	=	10.41
Mn	MnO	=	0.13	→ Mn	=	0.10
Ni	NiO	=	0.17	→ Ni	=	0.134
V	V_2O_3	=	0.17	→ V	=	0.116
Si	SiO_2	=	0.03	→ Si	=	0.014
TOTAL	TOTAL	=	100.17	O	=	35.54

WDS quantification using TEAM SW.pdf - Adobe Reader

File Edit View Window Help

Open | 7 / 22 | 115% | Tools | Fill & Sign | Comment

Pure element standards - 1



Let the software optimize the height (have it selected) and measure the standard. Be sure to first have the beam current measured (use 'check current' option; see also slides 4 +5). Open spectra as a group (from project tree, see picture at left) . Above EDS spectrum (left) and WDS spectrum (right) for pure Fe are shown.

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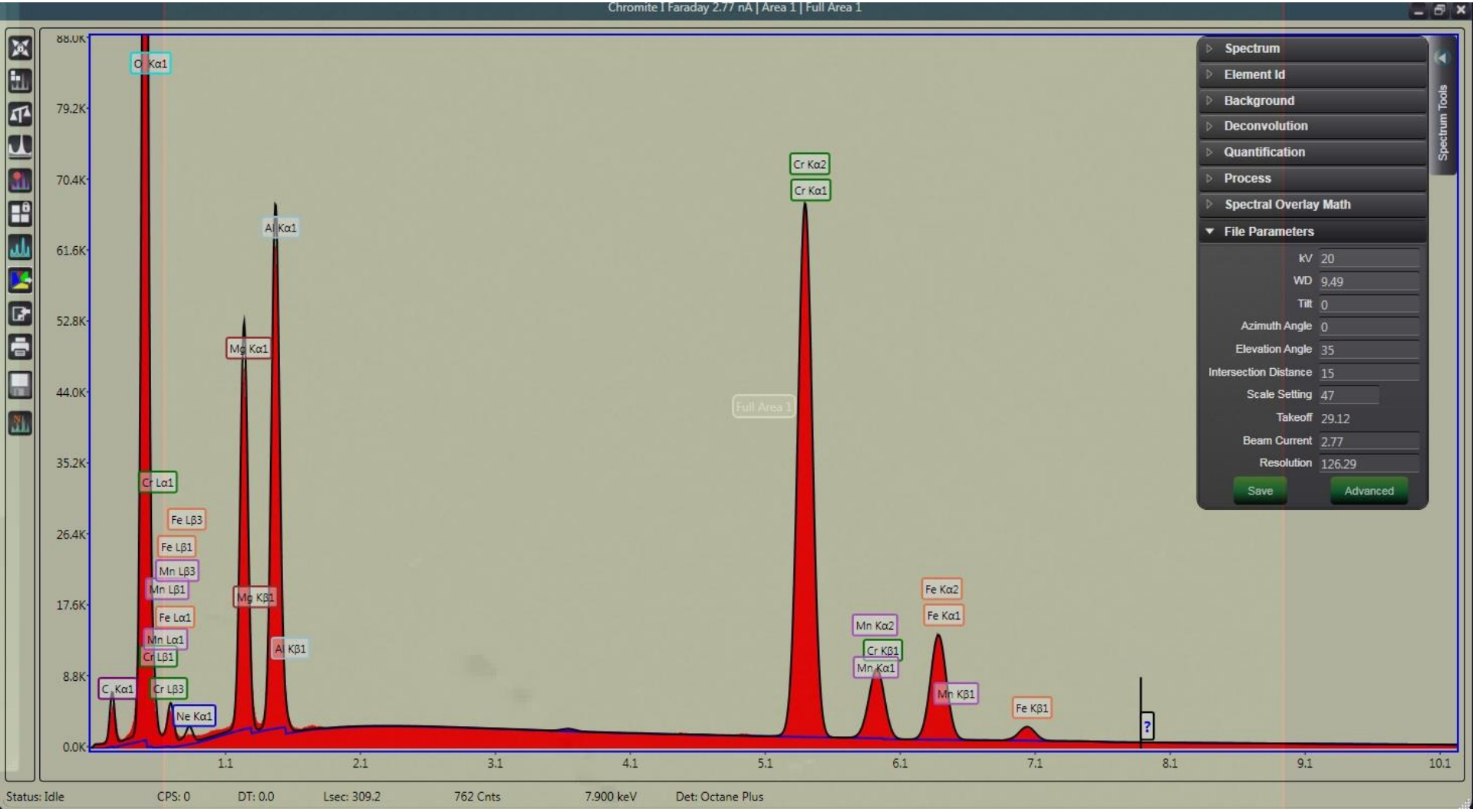
EDAX Smart Insight

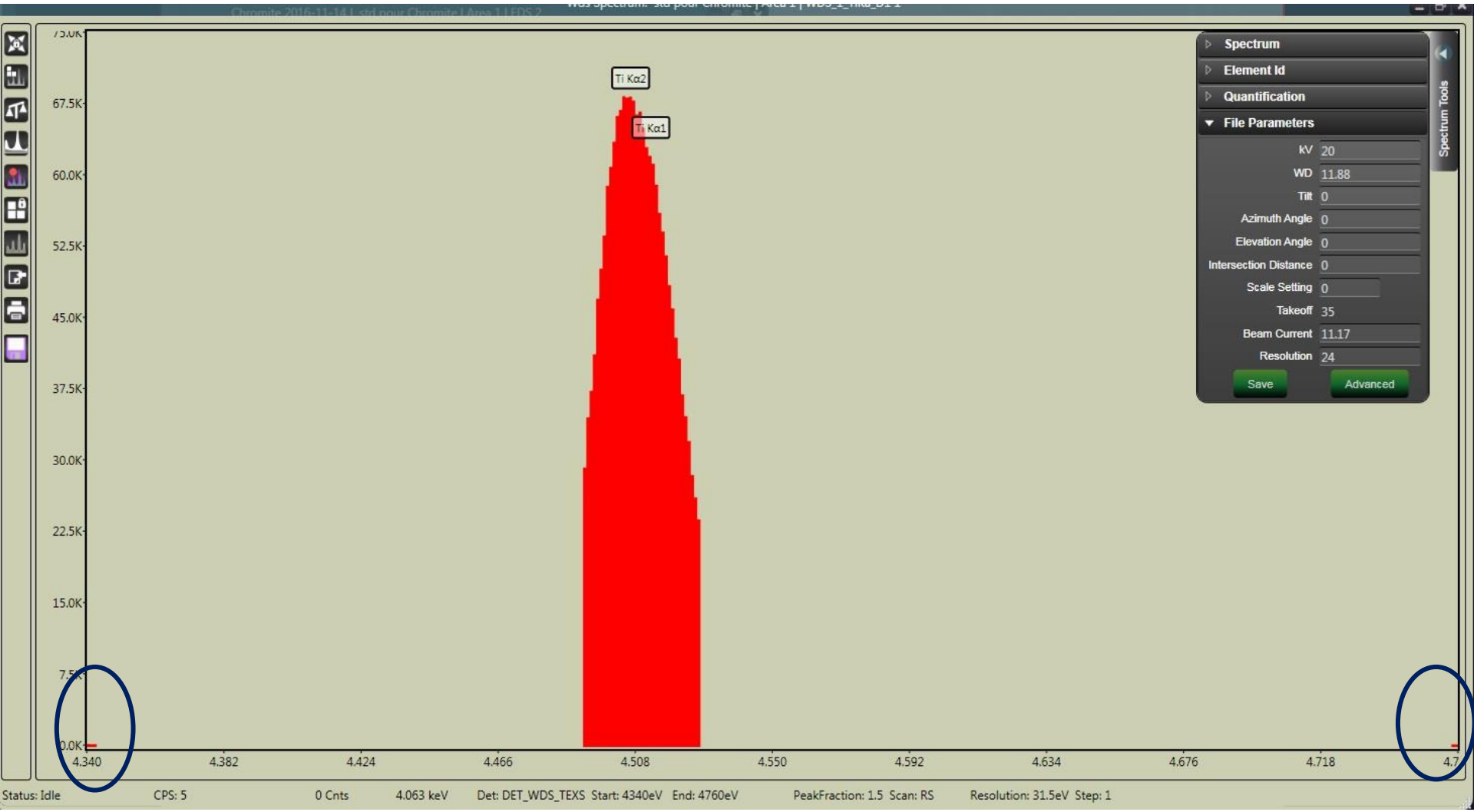
7

Source: EDAX- Harry Verhulst

Elements des standards et des échantillons – mesure dans les mêmes conditions (step size, scan size, début et fin du balayage en énergie).
MAIS: les conditions sont déterminées à partir du spectre EDS de la Chromite

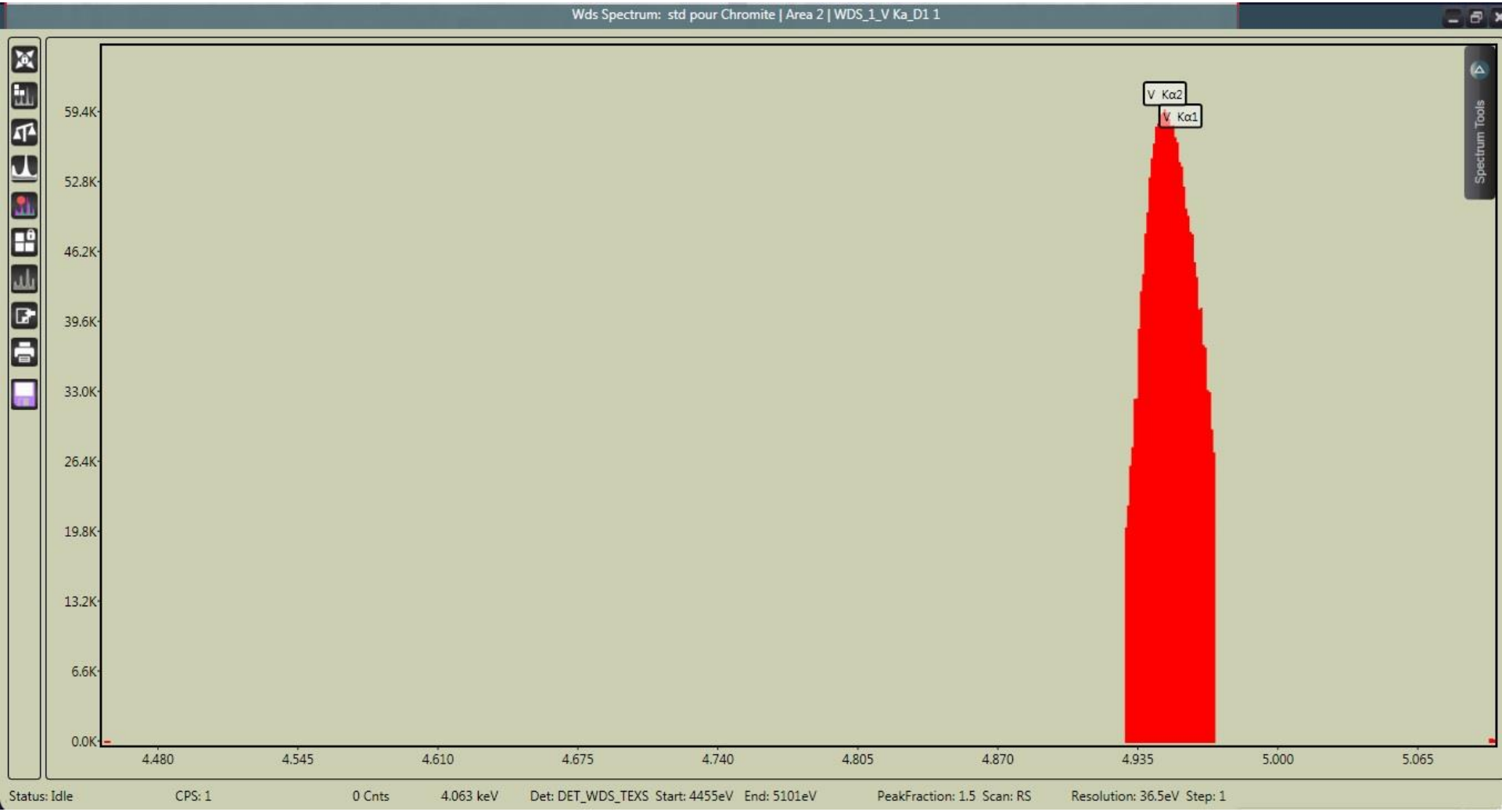


Standard Ti

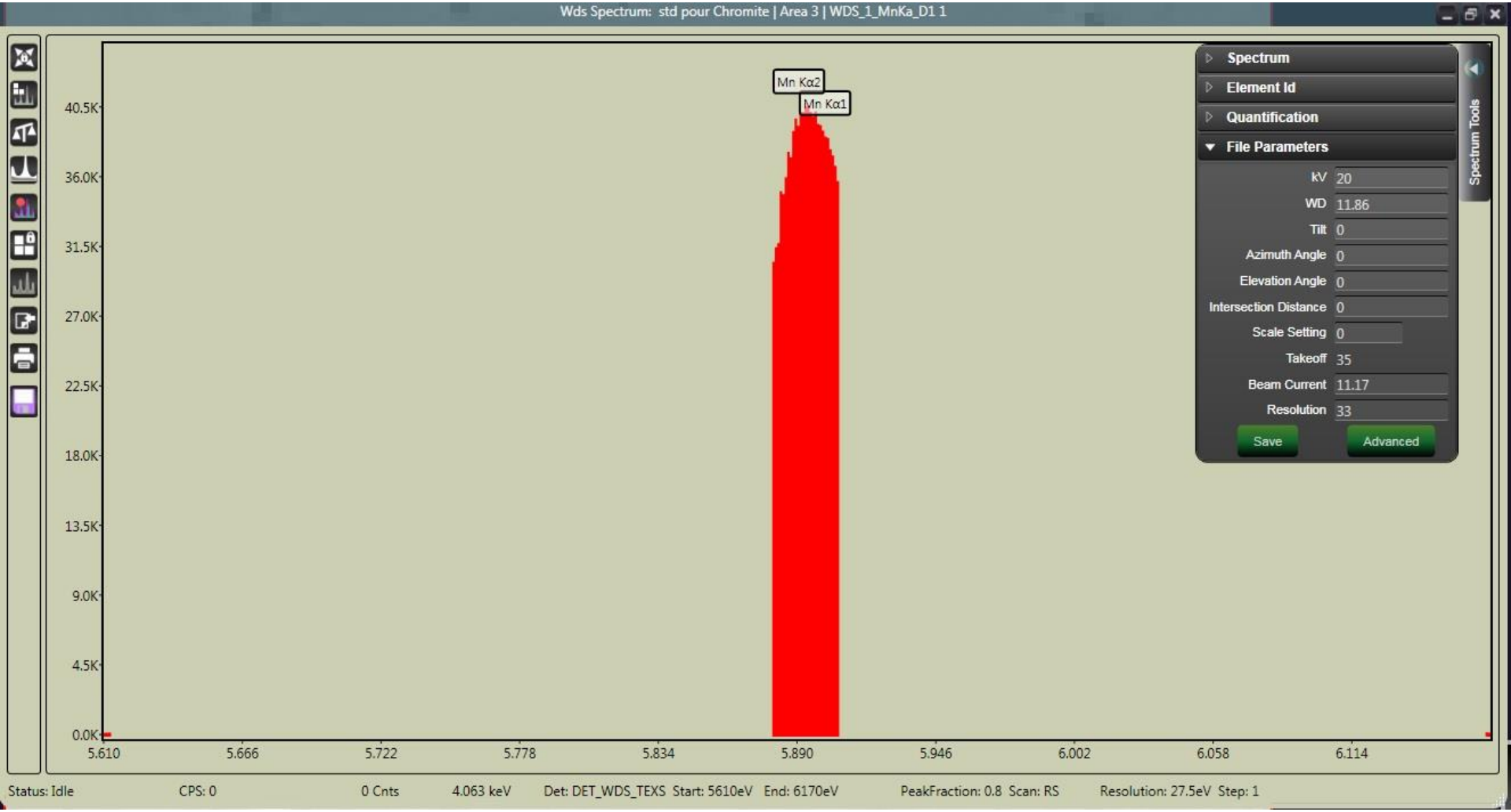


La raie TiKα en R - reduced scanning mode. Les bruits de fond avant et après le pic sont encerclés en bleu.

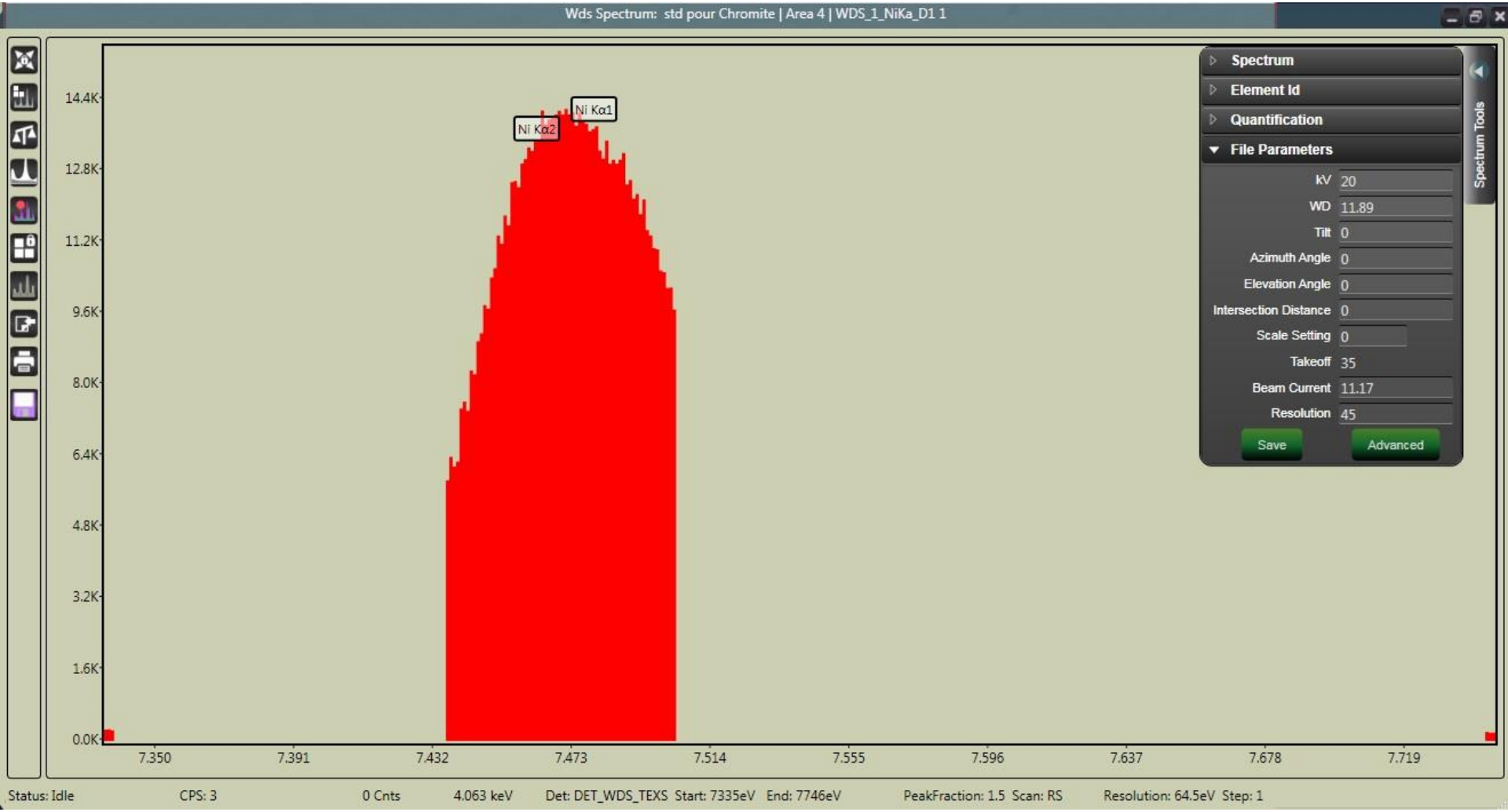
Standard V


Wds Spectrum: std pour Chromite | Area 2 | WDS_1_V Ka_D1 1
V Kα2
V Kα1
Spectrum Tools
59.4K
52.8K
46.2K
39.6K
33.0K
26.4K
19.8K
13.2K
6.6K
0.0K
4.480
4.545
4.610
4.675
4.740
4.805
4.870
4.935
5.000
5.065
Status: Idle
CPS: 1
0 Cnts
4.063 keV
Det: DET_WDS_TEXS Start: 4455eV End: 5101eV
PeakFraction: 1.5 Scan: RS
Resolution: 36.5eV Step: 1

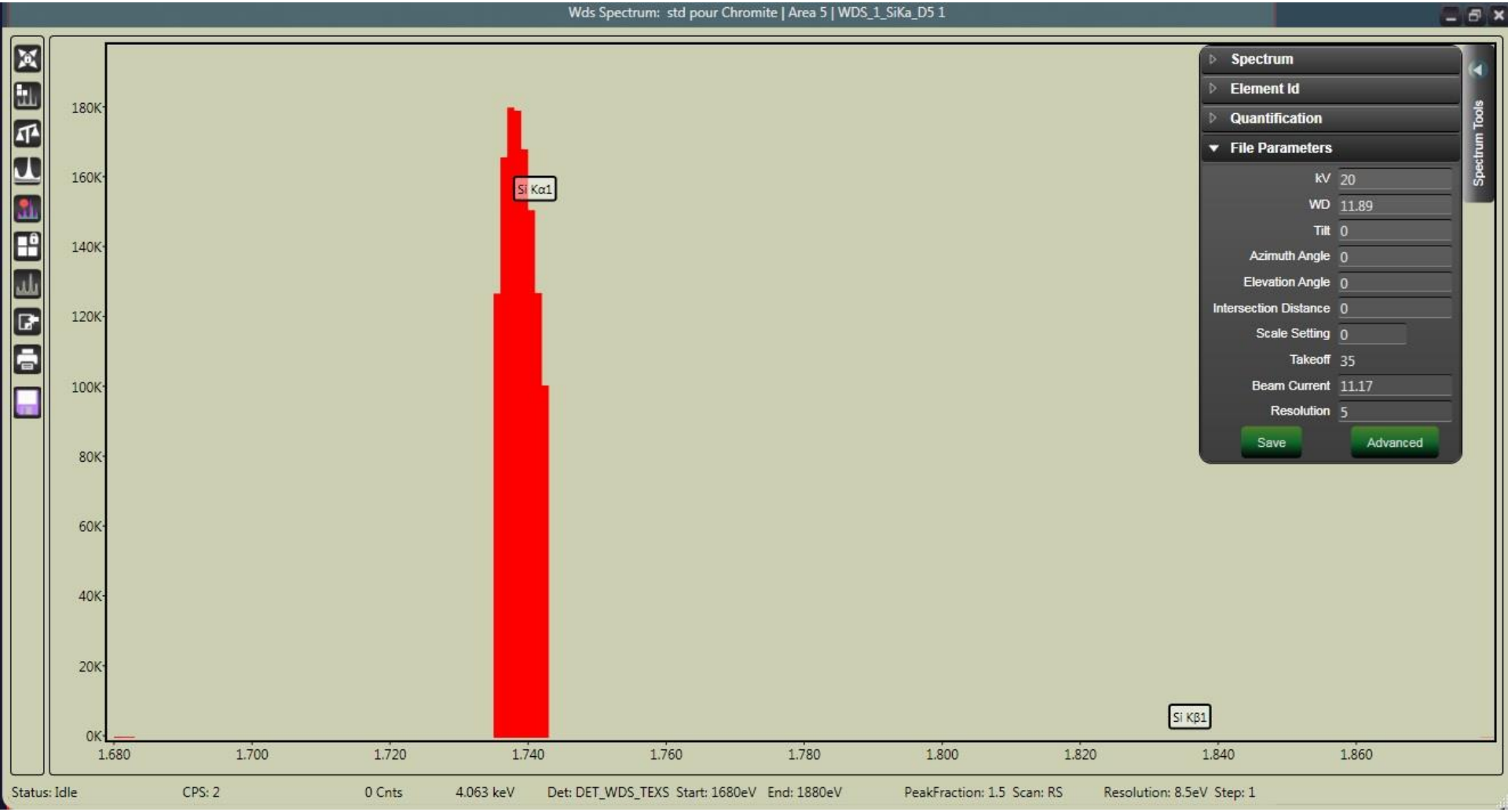
Standard Mn


Wds Spectrum: std pour Chromite | Area 3 | WDS_1_MnKa_D1 1
Mn Kα2
Mn Kα1
40.5K
36.0K
31.5K
27.0K
22.5K
18.0K
13.5K
9.0K
4.5K
0.0K
5.610
5.666
5.722
5.778
5.834
5.890
5.946
6.002
6.058
6.114
Spectrum
Element Id
Quantification
File Parameters
kV 20
WD 11.86
Tilt 0
Azimuth Angle 0
Elevation Angle 0
Intersection Distance 0
Scale Setting 0
Takeoff 35
Beam Current 11.17
Resolution 33
Save
Advanced
Spectrum Tools
Status: Idle
CPS: 0
0 Cnts
4.063 keV
Det: DET_WDS_TEXS Start: 5610eV End: 6170eV
PeakFraction: 0.8 Scan: RS
Resolution: 27.5eV Step: 1

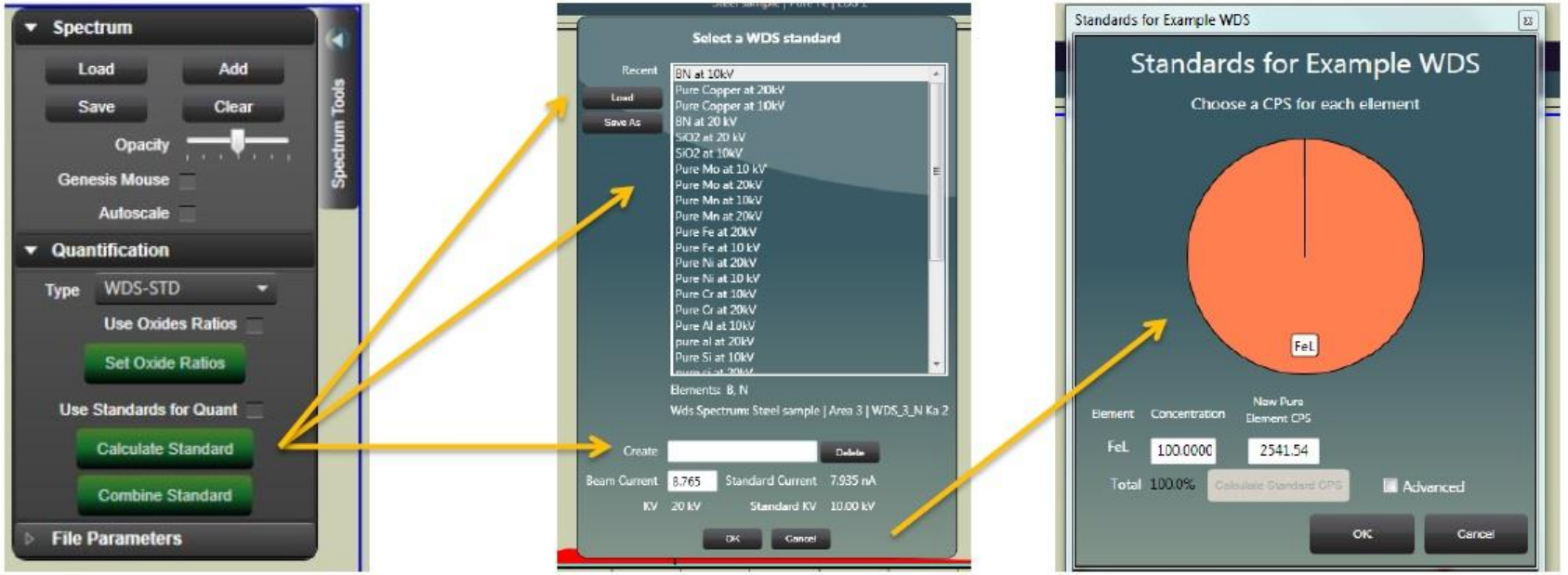
Standard Ni


Wds Spectrum: std pour Chromite | Area 4 | WDS_1_NiKa_D1 1
Ni Kα1
Ni Kα2
14.4K
12.8K
11.2K
9.6K
8.0K
6.4K
4.8K
3.2K
1.6K
0.0K
7.350
7.391
7.432
7.473
7.514
7.555
7.596
7.637
7.678
7.719
Spectrum
Element Id
Quantification
File Parameters
kV 20
WD 11.89
Tilt 0
Azimuth Angle 0
Elevation Angle 0
Intersection Distance 0
Scale Setting 0
Takeoff 35
Beam Current 11.17
Resolution 45
Save
Advanced
Spectrum Tools
Status: Idle
CPS: 3
0 Cnts
4.063 keV
Det: DET_WDS_TEXS Start: 7335eV End: 7746eV
PeakFraction: 1.5 Scan: RS
Resolution: 64.5eV Step: 1

Standard Si

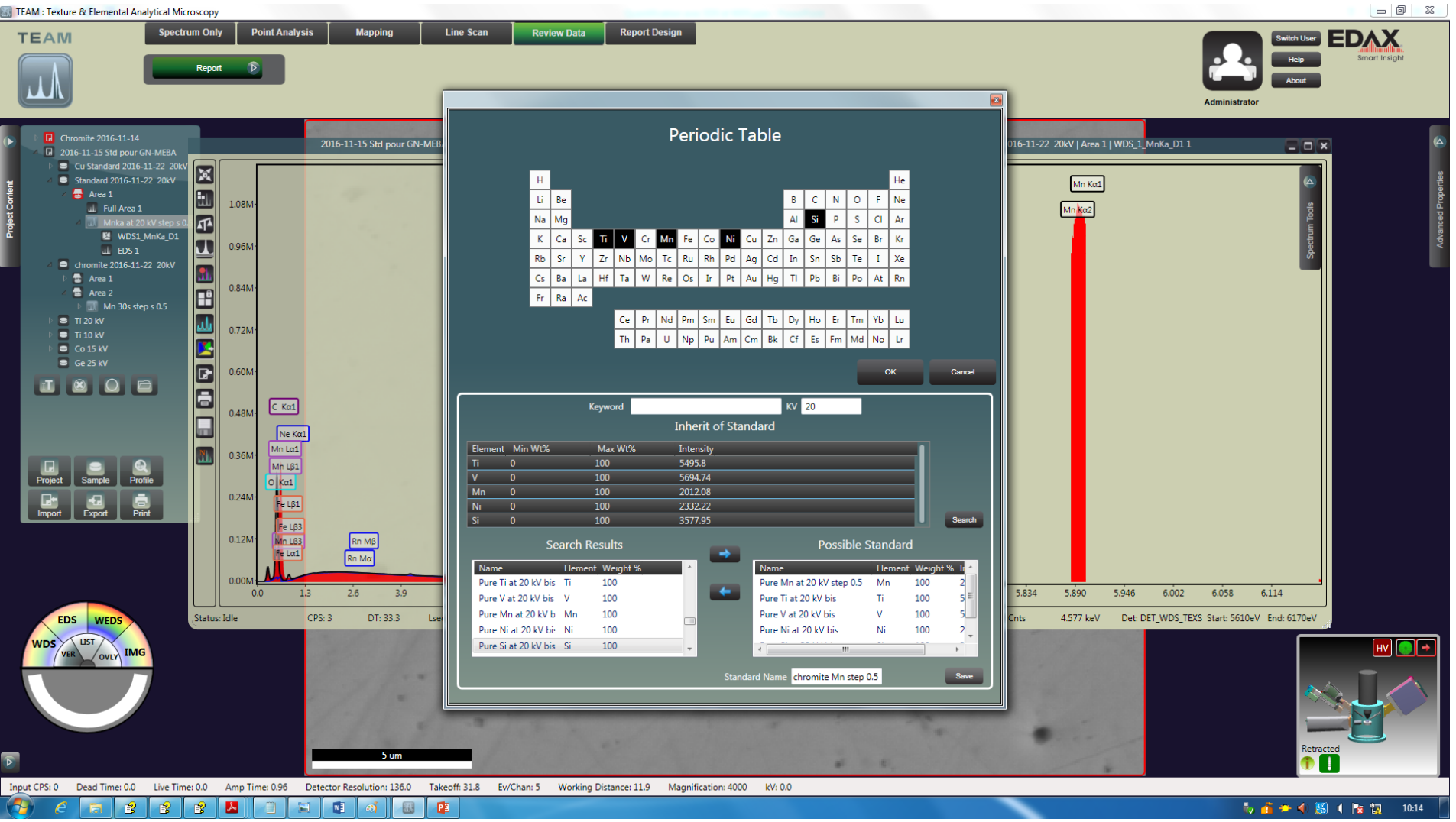

Wds Spectrum: std pour Chromite | Area 5 | WDS_1_SiKa_D5 1
Si Kα1
Si Kβ1
180K
160K
140K
120K
100K
80K
60K
40K
20K
0K
1.680
1.700
1.720
1.740
1.760
1.780
1.800
1.820
1.840
1.860
Spectrum
Element Id
Quantification
File Parameters
kV 20
WD 11.89
Tilt 0
Azimuth Angle 0
Elevation Angle 0
Intersection Distance 0
Scale Setting 0
Takeoff 35
Beam Current 11.17
Resolution 5
Save
Advanced
Spectrum Tools
Status: Idle
CPS: 2
0 Cnts
4.063 keV
Det: DET_WDS_TEXS Start: 1680eV End: 1880eV
PeakFraction: 1.5 Scan: RS
Resolution: 8.5eV Step: 1

Pure element standards - 2

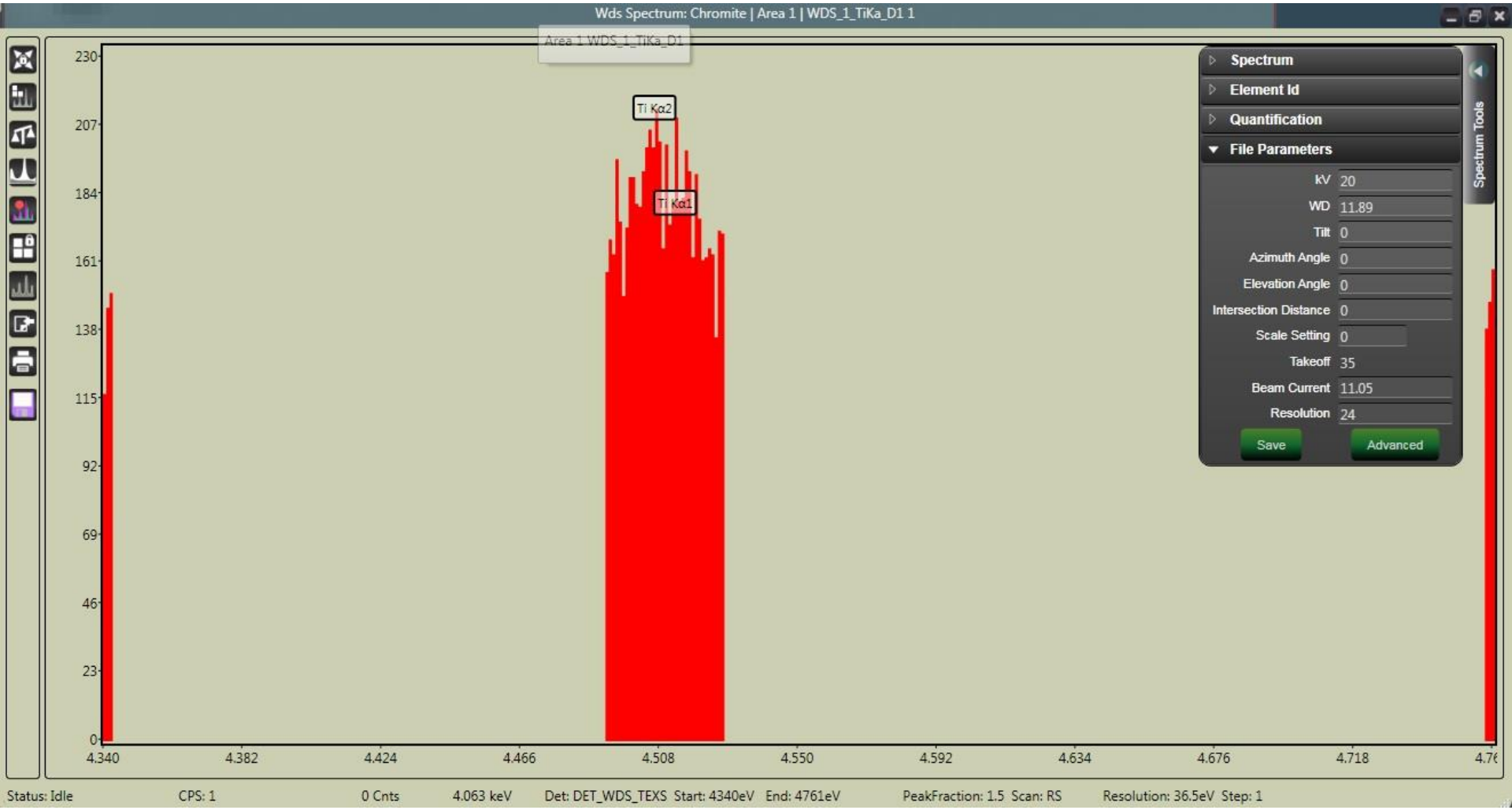


After spectra have been recorded and then loaded from project tree, go to Spectrum tools and then quantification. Select 'Calculate Standard' and either open an existing file or type in a name at 'create'. Make sure beam current shown is correct or edit this value. After pressing ok, pie appears. Just make sure concentration value is at 100 and press 'Calculate Standard cps' and then ok. Now pure intensity value for FeLa is setup. Repeat the same procedure using EDS spectrum (simultaneously recorded with WDS run).

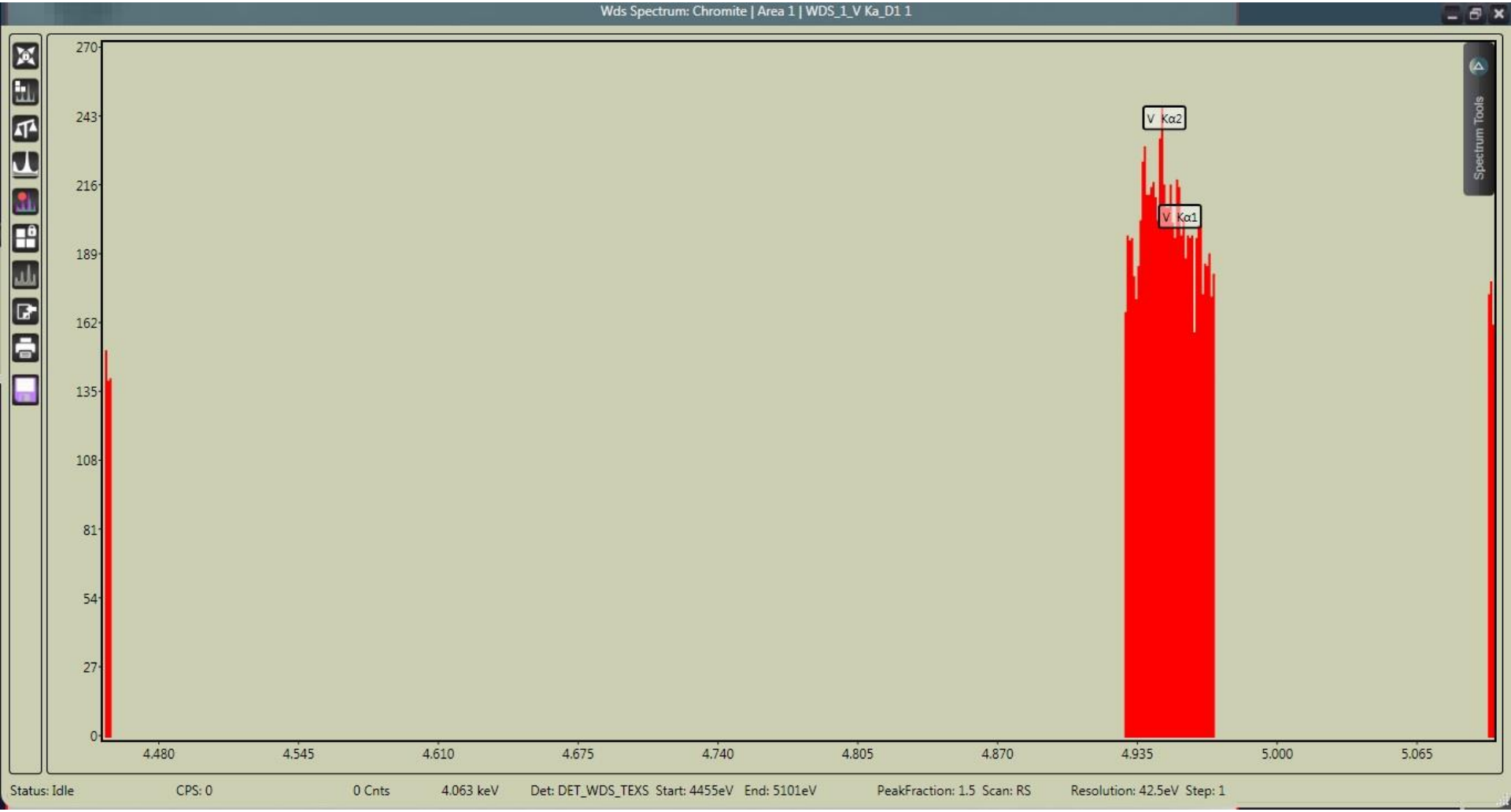
Source: EDAX- Harry Verhulst



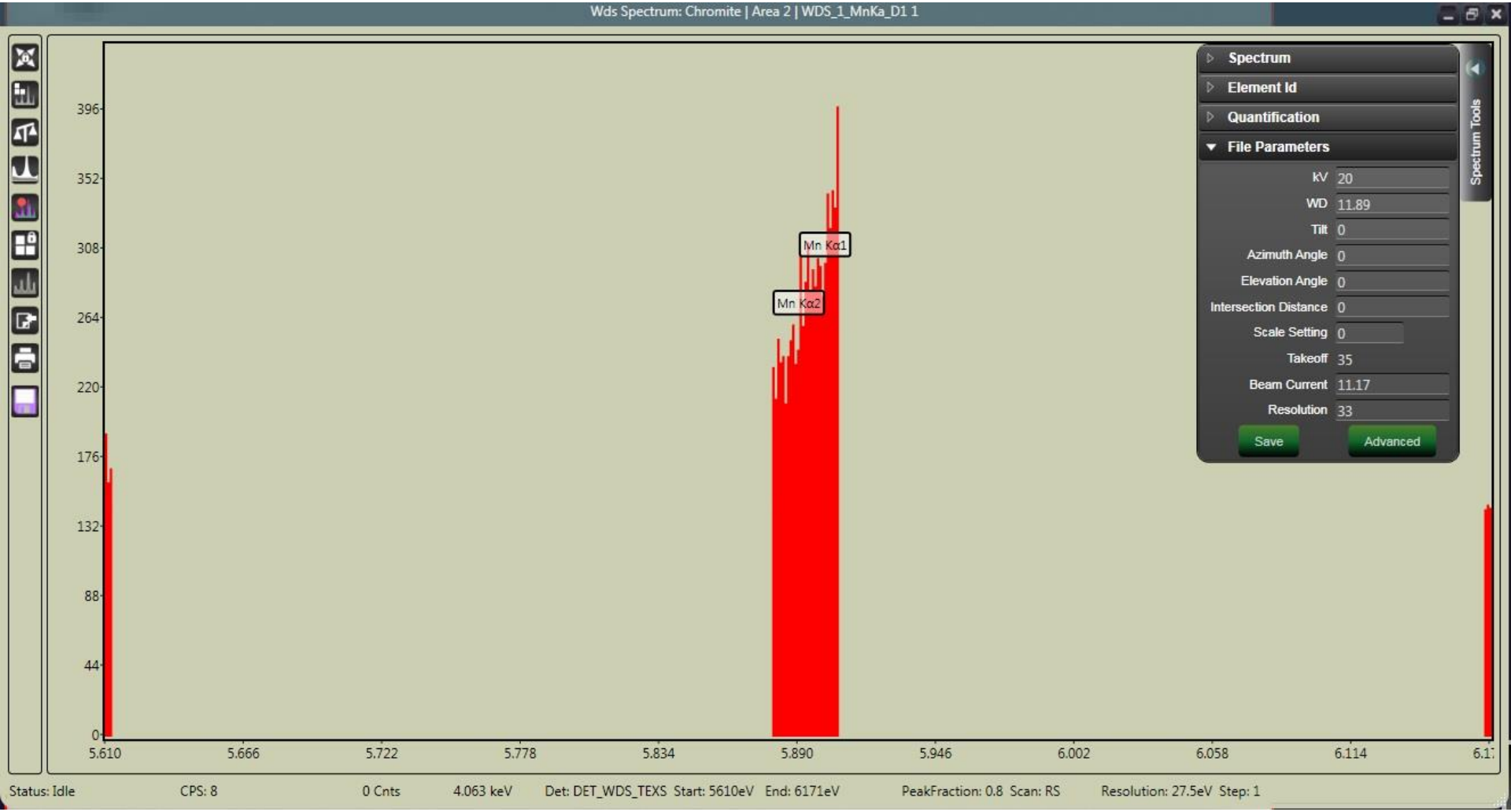
Chromite Ti


Wds Spectrum: Chromite | Area 1 | WDS_1_TiKa_D1 1
Area 1 WDS_1_TiKa_D1
Ti Kα2
Ti Kα1
230
207
184
161
138
115
92
69
46
23
0
4.340
4.382
4.424
4.466
4.508
4.550
4.592
4.634
4.676
4.718
Spectrum
Element Id
Quantification
File Parameters
kV 20
WD 11.89
Tilt 0
Azimuth Angle 0
Elevation Angle 0
Intersection Distance 0
Scale Setting 0
Takeoff 35
Beam Current 11.05
Resolution 24
Save
Advanced
Spectrum Tools
Status: Idle
CPS: 1
0 Cnts
4.063 keV
Det: DET_WDS_TEXS Start: 4340eV End: 4761eV
PeakFraction: 1.5 Scan: RS
Resolution: 36.5eV Step: 1

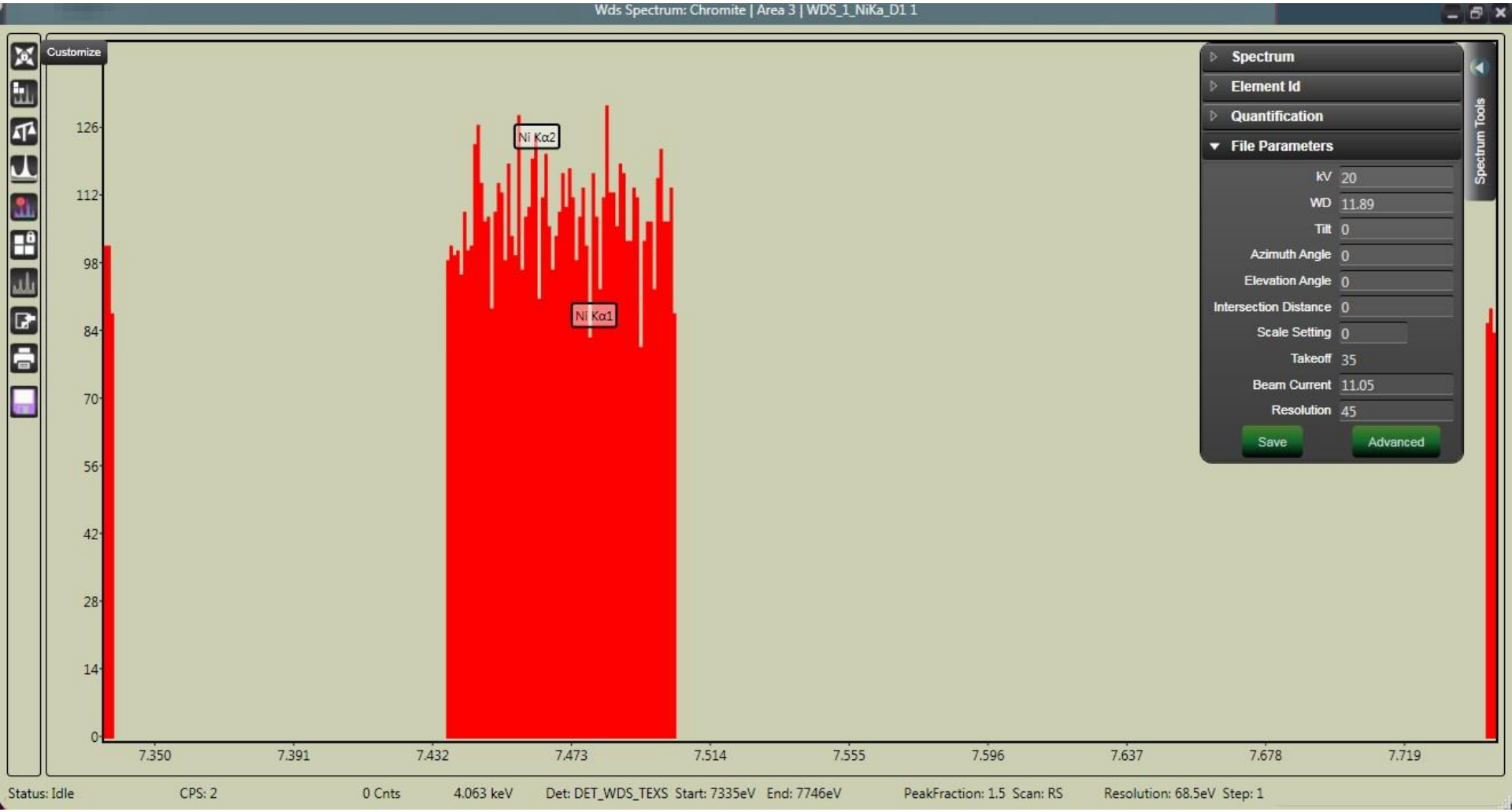
Chromite V


Wds Spectrum: Chromite | Area 1 | WDS_1_V Ka_D1 1
V Kα2
V Kα1
Spectrum Tools
270
243
216
189
162
135
108
81
54
27
0
4.480
4.545
4.610
4.675
4.740
4.805
4.870
4.935
5.000
5.065
Status: Idle
CPS: 0
0 Cnts
4.063 keV
Det: DET_WDS_TEXS Start: 4455eV End: 5101eV
PeakFraction: 1.5 Scan: RS
Resolution: 42.5eV Step: 1

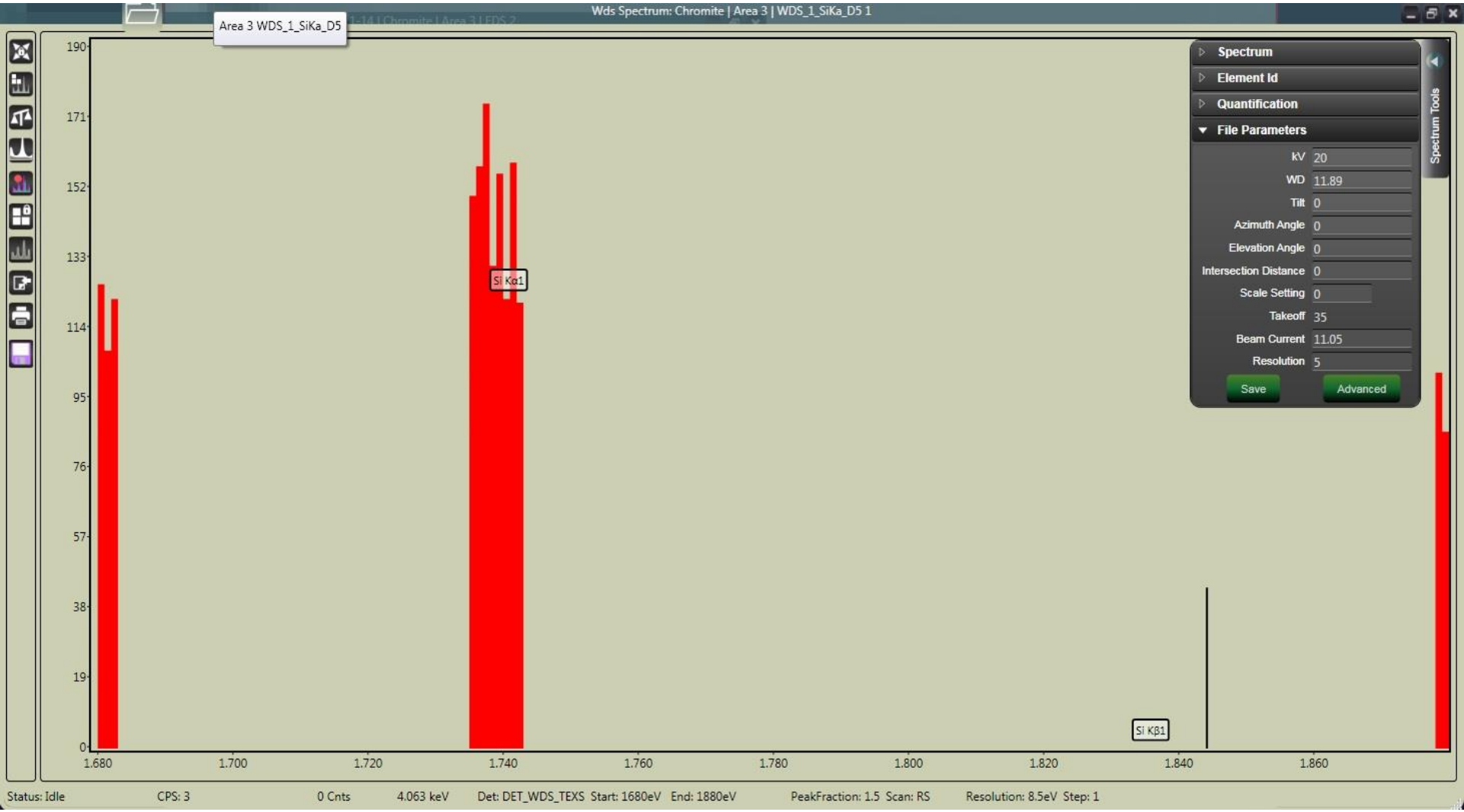
Chromite Mn


Wds Spectrum: Chromite | Area 2 | WDS_1_MnKa_D1 1
Mn Kα1
Mn Kα2
Spectrum
Element Id
Quantification
File Parameters
kV 20
WD 11.89
Tilt 0
Azimuth Angle 0
Elevation Angle 0
Intersection Distance 0
Scale Setting 0
Takeoff 35
Beam Current 11.17
Resolution 33
Save
Advanced
Spectrum Tools
396
352
308
264
220
176
132
88
44
0
5.610
5.666
5.722
5.778
5.834
5.890
5.946
6.002
6.058
6.114
Status: Idle
CPS: 8
0 Cnts
4.063 keV
Det: DET_WDS_TEXS Start: 5610eV End: 6171eV
PeakFraction: 0.8 Scan: RS
Resolution: 27.5eV Step: 1

Chromite Ni


Wds Spectrum: Chromite | Area 3 | WDS_1_NiKa_D1 1
Customize
Ni Kα2
Ni Kα1
126
112
98
84
70
56
42
28
14
0
7.350
7.391
7.432
7.473
7.514
7.555
7.596
7.637
7.678
7.719
Spectrum
Element Id
Quantification
File Parameters
kV 20
WD 11.89
Tilt 0
Azimuth Angle 0
Elevation Angle 0
Intersection Distance 0
Scale Setting 0
Takeoff 35
Beam Current 11.05
Resolution 45
Save
Advanced
Spectrum Tools
Status: Idle
CPS: 2
0 Cnts
4.063 keV
Det: DET_WDS_TEXS Start: 7335eV End: 7746eV
PeakFraction: 1.5 Scan: RS
Resolution: 68.5eV Step: 1

Chromite Si


Wds Spectrum: Chromite | Area 3 | WDS_1_SiKa_D5 1
Area 3 WDS_1_SiKa_D5
Si Kα1
Si Kβ1
Spectrum
Element Id
Quantification
File Parameters
kV 20
WD 11.89
Tilt 0
Azimuth Angle 0
Elevation Angle 0
Intersection Distance 0
Scale Setting 0
Takeoff 35
Beam Current 11.05
Resolution 5
Save
Advanced
Spectrum Tools
190
171
152
133
114
95
76
57
38
19
0
1.680
1.700
1.720
1.740
1.760
1.780
1.800
1.820
1.840
1.860
Status: Idle
CPS: 3
0 Cnts
4.063 keV
Det: DET_WDS_TEXS Start: 1680eV End: 1880eV
PeakFraction: 1.5 Scan: RS
Resolution: 8.5eV Step: 1

Quantification

Trois possibilités:

- avec standards WDS
- avec standards WDS et EDS
- avec standards WDS et EDS sans standards ("standardless")

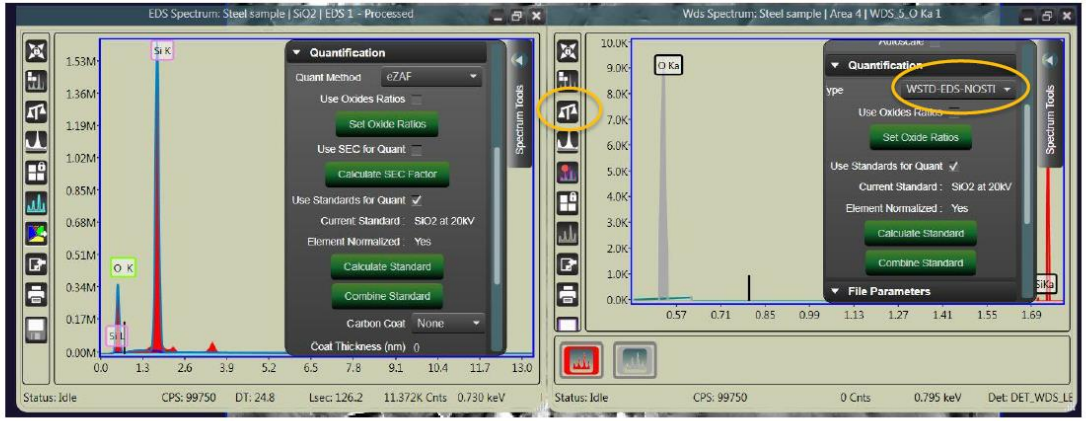
normalisée ou non-normalisée

WDS quantification using TEAM SW.pdf - Adobe Reader

File Edit View Window Help

Open 20 / 22 115% Tools Fill & Sign Comment

WDS standards and EDS stndless - 1



Have the correct pure intensity file loaded for WDS and select 'WSTD-EDS NOSTD' or 'combine WDS standards with EDS standardless'.

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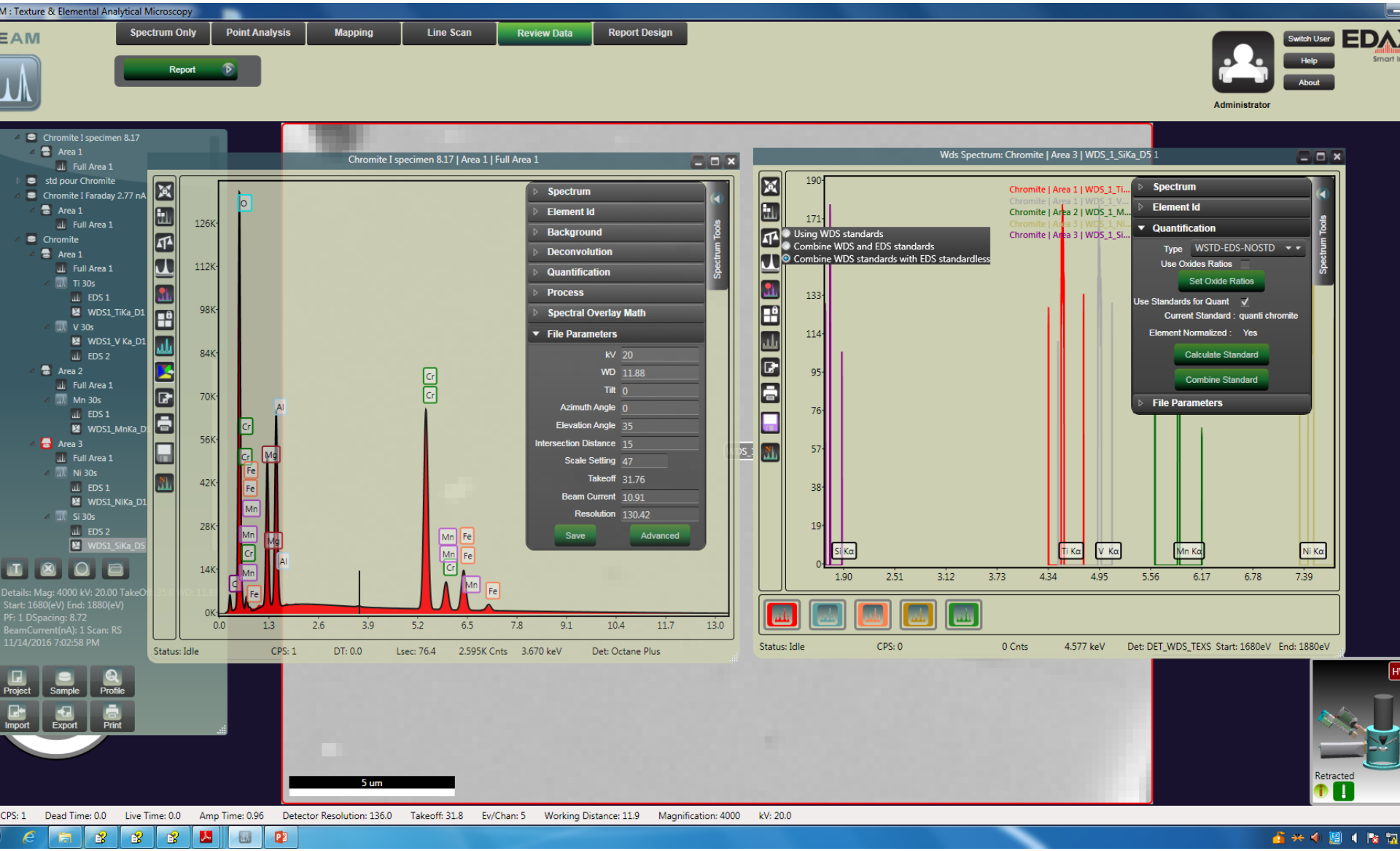
EDAX Smart Insight

Source: EDAX- Harry Verhulst



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NORMALISÉE

WDS Quant Smart Quant Results with Standards (quanti chromite-Element Normalized : Yes)

File Quant Method View Tools

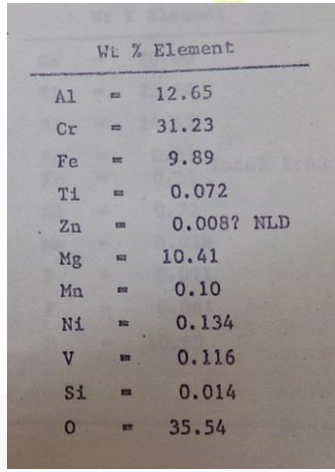
Drag a column header here to group by that column

using	Element	Weight %	Atomic %	Error %	Net Int.	K Ratio	Z	A	F	Label	SpecLabel
☐	O K	32.21	53.11	0.06	22156.97	0.2674	1.0789	0.4780	1.0017		Chromite I specimen 8.17 | Area 1 | Full Area 1
☐	Mg K	11.24	12.19	0.08	9719.41	0.0652	1.0347	0.3742	1.0025		Chromite I specimen 8.17 | Area 1 | Full Area 1
☐	Al K	13.47	13.18	0.07	13377.15	0.0891	1.0041	0.4384	1.0006		Chromite I specimen 8.17 | Area 1 | Full Area 1
☑	Si K	0.05	0.05	16.21	9.34	0.0004	1.0333	0.4876	1.0011	DET_WDS_TEXS_5	Chromite | Area 3 | WDS_1_SiKa_D5 1
☑	Ti K	0.08	0.04	7.88	45.61	0.0011	0.9190	0.9824	1.0946	DET_WDS_TEXS_1	Chromite | Area 1 | WDS_1_TiKa_D1 1
☑	V K	0.09	0.05	8.39	49.26	0.0012	0.8998	0.9929	1.0147	DET_WDS_TEXS_1	Chromite | Area 1 | WDS_1_V Ka_D1 1
☐	Cr K	32.36	16.42	0.02	22743.75	0.4425	0.9160	0.9997	1.0201		Chromite I specimen 8.17 | Area 1 | Full Area 1
☑	Mn K	0.37	0.18	3.34	108.92	0.0049	0.8992	1.0041	1.0002	DET_WDS_TEXS_1	Chromite | Area 2 | WDS_1_MnKa_D 1 1
☐	Fe K	9.99	4.72	0.02	4890.04	0.1264	0.9160	0.9508	1.0003		Chromite I specimen 8.17 | Area 1 | Full Area 1
☑	Ni K	0.15	0.07	12.17	32.07	0.0019	0.9299	0.9598	1.0000	DET_WDS_TEXS_1	Chromite | Area 3 | WDS_1_NiKa_D1 1
☐	Total	100.00	100.00								

Graph Statistics

Options

Spectrum 1



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NON- NORMALISÉE

WDS Quant Smart Quant Results with Standards (quanti chromite-Element Normalized : No)

File Quant Method View Tools

Drag a column header here to group by that column

using	Element	Weight %	Atomic % norm	Error %	Net Int.	K Ratio	Z	A	F	Label	SpecLabel
☐	O K	33.76	54.65	0.06	21886.21	0.1761	1.0767	0.4838	1.0016		Chromite I specimen 8.17 | Area 1 | Full Area 1
☐	Mg K	11.14	11.86	0.08	9719.18	0.0435	1.0326	0.3771	1.0025		Chromite I specimen 8.17 | Area 1 | Full Area 1
☐	Al K	13.41	12.88	0.07	13377.98	0.0594	1.0021	0.4419	1.0006		Chromite I specimen 8.17 | Area 1 | Full Area 1
☑	Si K	0.05	0.04	16.21	9.34	0.0002	1.0312	0.4911	1.0011	DET_WDS_TEXS_5	Chromite | Area 3 | WDS_1_SiKa_D5 1
☑	Ti K	0.08	0.04	7.88	45.61	0.0008	0.9171	0.9832	1.0931	DET_WDS_TEXS_1	Chromite | Area 1 | WDS_1_TiKa_D1 1
☑	V K	0.09	0.04	8.39	49.26	0.0008	0.8978	0.9936	1.0144	DET_WDS_TEXS_1	Chromite | Area 1 | WDS_1_VKa_D1 1
☐	Cr K	31.65	15.77	0.02	22743.46	0.2951	0.9140	1.0002	1.0197		Chromite I specimen 8.17 | Area 1 | Full Area 1
☑	Mn K	0.36	0.17	3.34	108.92	0.0032	0.8973	1.0045	1.0002	DET_WDS_TEXS_1	Chromite | Area 2 | WDS_1_MnKa_D1 1
☐	Fe K	9.68	4.49	0.02	4890.12	0.0843	0.9140	0.9526	1.0003		Chromite I specimen 8.17 | Area 1 | Full Area 1
☑	Ni K	0.14	0.06	12.17	32.07	0.0012	0.9278	0.9614	1.0000	DET_WDS_TEXS_1	Chromite | Area 3 | WDS_1_NiKa_D1 1
☐	Total	100.35	100.00								

Graph Statistics

Options

Spectrum 1

Wt % Element		
Al	=	12.65
Cr	=	31.23
Fe	=	9.89
Ti	=	0.072
Zn	=	0.008? NLD
Mg	=	10.41
Mn	=	0.10
Ni	=	0.134
V	=	0.116
Si	=	0.014
O	=	35.54

Wt % Element

Al	=	12.65
Cr	=	31.23
Fe	=	9.89
Ti	=	0.072
Zn	=	0.008? NLD
Mg	=	10.41
Mn	=	0.10
Ni	=	0.134
V	=	0.116
Si	=	0.014
O	=	35.54

Normalisée

using	Element	Weight %	Atomic %	Error %	Net Int.	K Ratio
☐	O K	32.21	53.11	0.06	22156.97	0.2674
☐	Mg K	11.24	12.19	0.08	9719.41	0.0652
☐	Al K	13.47	13.18	0.07	13377.15	0.0891
☑	Si K	0.05	0.05	16.21	9.34	0.0004
☑	Ti K	0.08	0.04	7.88	45.61	0.0011
☑	V K	0.09	0.05	8.39	49.26	0.0012
☐	Cr K	32.36	16.42	0.02	22743.75	0.4425
☑	Mn K	0.37	0.18	3.34	108.92	0.0049
☐	Fe K	9.99	4.72	0.02	4890.04	0.1264
☑	Ni K	0.15	0.07	12.17	32.07	0.0019
☐	Total	100.00	100.00			

Non-Normalisée

	Element	Weight %	Atomic % norm	Error %	Net Int.	K Ratio
	O K	33.76	54.65	0.06	21886.21	0.1761
	Mg K	11.14	11.86	0.08	9719.18	0.0435
☐	Al K	13.41	12.88	0.07	13377.98	0.0594
☑	Si K	0.05	0.04	16.21	9.34	0.0002
☑	Ti K	0.08	0.04	7.88	45.61	0.0008
☑	V K	0.09	0.04	8.39	49.26	0.0008
☐	Cr K	31.65	15.77	0.02	22743.46	0.2951
☑	Mn K	0.36	0.17	3.34	108.92	0.0032
☐	Fe K	9.68	4.49	0.02	4890.12	0.0843
☑	Ni K	0.14	0.06	12.17	32.07	0.0012
☐	Total	100.35	100.00			

eZAF Smart Quant Results

File Quant Method View Tools

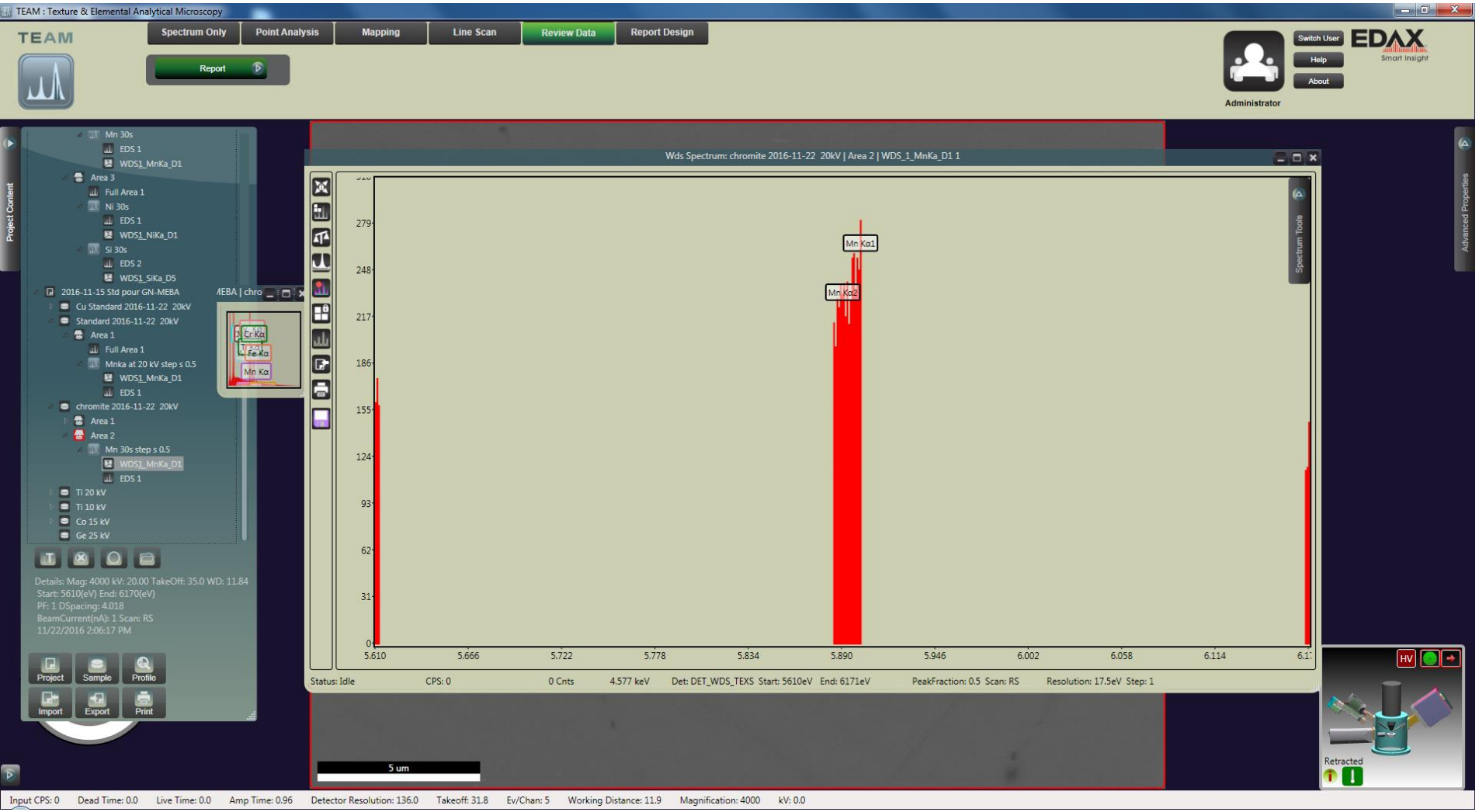
Drag a column header here to group by that column

Element	Weight %	Atomic %	Net Int.	F
O K	31.3	52.0	22135.2	1.0000
Mg K	11.4	12.5	9719.4	1.0047
Al K	13.7	13.5	13378.6	1.0045
Si K	0.3	0.2	276.2	1.0073
Ti K	0.1	0.0	61.3	1.2168
V K	0.1	0.0	44.1	1.1113
Cr K	31.9	16.3	22743.7	1.0497
Mn K	1.3	0.6	754.3	1.0396
Fe K	9.8	4.7	4890.0	1.0383
Ni K	0.2	0.1	64.8	1.0579

Wt % Element

Al = 12.65
Cr = 31.23
Fe = 9.89
Ti = 0.072
Zn = 0.008? NLD
Mg = 10.41
Mn = 0.10
Ni = 0.134
V = 0.116
Si = 0.014
O = 35.54

Mn avec 0.5 de la largeur a mi hauteur theorique



WDS Quant Smart Quant Results with Standards (quanti chromite-Element Normalized : Yes)

File Quant Method View Tools

Mn avec 0.5 de la largeur à mi hauteur theorique

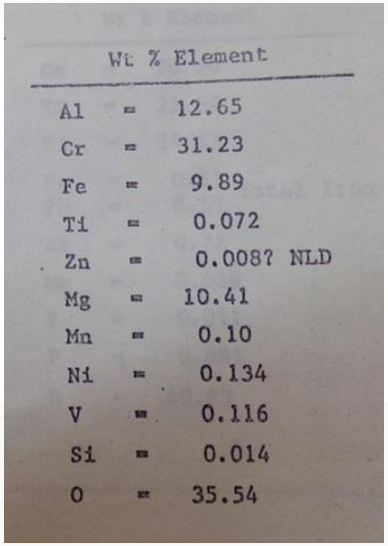
Drag a column header here to group by that column

using	Element	Weight %	Atomic %	Error %	Net Int.	K Ratio	Z	A	F	Label	SpecLabel
☐	O K	32.54	53.50	0.06	21368.44	0.2712	1.0784	0.4794	1.0017		chromite 2016-11-22 20kV | Area 2 | EDS 1
☐	Mg K	11.17	12.08	0.07	9187.14	0.0648	1.0342	0.3745	1.0025		chromite 2016-11-22 20kV | Area 2 | EDS 1
☐	Al K	13.40	13.07	0.07	12665.97	0.0887	1.0037	0.4390	1.0006		chromite 2016-11-22 20kV | Area 2 | EDS 1
☑	Si K	0.05	0.05	16.21	9.34	0.0004	1.0328	0.4885	1.0011	DET_WDS_TEXS_5	Chromite | Area 3 | WDS_1_SiKa_D5 1
☑	Ti K	0.08	0.04	7.88	45.61	0.0011	0.9186	0.9826	1.0945	DET_WDS_TEXS_1	Chromite | Area 1 | WDS_1_TiKa_D1 1
☑	V K	0.09	0.05	8.39	49.26	0.0012	0.8993	0.9931	1.0141	DET_WDS_TEXS_1	Chromite | Area 1 | WDS_1_V Ka_D1 1
☐	Cr K	32.37	16.37	0.02	21636.97	0.4424	0.9156	0.9998	1.0200		chromite 2016-11-22 20kV | Area 2 | EDS 1
☑	Mn K	0.19	0.09	5.26	51.07	0.0025	0.8988	1.0042	1.0002	DET_WDS_TEXS_1	chromite 2016-11-22 20kV | Area 2 | WDS_1_MnKa_D 1 1
☐	Fe K	9.96	4.69	0.02	4637.89	0.1260	0.9156	0.9509	1.0003		chromite 2016-11-22 20kV | Area 2 | EDS 1
☑	Ni K	0.15	0.07	12.17	32.07	0.0019	0.9295	0.9601	1.0000	DET_WDS_TEXS_1	Chromite | Area 3 | WDS_1_NiKa_D1 1
☐	Total	100.00	100.00								

Graph Statistics

Options

Spectrum 1



NON-NORMALISÉE: total 99.99%

Remerciements

EDAX – Harry Verhulst et Tomas Vikstrom
EDEN – Dominique Condamin et Stéphane Brassac
CP2M
GN- MEBA

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andrea.campos@univ.amu.fr

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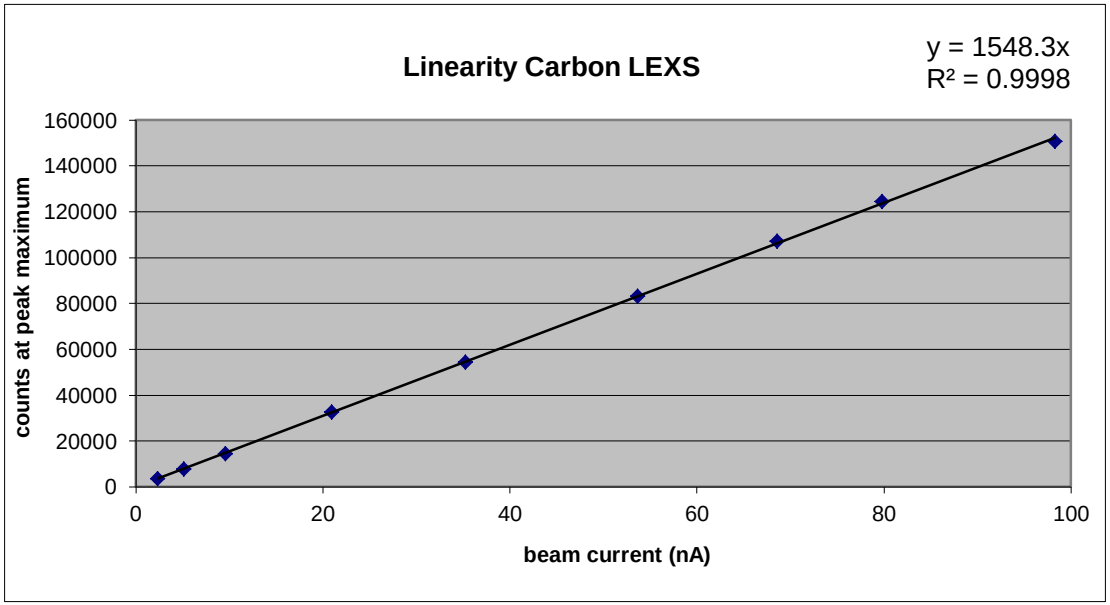
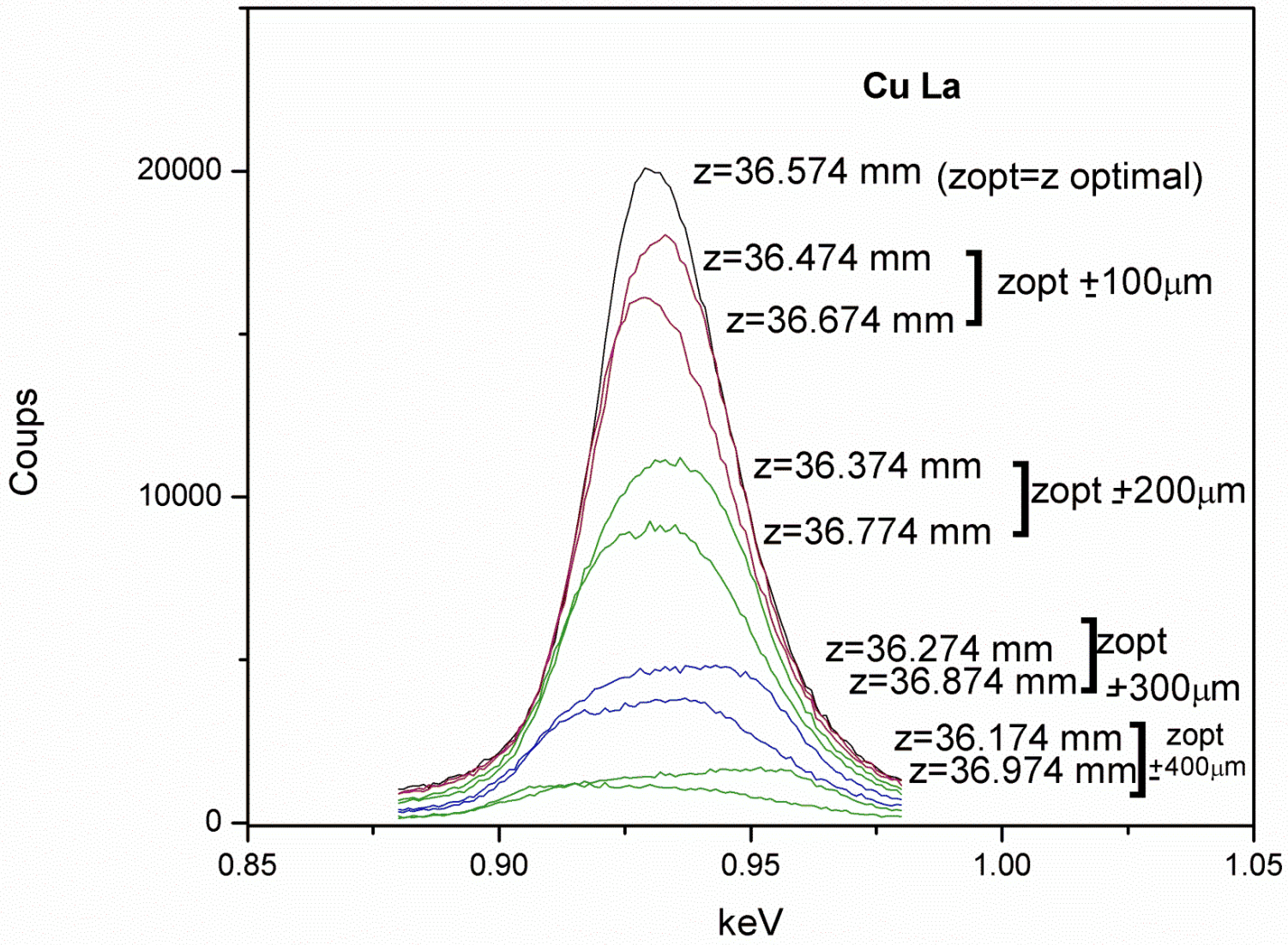


Optimization de Z - ESSENTIEL!!!

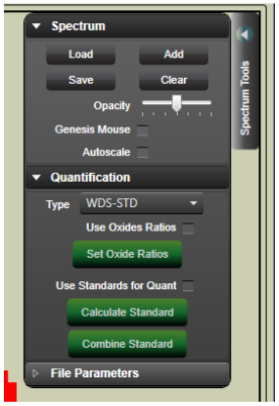


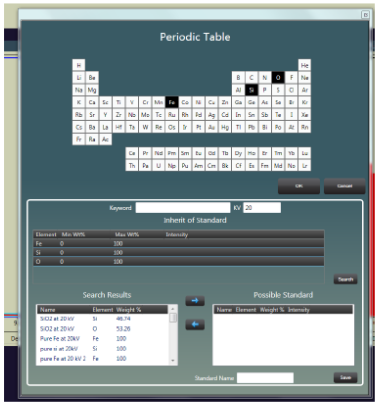
WDS quantification using TEAM SW.pdf - Adobe Reader

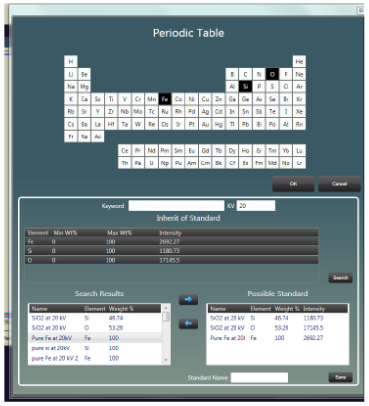
File Edit View Window Help

Open 12 / 22 115% Tools Fill & Sign Comment

Combined standard - 1







At Spectrum Tools and then Quantification section, press 'Combine Standard'. See middle picture: periodic table appears where elements can be selected, concentration ranges, kV and then a search should be done. See third screen, where Fe, Si and O at 20 kV were found and with the arrows moved into the combined file. After being done, give file a name and save. Do the same thing for EDS. In this way, pure intensity tables for different kV values can be created.

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18:57

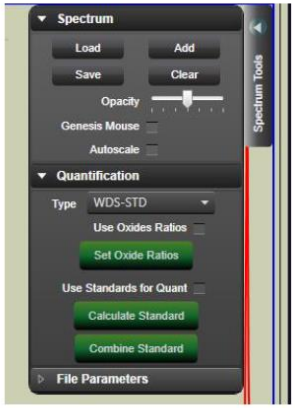
Source: EDAX- Harry Verhulst

WDS quantification using TEAM SW.pdf - Adobe Reader

File Edit View Window Help

Open 13 / 22 115% Tools Fill & Sign Comment

Combined standard - 2



At Spectrum Tools and then Quantification section, select 'Use Standards for Quant' and select from list the one desired. See picture at right side: at the bottom is shown which elements are in the WDS standards file and what the kV is. Normalization can be switched off here (or, as shown later on slide 17, when doing the quantification).

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13

Source: EDAX- Harry Verhulst