#### High-Speed Particle Analysis using Feature in the ESPRIT Software



Bruker Nano Analytics, Berlin, Germany Webinar, September, 2020



Innovation with Integrity

#### Presenters





#### Max Patzschke

Application Scientist EDS Bruker Nano Analytics, Berlin, Germany



Andrew Menzies, PhD

Sr. Applications Scientist Geology and Mining, Bruker Nano Analytics, Berlin, Germany

#### High-Speed Particle Analysis using Feature in the ESPRIT Software



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Innovation with Integrity

# Feature in the ESPRIT Software: Particle Analysis Workflow



Particle Sample (BSE Image)

**Image Binarization** 

Automatic detection of particles and image analysis  $\rightarrow$  particle morphology (area, length, width, aspect ratio, diameter, ...)

Accepted	Ares	Length	width	Average Diameter	PosX	Des V	÷.
			42	96	244		
.82	3546	100	51	85	-465	-594	
45	3290	95.	52	75	-209	-585	
- 114	4535	115	62	91	545	-582	
R5.			82	143			
P6	2462	68	:57	64	-498	+749	
17	2445	80	47	63		-541	
. 145	8468	164	76	132	205	-553	
-010	2642		57	65			
P10		105	62	06	-741	-514	
911	1118	-48	1427	46	274	-485	
952	6757	123	.76	103	434	-518	
			81	92		-495	
F14	4878		59	93		-492	
P15	2097	75	41	00	745	-665	
	5417	1.0.0	24	44		1244	

Automated collection of EDS spectra (each particle)  $\rightarrow$  quantification of EDS spectra

Classification of particles based on pre-defined chemical groups

Review, data analysis and reporting







#### Example Reference Standard: KC1a



Energy Mines and Energie Mines at Resources Canada Ressources Canada

CANADA CENTRE FOR MINERAL AND ENERGY TECHNOLOGY

#### REFERENCE ZINC-LEAD-TIN-SILVER ORE KC-Ia



Recommended Values *	95%	Confidenc		Interval	
Zn		34.65	*	0.15%	
Ръ		2.24	*	0.03\$	
Cu		0.629	*	0.015%	
Sn		0.61	*	0.02%	
Ag		0.167	*	0.002%	
and which the second			_		

#### WARNING

Bottles of KC-la have been sealed under nitrogen in laminated foil pouches to provide longterm protection against exidation during storage at CANMET. The recommended values for the certified elements pertain to the date of issue and the Canadian Certified Reference Materials Project cannot be responsible for changes occuring after receipt by the user. It is strongly recommended that opened bottles be stored under an inert gas in a desiccator or in a new heat-sealed foil pouch. Also the contents of the bottle should be exposed to air for the shortest time possible when taking subsamples. Unless these precautions are followed, the recommended values

#### DESCRIPTION

KC-la is intended to replace reference ore KC-l the supply of which is rapidly being depleted. The raw material for KC-la was handpicked at Kidd Creek Mines Ltd. Timmins, Ontario, and is from a zone of massive sphalerite-pyrite containing native silver and galena. It was dryground to minus 74 µm, blended and bottled in 200 g units. The stock was sampled systematically and analyzed form zinc and silver by chemical procedures to demonstrate homogeneity sufficient for use as a compositional reference material.

fineral	Mass 1
bhalerite	51.7
artz	21.4
rite	17.1
lena	2.6
alcopyrite	1.8
issiterite	0.8
lver	0.16
irbon	0.02
	Catradian Certified Reference Insternals PROJECT

- This is an ore sample from CCRMP.
- Commonly used for whole rock XRF analysis.
- Sample is crushed to -74 um.
- Known elemental concentrations and mineralogy.
- Sample from Kid Creek Mines Ltd. (Timmins, Ontario, Canada) from a zone of massive sphalerite-pyrite containing native silver and galena.

Mineralogical composition						
Mineral	Mass %					
Sphalerite	51.7					
Quartz	21.4					
Pyrite	17.1					
Galena	2.6					
Chalcopyrite	1.8					
Cassiterite	0.8					
Silver	0.16					
Carbon	0.02					



#### Measurement Conditions

#### Classification Criteria

EDIT CHEMICAL CLA	SSIFICATION			T					?
Measurement tim	e			Chemical particl	e properties			10 Clas	5985.C
1st Tier - Normal	I	2nd Tier - Exte	ended	Add class	Delete	class I<	<	0 >	>1
O Automatic	Precise +	O Automatic	Exhaustive 🝷		Ag Booring		255		
O Real time [s]	60	O Real time [s]	30	Class name:	Ag-bearing		455	<u> </u>	
Live time [s]	0,2	O Live time [s]	30	Class description:	Silver				
O Counts	30000	Ounts	100000	Treat unclassifie	d narticles as h	uits Dual	licato	Eraco	all
Region start [keV	/] 0,	00			a particles as in	Dupi	nate	LIDE	all
Region end [keV	] 15,0	00		Active Primary Op	er. Secondary	Туре	Min.	Max.	~
Scan full narticle	G	uard hand size [um]	0	Ag		Mass % (norm.)	20,0	100,0	+
Discard if no hits	G	and band size [pin]	<u> </u>						Add
Unclassified if ma	asc% <	30 %							~
La officialisation inte				Color palette		-			
Max particles per fie	≥ld	200		Ag-Bearing	• (	Chalcopyrite (CuF		Quartz (SiO	2)
Max particles overal		100000		<ul> <li>Sphalerite (Z</li> </ul>	nS) 🛛 🚺 🤇	Cu Sulphide	٠	Silicate	
Max hits overall		0		<ul> <li>Pyrite (FeS2)</li> </ul>	• (	Cassiterite (SnO2)			
				<ul> <li>Galena (PbS)</li> </ul>	) • F	Fe-Oxide			
Automatic analysis	s								
EDS MinClassQuant - I	KC1Av1 👻								
						-		100	
Classifier: MinCl	lass - KC1Av2	Ready.					Load		Save
Description: Classi	ification Minerals	KC1A					Annly		ancel
							white		arrout



#### Measurement Conditions

#### Set up Quantification Method

- Important for ability to classify minerals or phases correctly
- Affects overall analytical time

reasonement un	ne			Che	mical particle	propertie	es			d Classes
1st Tier - Norma	al	2nd Tier - Ext	ended		Add class	Dele	te class		0	
Automatic	Precise -	O Automatic	Exhaustive 🔸							
Real time [s]	60	O Real time [5]	30	Cla	ass name:	Ag-Bearin		ass		
Live time [s]	0,2	O Live time [s]	30	Class	description:	Silver				
Counts	30000	Ounts	100000		a at unclassified	clos a	chite Dual			Cases all
Region start [ke	V] 0,	00			eat unclassified	acies a	Dupi	Icate		Erase all
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					Ag		Mass % (norm.)	20,0	10	0,0
Scan full particle	e Gi	uard band size [µm	]0							A
Discard if no hits	s									
Unclassified if m	nass% <	30 %		Color	palette		•			
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Unclassified if m ax particles per fi ax particles over	nass% < ield all	30 %		Color	palette Ag-Bearing Sphalerite (Zn	s) •	Chalcopyrite (CuF Cu Sulphide		Quar	tz (SiO2)
Unclassified if m ax particles per fi ax particles overa ax hits overall	nass% < ield all	30 % 200 10 0		Color	palette Ag-Bearing Sphalerite (Zn Pyrite (FeS2)	s) •	Chalcopyrite (CuF Cu Sulphide Cassiterite (SnO2)		Quar Silicat	tz (SiO2) ie
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Unclassified if m lax particles per fi lax particles over lax hits overall utomatic analys	nass% < ield all	30 % 200 10400 0		Color	palette Ag-Bearing Sphalerite (Zn Pyrite (FeS2) Galena (PbS)	s)	Chalcopyrite (CuF Cu Sulphide Cassiterite (SnO2) Fe-Oxide		Quar	tz (SiO2) ie
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Elements       ^         H       Use spectrum elements       He         Li Be       Use list elements       B       C       N       O       F       Ne         Na Mg       Search additional elements       B       C       N       O       F       Ne         K Ca Sc Ti V Cr Mn Fe Co Ni Cu Zn Ga Ge As Se Br Kr       Rb Sr Y Zr Nb Mo Tc Ru Rh Pd Ag Cd In Sn Sb Te I Xe       Xe       Cs Ba La Hf Ta W Re Os Ir Pt Au Hg Tl Pb Bi Po At Rn       Fr Ra Ac       Ce Pr Nd Pm Sm Eu Gd Tb Dy Ho Er Tm Yb Lu       Th Pa U Np Pu Am Cm Bk Cf Es Fm Md No Lr         Clear all       Minimum concentration 2.00 %       %         Element overview list       •         Standards       •         Quantification model       P/B - ZAF •         Additional settings       \$eries fit •	Set	ting	şs																
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Fr       Ra       Ac       Ce       Pr       Nd       Pm       Sm       Eu       Gd       Tb       Dy       Ho       Er       Tm       Yb       Lu         Th       Pa       U       Np       Pu       Am       Cm       Bk       Cf       Es       Fm       Md       No       Lr         Clear all       Minimum concentration       2.00       %       %       Standards       •       Standards       •       Standards       •       %         Background settings       SEM, Automatic       •       •        %	Cs	Ba	La	Hf	Та	W	Re	Os	Ir	Pt	Au	Hg	тΙ	Pb	Ві	Po	At	Rn	
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Standards <ul> <li>Background settings</li> <li>Deconvolution settings</li> <li>Series fit</li> <li>Quantification model</li> <li>P/B - ZAF</li> <li>Additional settings</li> <li> <ul></ul></li></ul>	_		Cle	ar a		_	)									2.0	00	%	
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#### Description

 Automatic element ID - 0.5 wt.% minimum -C deconvolution

 Load
 Save

 To project
 Apply









#### Feature: Data Processing Results



The following information is available for review and extraction:

- Field (Panorama)
- Particle Image,
- Particle Data,
- Particle Classification,
- Particle Spectrum, and
- Quantification Results

# Review functionality

- Results can be sorted according to classes
- Search for and drive to specific particle or field
- Check for Important Particles (Hit Classes)
- Re-classification and/or requantification without reacquisition

#### Feature: Data Processing Results



#### Field and Particle Image

# Particle Spectrum and Quantification Results



#### Particle Data, Classification and Quantification Results

# Feature: Data Processing Data analysis: histograms and charts



- Available diagrams for data analysis and reporting:
  - $\rightarrow$  histogram
  - $\rightarrow$  binary charts
  - $\rightarrow$  ternary charts
- Any particle property (morphological parameter) or element (wt%, atom%, ...) can be plotted
- Link between data point in diagrams, particle list and spectrum to find specific particles of interest



	Chem. class	Count	Area A	verage diameter P	erimeter O	rientation M	ax circle radius A	rea in pixels \	/olume of sphere E	quivalent diameter
	Ag-Bearing	13	773	33.6	114	68	11.6	484	57760	30.0
•	Sphalerite (ZnS)	4695	688	31.5	104	67	10.5	431	52311	27.5
	Pyrite (FeS2)	1095	701	31.6	104	64	10.4	439	54665	27.5
	Galena (PbS)	102	688	31.8	105	65	10.5	431	51942	27.8
•	Chalcopyrite (CuFeS2)	10	562	30.4	96	61	9.9	352	33911	26.1
•	Cu Sulphide	14	643	29.0	95	50	9.9	403	51108	25.4
	Cassiterite (SnO2)	16	622	29.4	93	73	10.1	390	45437	25.8
	Fe-Oxide	40	786	33.2	112	67	11.0	492	66256	29.1
	Quartz (SiO2)	3565	637	31.4	101	70	9.9	399	45251	26.9
	Silicate	55	593	30.8	99	62	9.7	371	39939	26.2
•	Unclassified	4423	645	30.1	98	61	9.5	404	49731	25.7
	Min		168	15.8	48	0	7.1	105	5132	14.6
	Max		5111	97.0	388	179	33.9	3200	863429	80.7
	Average		699	32.5	105	82	10.6	438	52575	28.1
	Std. dev.		560	12.0	43	53	3.5	351	71739	9.9

#### Feature: Data Processing Build Panorama



Individual Fields can be combined into single Panorama

Maintain overall particle locations with sample





#### Feature: Data Processing Grain Compositions





# Example: Reference Standard: KC1a Mineralogy





# Example: Reference Standard: KC1a Mineralogy





#### Example: Reference Standard: KC1a Grain Sizes



#### Sample crushed to -74 um



# Example: Reference Standard: KC1a Ag-Bearing Phases



Important Classes:

- Ag-Bearing: Ag-Sulphides
- Other Phases with Ag likely mixed spectra due to inclusions



# Feature in the ESPRIT Software: Particle Analysis Workflow



Particle Sample (BSE Image)

**Image Binarization** 

Automatic detection of particles and image analysis  $\rightarrow$  particle morphology (area, length, width, aspect ratio, diameter, ...)

Automated collection of EDS spectra (each particle)

Accepted	Ares	Length	width	Average Diameter	PosX	Des V	4
				96			
.82	3546	156	51	85	-465	-504	
#5	3290	95	52	75	-209	-555	
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F14	4878		59	93		-492	
	2097	75	41	00	745	-665	
	5417.	1.0.0	24	44		-2.24	

Classification of particles based on pre-defined chemical groups

 $\rightarrow$  quantification of EDS spectra

Review, data analysis and reporting



# Feature in the ESPRIT Software: Summary and Conclusion



- Complete and integrated particle analysis with chemical classification
- Morphology: detection of one or multiple phases; loading and analyzing of any image
- Chemical classification: set up hundreds of classes, modifiable quantification method
- Analysis-Method-Development with offline re-classification and re-quantification of results
- Data analysis and reporting with charts and histograms
- Link between particle list, feature image, histogram and spectrum
- Data export (particle list) as text or excel file
- Map data (HyperMap) can also be loaded and used for particle analysis
- Automation: stage movement, analysis of large areas, drive back to frame or particles





# **Are There Any Questions?**

Please type in the questions you might have in the Q&A box and press *Send*. More Information



### For more information, please contact us:

info.bna@bruker.com



#### Innovation with Integrity

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