X-ray Analysis Methods in Geology

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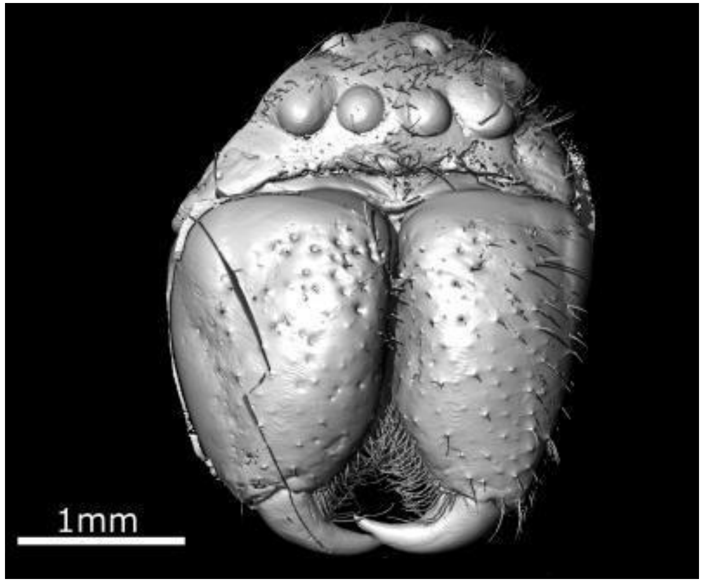
X-ray Analytical techniques:

Diffraction

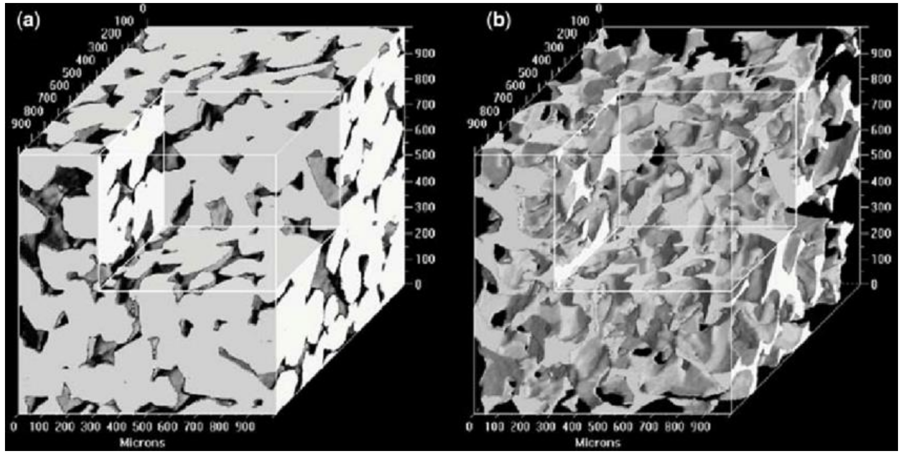
Fluorescence

Not:

Medical tomography



Computed x-ray tomography



Computed micro-tomography

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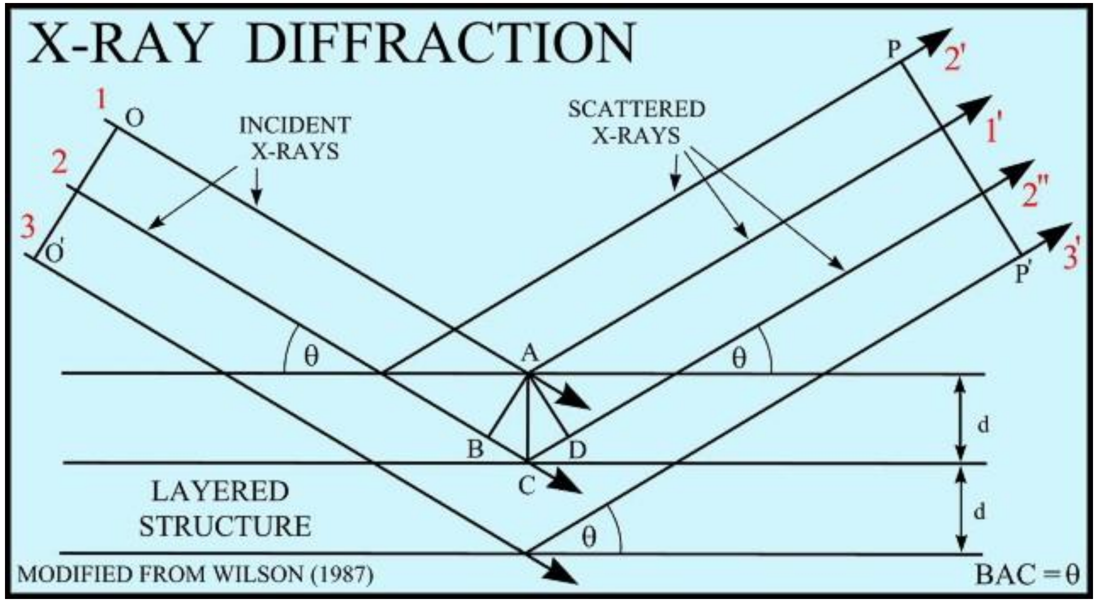

Bragg Equation:

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

N.B. fluorescence is still possible at the same time

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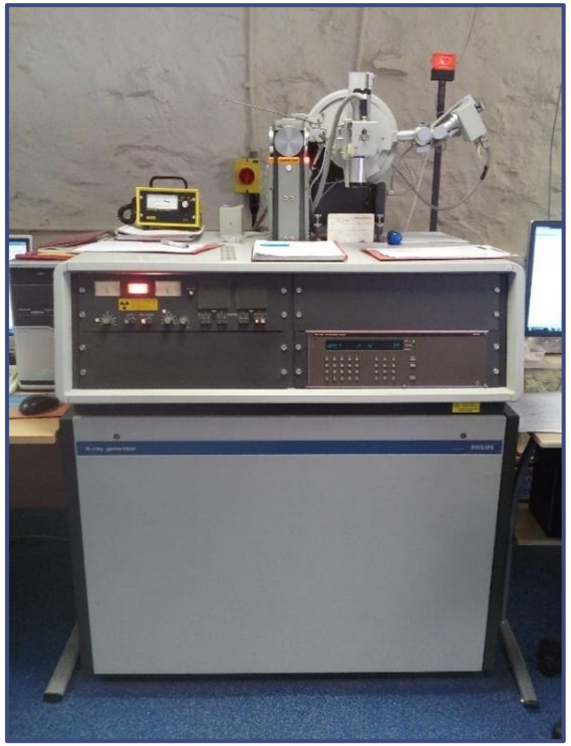
XRD Instrumentation

XRD-1
Philips PW1730



XRD-2
Philips PW1730

XRD-3
Siemens D5000

XRD-4
PANalytical X'Pert3
State of the art instrument

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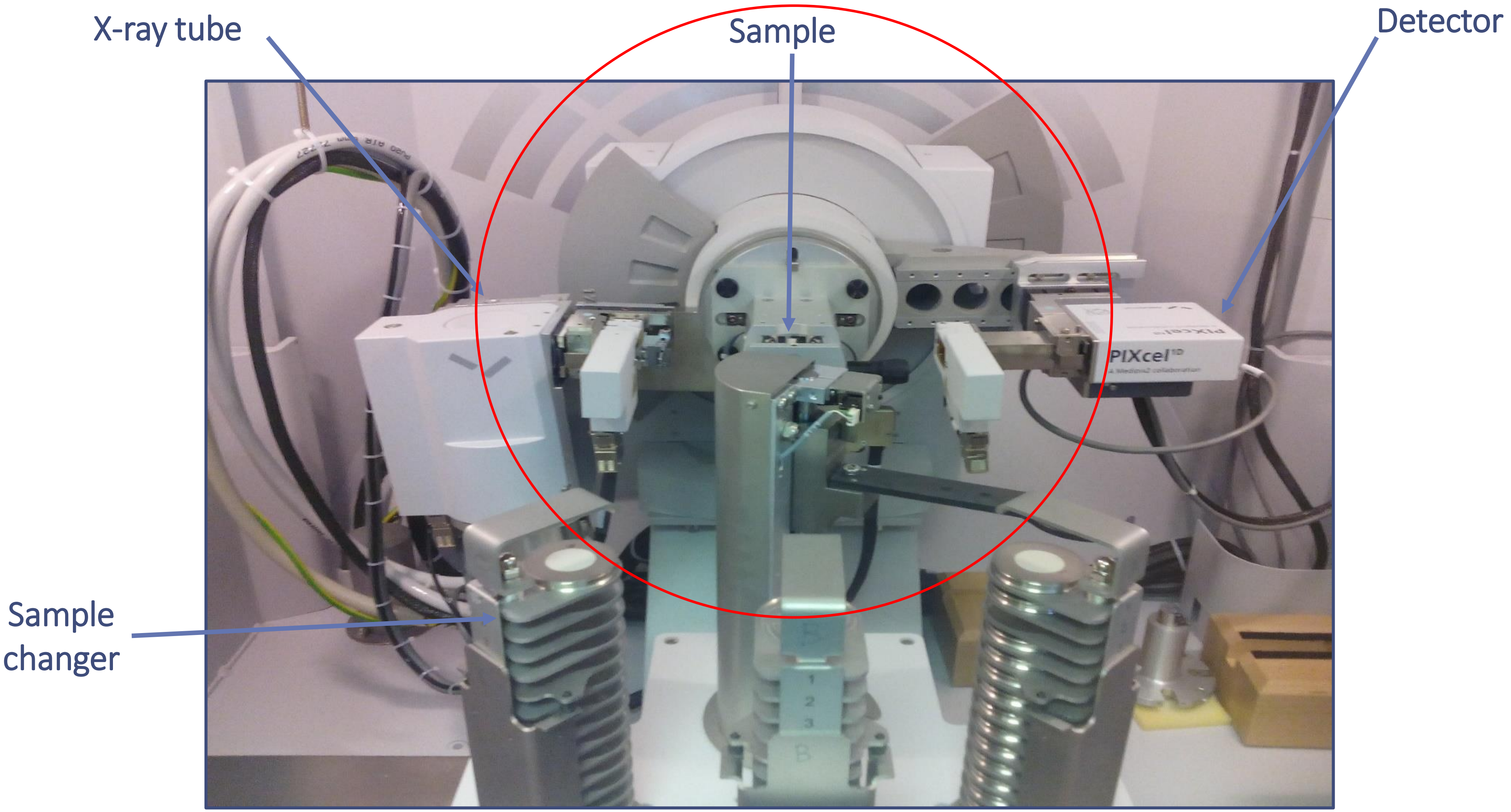
X-RAY MINERAL SERVICES INSTRUMENTATION



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XRD - WHOLE ROCK SAMPLE PREPARATION

1

2g of sample subsampled and pre-crushed

2

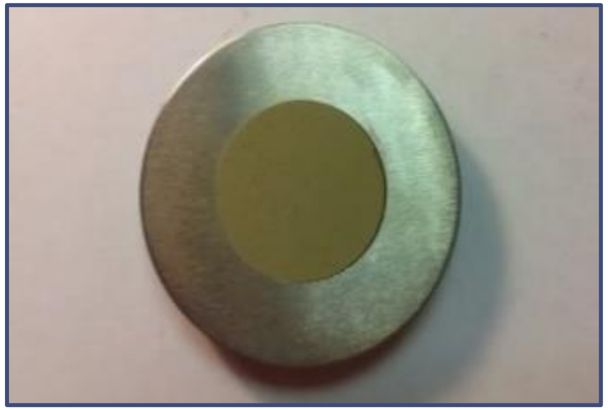
Micronised in water or ethanol

3

Spray dried or evaporated

4

Sample mounted in sample holder and scanned



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XRD - WHOLE ROCK MINERAL IDENTIFICATION

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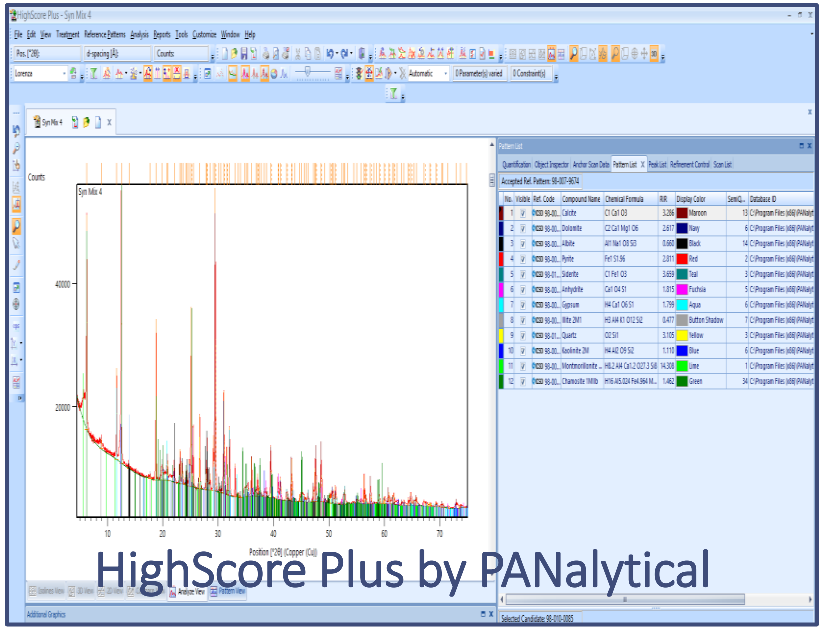
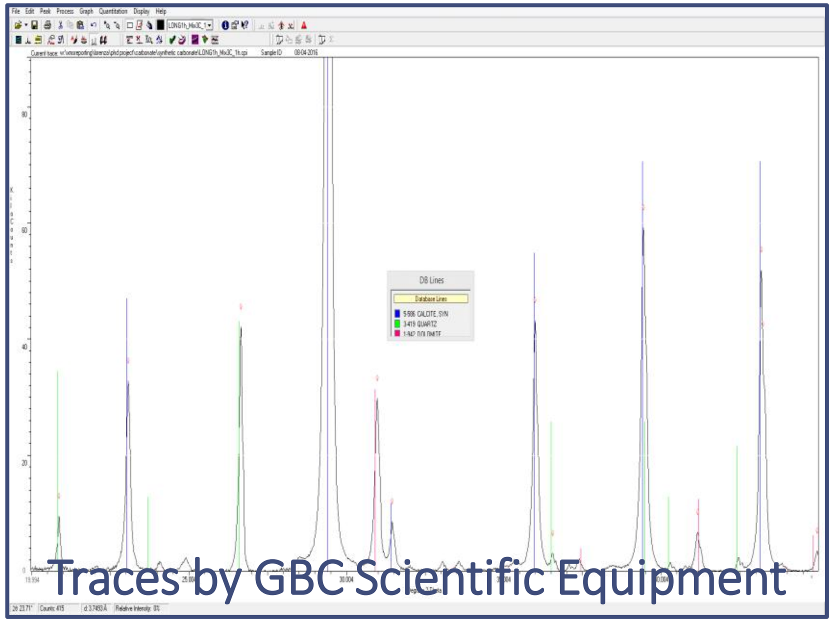
Powder Diffraction Files (PDF-4) ICDD Minerals Database (46,000 mineral structures) and Inorganic Crystal Structure Database (ICSD) with more than 210,000 crystal structures



HighScore Plus by PANalytical



Traces by GBC Scientific Equipment

TWO MINERAL QUANTIFICATION METHODS:

THE XMS Reference Intensity Ratio method (XMS RIR)

Rietveld method

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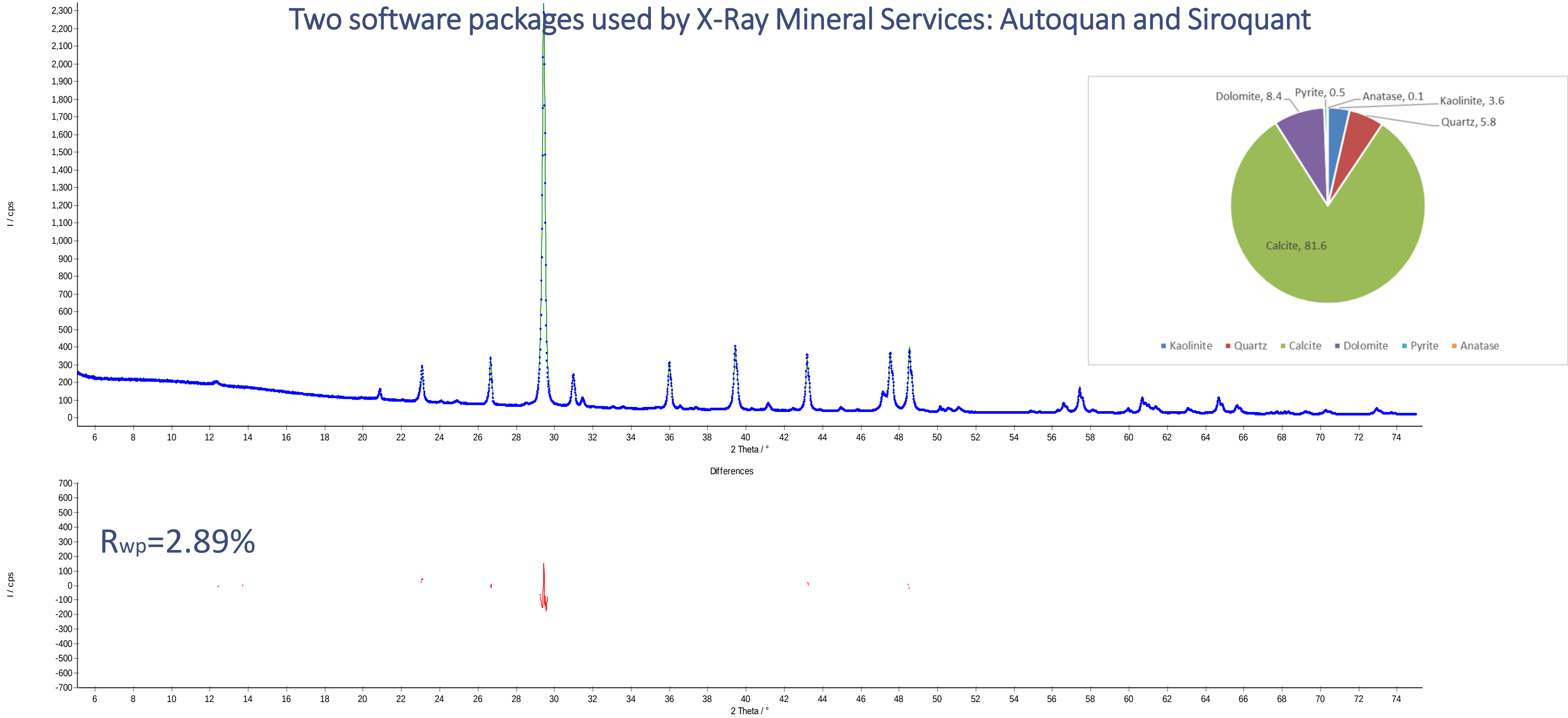
Rietveld method

Two software packages used by X-Ray Mineral Services: Autoquan and Siroquant



Observed diffractogram

Calculated diffractogram

Difference plot between observed and calculated diffractogram

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CLAY FRACTION SAMPLE PREPARATION

X-RAY MINERAL SERVICES

1

5g of sample subsampled

2

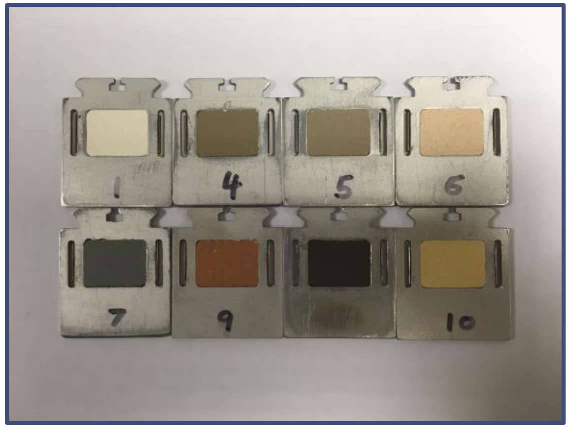
Suspended in water and ultrasound treated. Centrifuge <2 micron fraction

3

Clay suspension deposited on a glass micro-fibre filter

4

Sample is mounted in sample holder and scanned



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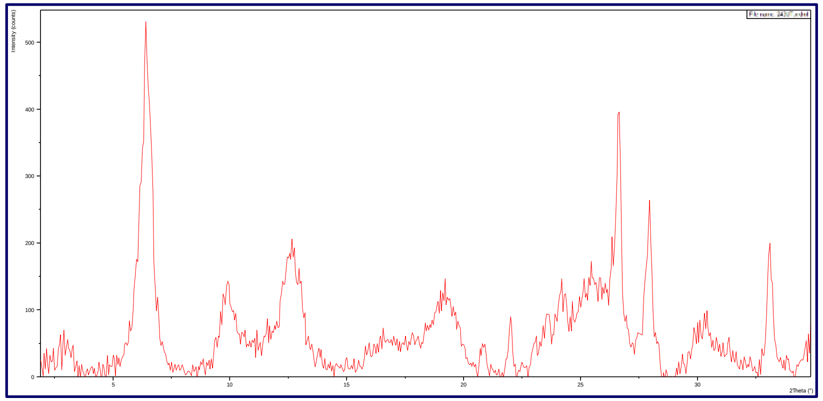
CLAY FRACTION ANALYSIS

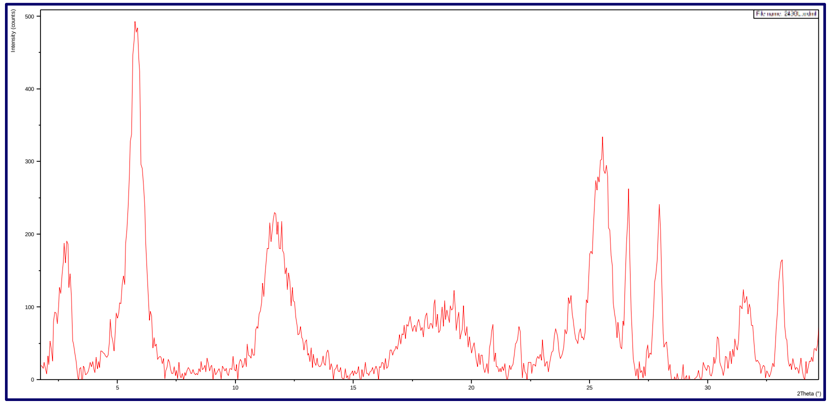
1

Sample is scanned untreated



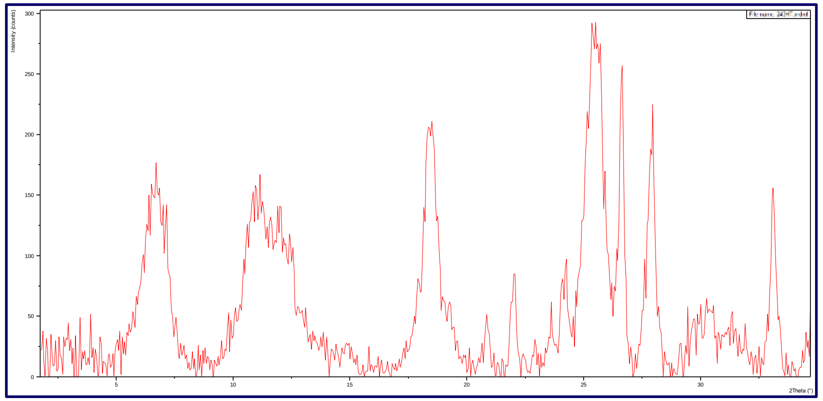
2

Sample is scanned after ethylene glycol solvation



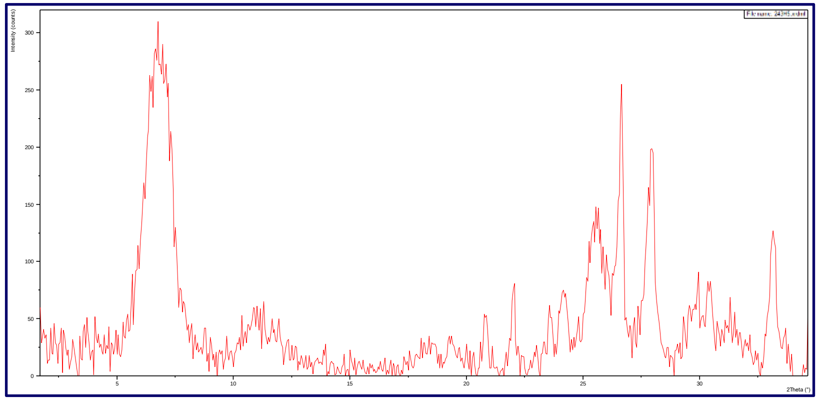
3

Sample is scanned after heat treatment to 380°C



4

Sample is scanned after heat treatment to 550°C



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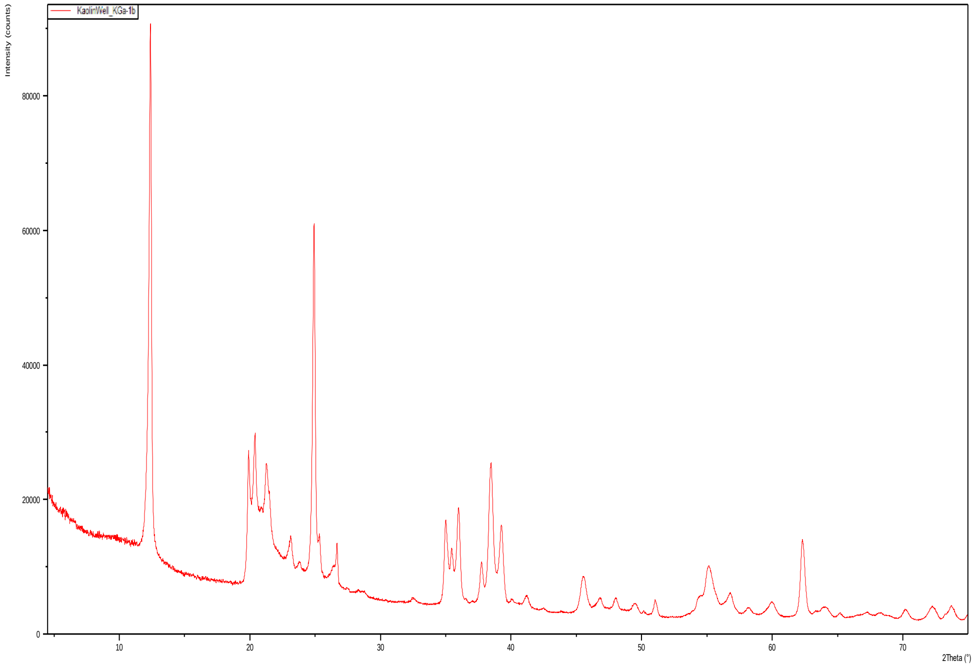
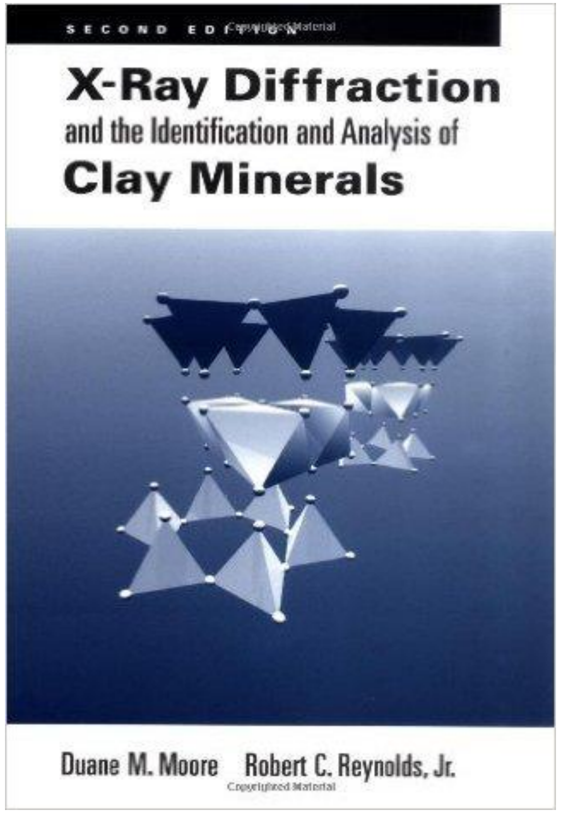
Comparison with Clay Minerals Society standards



Moore and Reynolds guidelines



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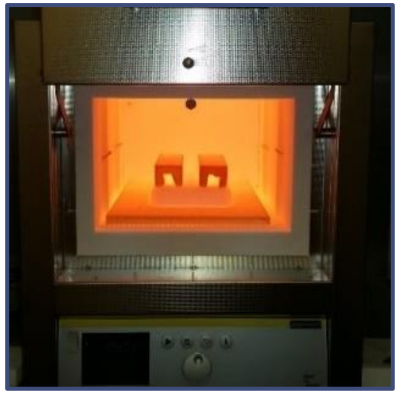
Major Elements

1

5g of dry sample subsampled and hand milled

2

LOI determined

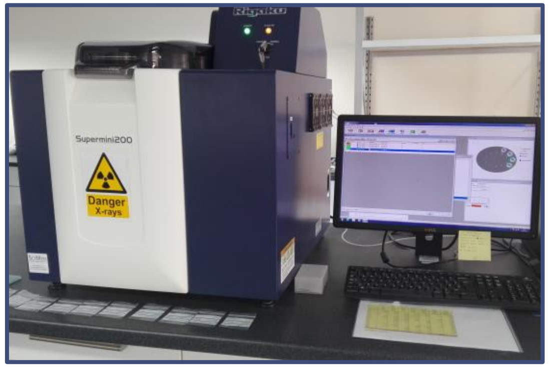


3

Beads fused

4

Beads analysed using a Rigaku Supermini200 Wavelength Dispersive Spectrometer



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Trace Elements

1
15g of dry sample subsampled and pre-crushed

2
Ball milled

3
Pellet pressed

4
Pellets analysed using a Rigaku
Nex-DE Energy Dispersive Spectrometer

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XRF Reports

X-RAY MINERAL SERVICES

Major elements

Sample	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	Mn_2O_3	Fe_2O_3	SrO	BaO	LOI
	%	%	%	%	%	%	%	%	%	%	%	%	%	%
1	0.73	2.03	11.64	45.48	0.06	0.88	2.17	15.61	0.52	0.14	3.83	0.05	0.04	16.67
2	<0.02	0.73	0.85	2.84	0.11	0.41	0.08	52.21	0.03	0.16	0.38	0.06	0.23	42.00
3	<0.02	0.73	0.72	6.34	0.11	0.21	0.04	50.78	0.03	0.18	0.36	0.08	0.03	40.69
4	<0.02	0.55	0.47	3.06	0.06	0.17	<0.01	52.33	0.02	0.18	0.29	0.07	0.03	43.05
5	<0.02	0.43	0.28	1.70	0.05	0.09	<0.01	54.25	<0.01	0.19	0.21	0.07	0.01	42.99
6	<0.02	0.46	1.01	7.12	0.13	0.57	0.04	49.93	0.03	0.22	0.53	0.11	0.02	39.63
7	0.25	1.91	6.48	17.76	0.32	1.91	0.91	36.77	0.17	0.17	1.93	0.08	0.04	31.17
8	0.06	0.88	2.45	8.75	0.18	1.13	0.12	46.94	0.06	0.14	0.96	0.10	<0.01	38.31
9	0.05	0.52	1.81	6.62	0.15	0.34	0.16	49.96	0.07	0.18	0.47	0.10	<0.01	39.84
10	<0.02	0.41	1.30	5.20	0.12	0.27	0.06	51.19	0.04	0.18	0.38	0.09	0.02	40.85
11	<0.02	0.34	1.06	6.70	0.11	0.27	0.02	50.33	0.04	0.22	0.31	0.10	0.01	40.61
12	<0.02	0.35	1.87	10.85	0.11	1.56	0.06	46.67	0.07	0.15	0.95	0.09	0.05	37.05
13	<0.02	0.37	0.66	4.84	0.11	0.30	0.03	51.69	0.02	0.22	0.36	0.12	<0.01	41.02
14	<0.02	0.25	0.49	18.11	0.08	0.26	<0.01	44.52	0.03	0.11	0.20	0.07	0.02	35.82
15	<0.02	0.23	0.30	9.79	0.06	0.14	<0.01	49.69	0.01	0.14	0.13	0.08	0.03	39.34
16	<0.02	0.24	0.34	11.44	0.07	0.24	<0.01	48.27	0.03	0.12	0.19	0.08	0.02	38.91
17	<0.02	0.24	0.40	8.50	0.08	0.62	0.02	49.40	0.03	0.12	0.16	0.09	0.92	39.40
18	<0.02	0.27	0.63	11.09	0.09	0.32	0.01	47.30	0.03	0.13	0.27	0.08	0.01	39.51
19	1.41	1.94	13.43	60.53	0.07	0.23	1.44	0.63	1.35	0.11	7.52	0.03	0.06	10.68
20	0.62	1.42	10.98	69.89	0.07	0.13	1.76	0.22	0.77	0.05	6.04	0.01	0.09	7.29
21	1.71	2.24	16.06	55.34	0.13	0.26	2.43	0.71	1.27	0.17	9.33	0.03	0.06	9.44
22	0.44	2.89	7.78	53.86	0.06	<0.01	1.07	1.25	0.33	1.78	17.84	<0.01	0.04	12.06
23	<0.02	0.25	0.57	11.62	0.09	0.24	<0.01	47.59	0.02	0.14	0.22	0.08	0.03	38.95
24	<0.02	0.37	0.53	3.62	0.09	0.15	0.01	52.68	0.02	0.22	0.28	0.11	0.02	42.04
25	0.03	0.32	0.69	11.86	0.09	0.34	0.04	48.08	0.02	0.14	0.28	0.08	0.02	38.00
26	<0.02	0.26	0.59	12.46	0.07	0.28	0.01	47.64	0.02	0.13	0.24	0.08	0.01	37.89
27	<0.02	0.20	0.32	4.89	0.06	0.15	<0.01	52.30	<0.01	0.12	0.15	0.08	<0.01	41.50
28	<0.02	0.24	0.63	10.23	0.08	0.25	0.03	49.01	0.02	0.12	0.22	0.09	0.02	38.89
29	<0.02	0.10	0.66	48.62	0.03	0.32	0.07	9.61	0.02	0.04	0.18	0.02	0.10	39.34
30	<0.02	0.24	0.29	10.67	0.04	0.13	<0.01	49.00	<0.01	0.12	0.16	0.07	<0.01	39.06
31	<0.02	0.26	0.38	8.30	0.06	0.11	<0.01	50.73	0.02	0.10	0.18	0.07	0.02	39.75
32	<0.02	0.37	0.74	15.69	0.10	0.29	0.01	45.72	0.02	0.10	0.28	0.08	<0.01	36.39
33	<0.02	0.33	0.38	13.53	0.06	0.08	0.02	47.27	0.02	0.08	0.19	0.08	0.01	37.84
34	<0.02	0.29	0.73	11.61	0.06	0.13	0.05	47.94	0.02	0.11	0.31	0.08	<0.01	38.29
35	<0.02	0.26	0.58	12.42	0.06	0.23	<0.01	47.05	0.02	0.10	0.22	0.08	0.03	38.42
36	<0.02	0.27	0.68	12.83	0.05	0.21	0.06	46.92	0.02	0.23	0.33	0.06	<0.01	37.74
37	<0.02	0.45	1.41	15.63	0.07	0.65	0.12	44.56	0.05	0.15	0.65	0.06	<0.01	35.70
38	0.06	0.39	1.51	29.52	0.07	0.21	0.20	36.64	0.07	0.19	0.48	0.06	0.03	30.34
39	0.30	1.00	5.31	58.79	0.14	0.43	1.41	15.92	0.27	0.15	1.31	0.03	<0.01	14.39
40	<0.02	0.73	0.85	2.84	0.11	0.41	0.08	52.21	0.03	0.16	0.38	0.06	0.23	42.00
41	0.11	2.14	2.42	8.87	0.29	1.66	0.33	39.16	0.16	5.86	3.14	0.08	0.03	35.13
42	1.04	3.85	14.00	57.96	0.07	0.29	2.46	0.31	0.78	0.17	11.22	0.01	0.03	7.07
43	1.06	2.69	12.45	56.71	0.08	0.38	2.47	4.81	0.91	0.53	7.46	0.03	0.03	9.76
44	0.95	2.44	13.38	64.37	0.07	0.19	2.53	0.29	0.90	0.08	6.17	0.02	0.01	8.01

Trace elements

Sample	V	Cr	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Rb	Sr	Y	Zr
	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
1	96	64	24	68	29	39	15	2	10	ND	71	476	14	83
2	ND	ND	12	ND	14	12	3	ND	ND	ND	ND	902	11	19
3	ND	ND	ND	ND	13	25	ND	ND	ND	ND	ND	600	9	21
4	ND	ND	ND	ND	ND	10	ND	ND	ND	ND	ND	572	8	19
5	ND	ND	ND	ND	14	10	4	ND	ND	ND	ND	757	16	26
6	ND	ND	ND	ND	17	29	3	ND	ND	ND	ND	766	12	26
7	ND	ND	ND	ND	11	9	ND	ND	ND	ND	ND	579	9	24
8	ND	ND	ND	ND	12	7	ND	ND	ND	ND	ND	678	10	20
9	ND	ND	ND	ND	13	13	3	ND	ND	ND	ND	667	11	23
10	ND	ND	ND	ND	11	8	ND	ND	ND	ND	ND	668	7	19
11	ND	ND	ND	ND	13	11	ND	ND	ND	ND	ND	631	10	22
12	ND	ND	ND	ND	12	8	3	ND	ND	ND	ND	597	6	20
13	ND	ND	11	ND	16	11	ND	ND	ND	ND	ND	655	10	25
14	ND	ND	ND	ND	11	7	ND	ND	ND	ND	ND	667	8	21
15	ND	ND	ND	ND	12	7	ND	ND	ND	ND	ND	662	8	23
16	ND	ND	ND	ND	14	13	4	ND	ND	ND	ND	510	12	29

ND = Not detected (concentration below effective limit of detection (LOD), see below)

LOD (ppm)	37	39	10	11	10	7	3	2	5	2	3	30	5	5

Sample	Nb	Mo	Sn	Sb	Cs	Ba	La	Ce	Nd	Hf	Pb	Th	U
	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
1	11	ND	ND	ND	4	212	25	48	23	4	21	8	ND
2	ND	ND	ND	ND	ND	134	14	15	ND	5	9	3	ND
3	ND	ND	ND	ND	ND	136	13	13	ND	5	8	3	ND
4	ND	ND	ND	ND	ND	128	10	13	ND	5	7	2	ND
5	ND	ND	ND	ND	ND	106	14	22	16	5	11	3	ND
6	ND	ND	ND	ND	ND	124	13	17	ND	5	9	3	ND
7	ND	ND	ND	ND	ND	73	8	12	ND	ND	8	2	ND
8	ND	ND	ND	ND	ND	134	12	13	ND	5	7	2	ND
9	ND	ND	ND	ND	ND	155	10	17	ND	6	8	3	ND
10	ND	ND	ND	ND	ND	59	8	14	8	4	7	2	ND
11	ND	ND	ND	ND	ND	104	11	15	ND	5	8	3	ND
12	ND	ND	ND	ND	ND	ND	10	12	ND	6	7	2	ND
13	ND	ND	ND	ND	ND	119	9	14	ND	5	9	3	ND
14	ND	ND	ND	ND	ND	ND	12	15	ND	4	8	3	ND
15	ND	ND	ND	ND	ND	ND	7	15	11	5	8	3	ND
16	ND	ND	ND	ND	ND	ND	15	31	23	4	8	3	ND

ND = Not detected (concentration below effective limit of detection (LOD), see below)

LOD (ppm)	5	20	2	2	3	56	6	9	2	4	5	2	2

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Used to check mineral quantification obtained from XRD

	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	MnO	TiO2	Fe_2O_3			
	%	%	%	%	%	%			%			
Illite/Smectite	0.11	3.2	19.76	51.1		1.6			0.8		Illite/Smectite	0
Illite+Mica	0.5	1.08	27.68	51.05	7.6	0.8			2.6		Illite+Mica	6.3
Kaolinite n1 D&Z		0.14	39.55	45.8	0.03	0.41			0.75		Kaolinite	0
Chlorite n3 D&Z		15.28	21.2	25.43		0.16	0.35	0.88	25.3		Chlorite	3
Quartz				100							Quartz	68.5
K Feldspar n4 D&Z	0.8		19.55	63.66	15.6	0.5			0.1		K Feldspar	16.3
Plagioclase n1 D&Z	11.1		19.6	67.8	0.3						Plagioclase	5.4
Calcite n1 D&Z		0.04				55.92					Calcite	0
Dolomite n5 D&Z		21.12				31.27			0.22		Dolomite	0
Hematite									100		Hematite	0.5
										TOT		
Calculated chemical analysis	0.8	0.5	6.6	86.5	3.0	0.1	0.0	0.0	1.4	99.1		
										0.0		
Actual chemical analysis	0.7	0.5	7.1	85.6	3.1	0.1	0.0	0.2	1.4	98.7	**LOI 1.1 %**	

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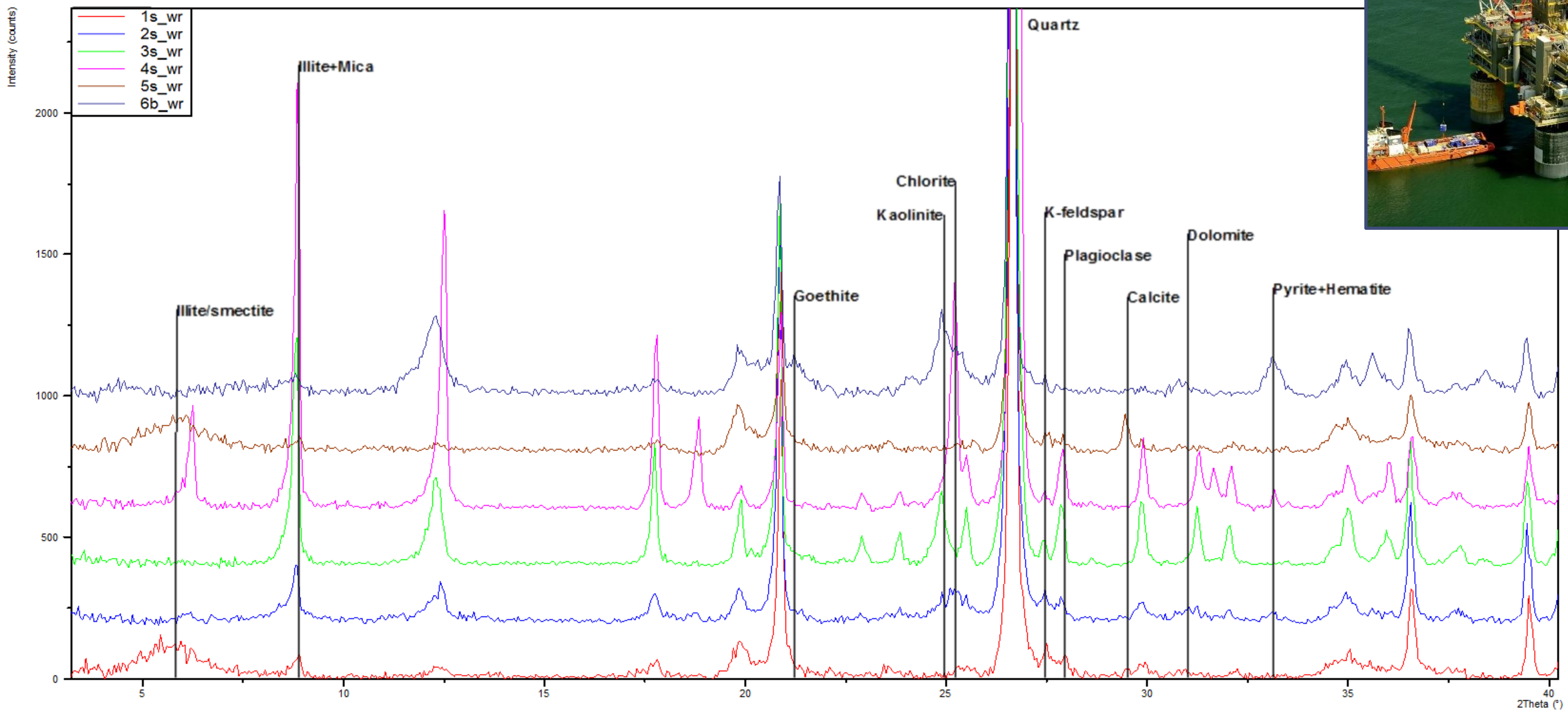
X-RAY MINERAL SERVICES

Analysis of cuttings from a petroleum reservoir



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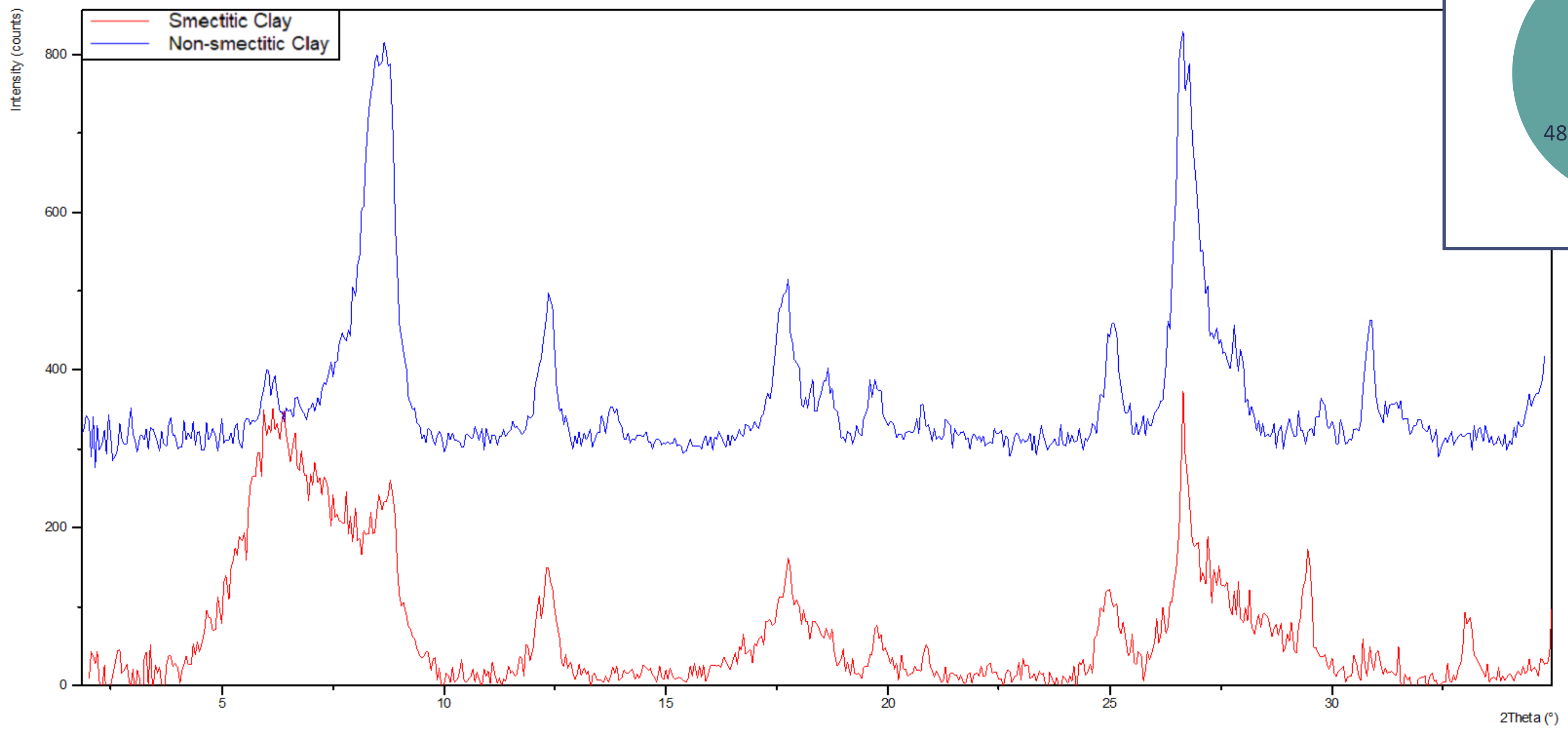
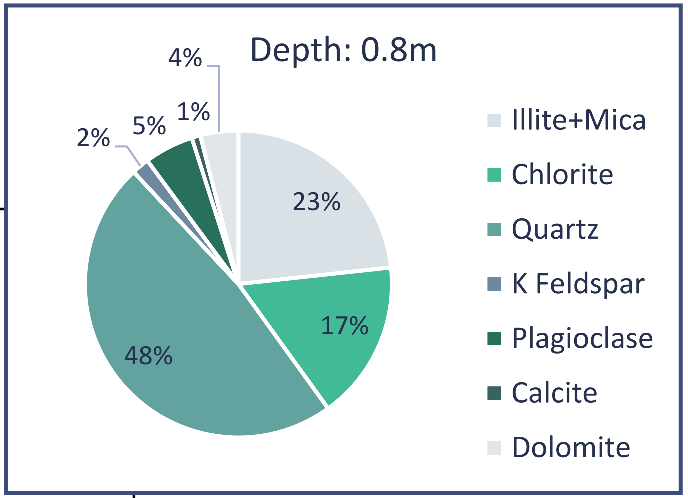
Construction

Foundation clay survey (HS2)





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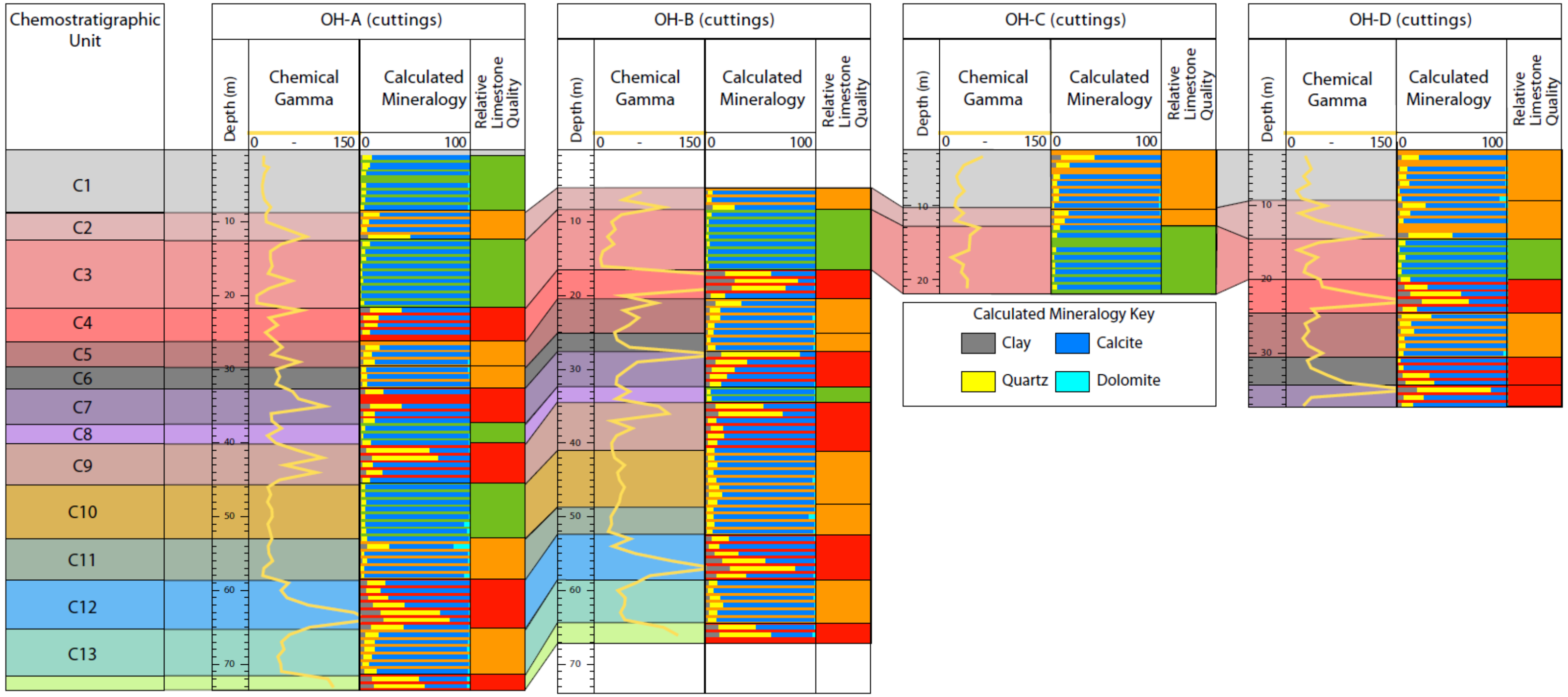
APPLICATIONS IN EXTRACTIVE INDUSTRIES



Geochemical data derived from cuttings have been used to chemostratigraphically correlate five wells that penetrate a limestone section.

In addiction, the data has been used to provide a gamma-ray response as well as evaluate the relative limestone purity

(green = best quality; red = worst quality)

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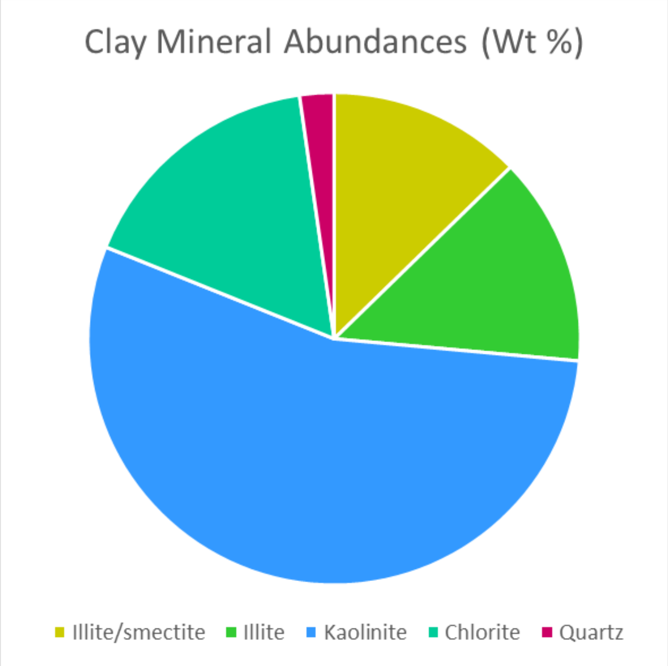
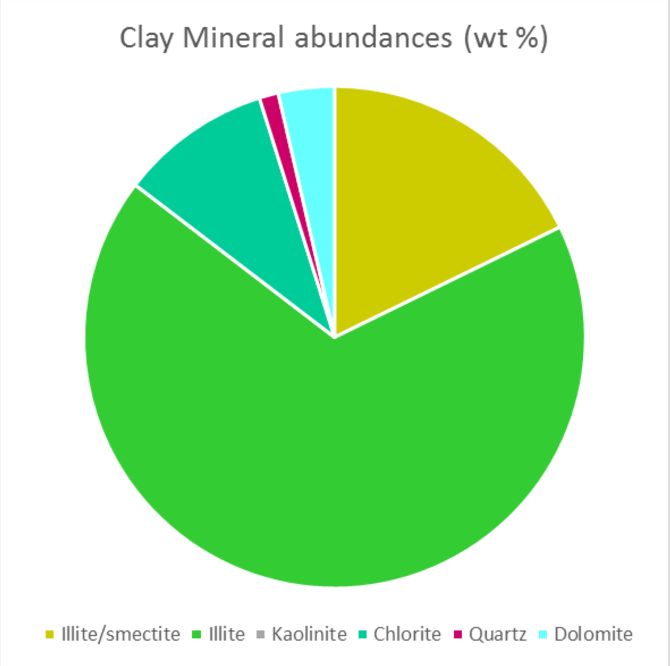
Quarry in the Mercia Mudstone Formation

Mercia Mudstones and Etruria ‘Marl’ are widespread,important sources of clay for brick and tile manufacture. Other sources are much more local e.g. Wealden Fm, Carboniferous seat-earths.

Customer comment:
“I was aware that increasing Ca content (or the ratio of Ca : Fe) gave a buff firing colour, but was not aware that patches of our clay contained so much Dolomite (<50%). Trouble is that they all look alike to the man on the digger in the quarry.”



Eturia Marl



Mercia Mudstone

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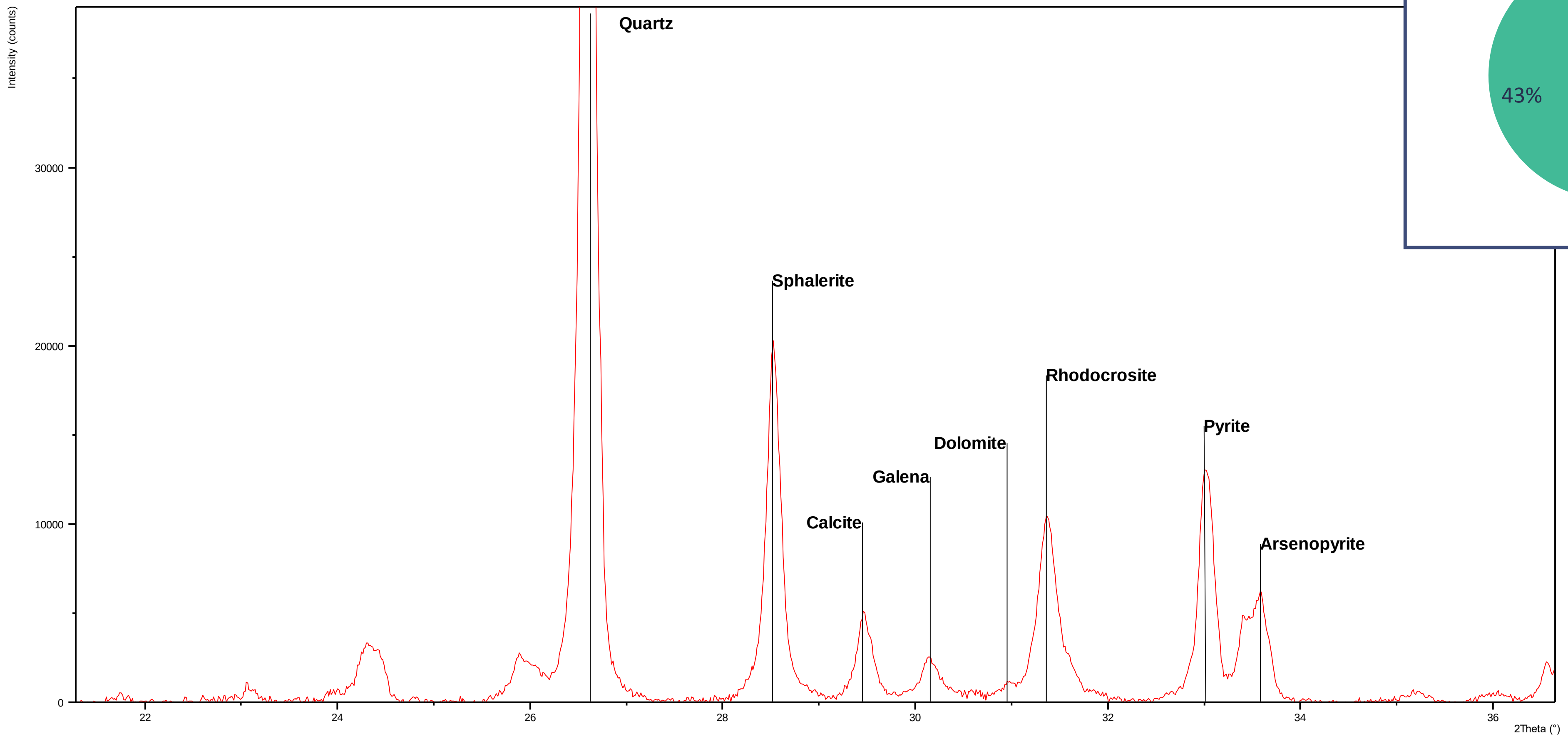
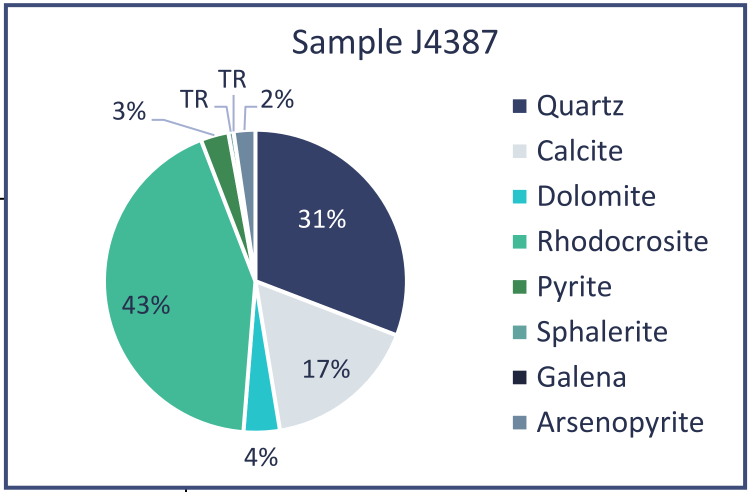
Mining

Ore – mineralogy and chemical composition





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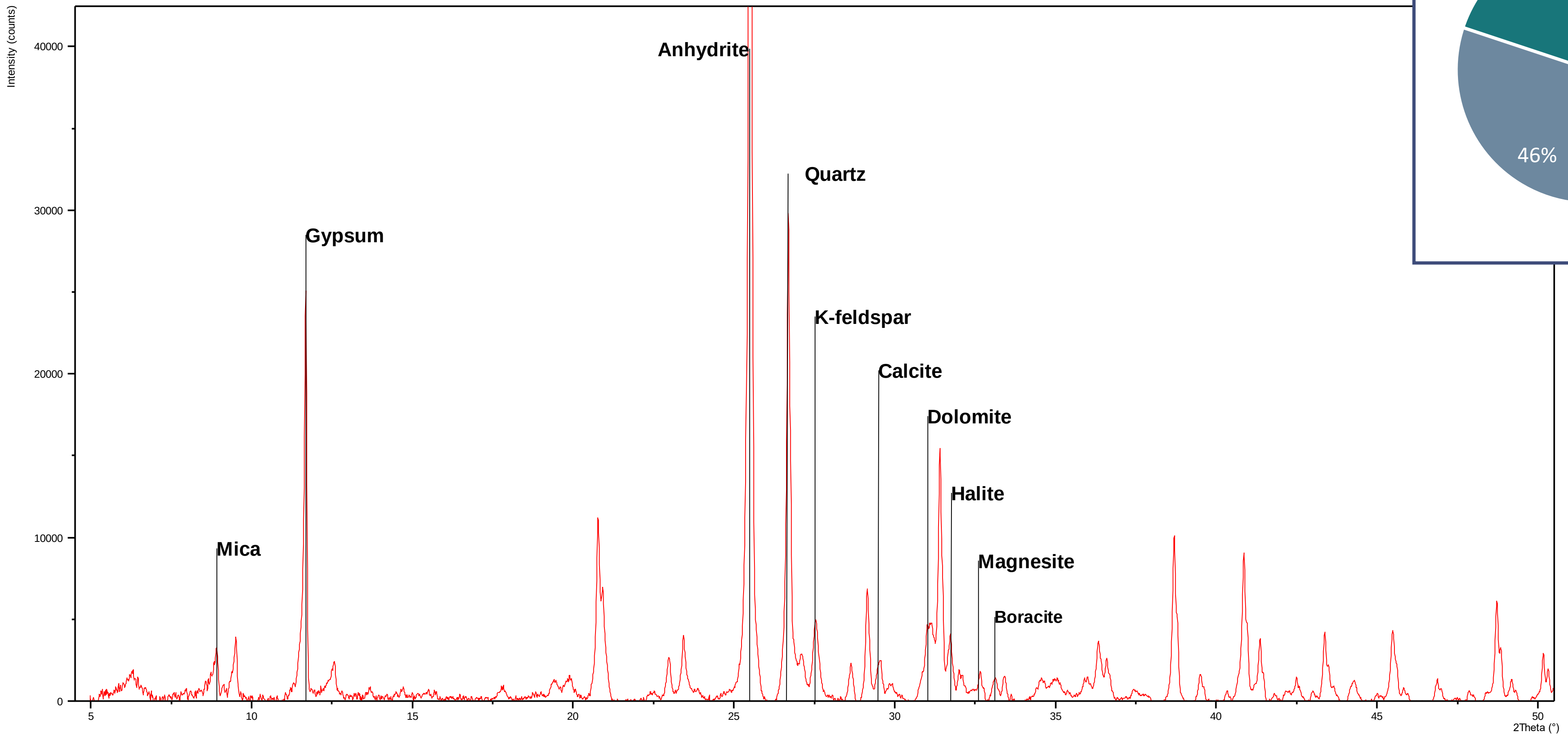
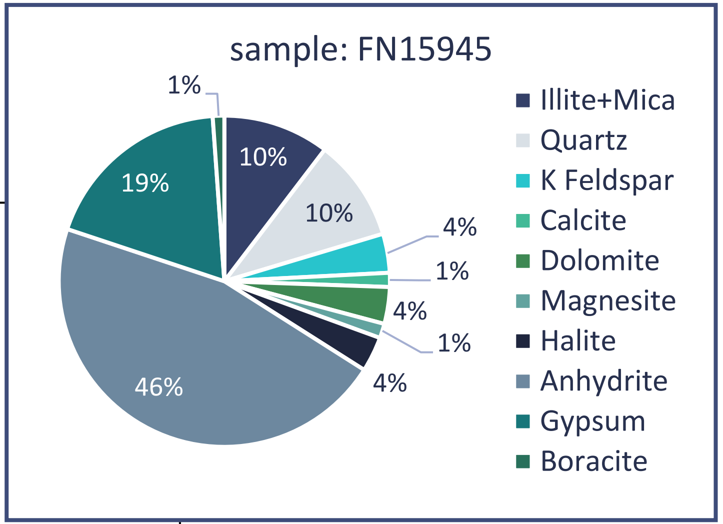
Environment

Mineralogical studies for disposal or reprocessing of mine tailings and arisings





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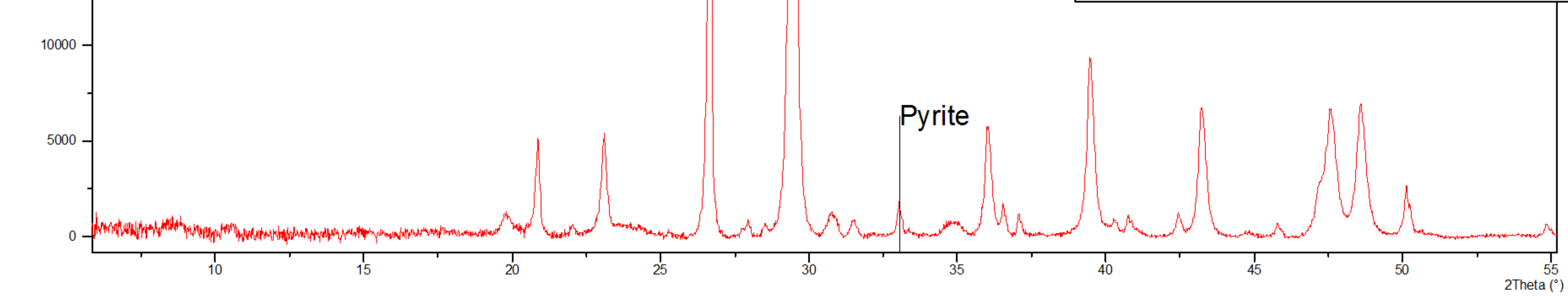
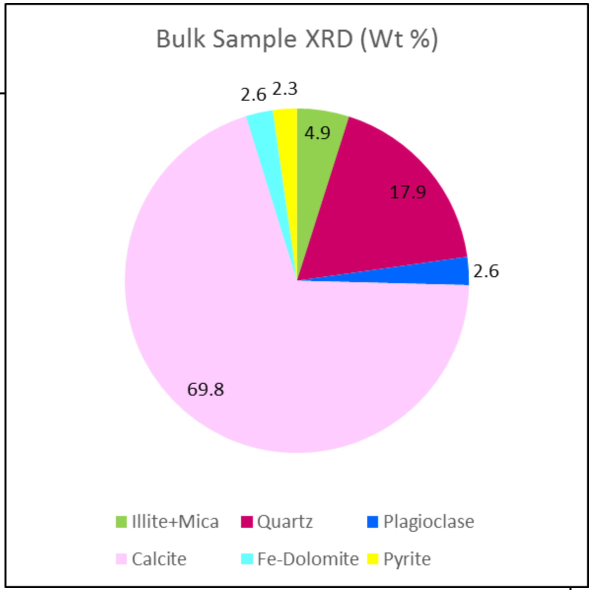
Architecture

Weathering of pyrite-bearing building materials





Pyritic limestone: e.g. ‘Calp’ dark, argillaceous, fine-grained Carboniferous limestones of the Dublin district .

Weathers to gypsum with expansion.

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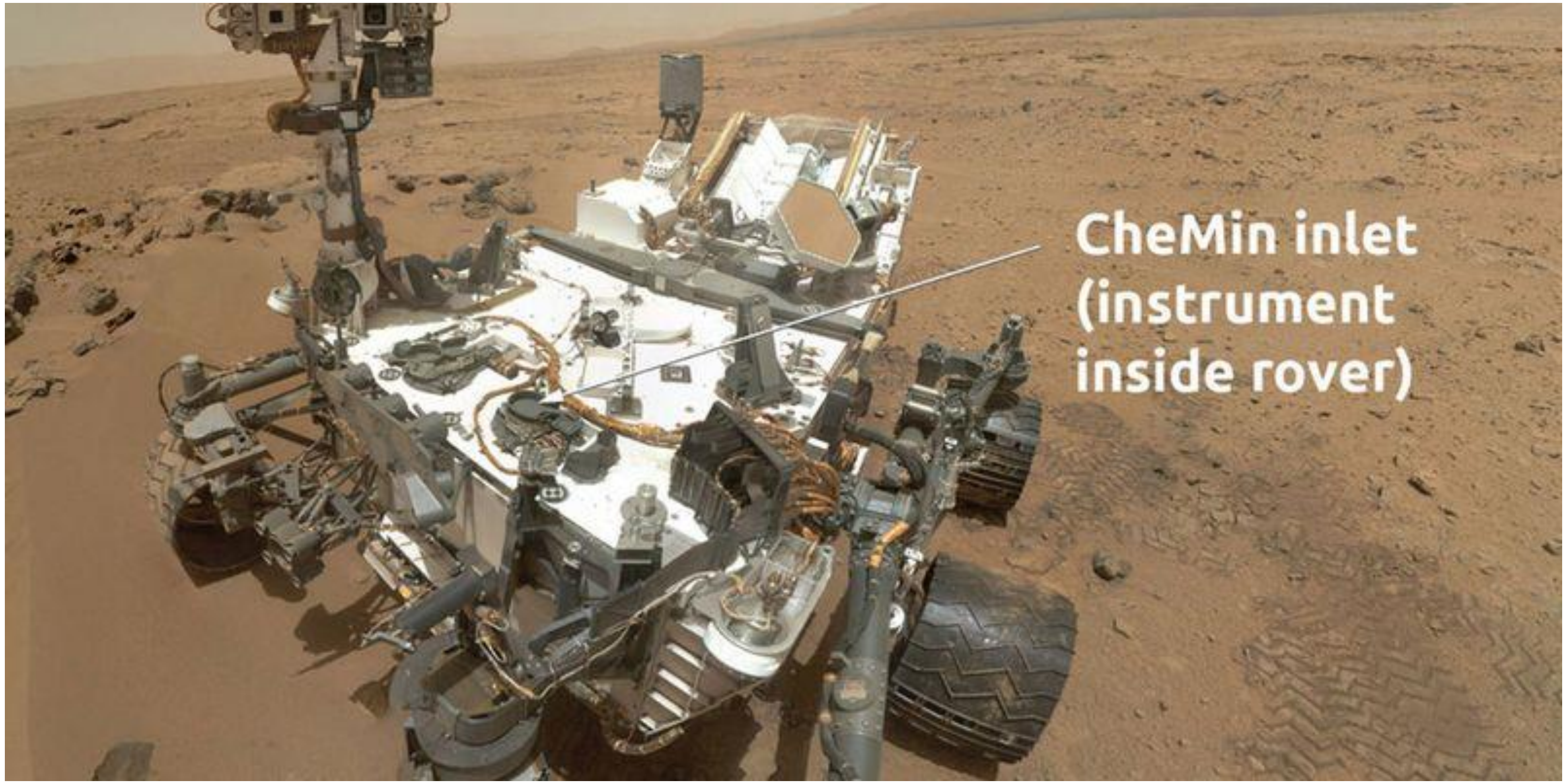
Curiosity Rover: NASA CheMin Instrument



Location:
Mars, Gale Crater since August, 2012 and celebrating 3000 sols in operation

Curiosity drills into rocks, collects the resulting fine powder, or uses a scoop for collecting soil, delivering it to a sample holder.

CheMin directs a narrow beam of X-rays through the powder sample; some are absorbed by atoms in the sample and re-emitted or fluoresced at characteristic energies, some are diffracted.

A charge-coupled device (CCD) collects both diffraction and fluorescence information. An analysis can take up to 10 hours to complete, but yields chemical and mineralogical analyses critical to the environmental characterisation mission.

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WHY CHOOSE X-RAY MINERAL SERVICES?

The only way to confirm the accuracy and precision of a technique applied to natural samples is to check against synthetic mixtures of appropriate mineral standards.
At X-ray Mineral Services, we participate in international Quantitative Phase Analysis (QPA) Round Robin events and routinely prepare our own standard mixtures to verify our sample preparation and QPA methods.

"If you are considering getting a mineral analysis of a clay-bearing sample from any type of laboratory, and where accuracy is important, simply ask the lab where they have placed in the Reynolds Cup"

Douglas K. McCarty
President of the Clay Minerals Society

2016 - 4th position on the shale sample

2018 - 8th position overall

2020 - 9th position overall

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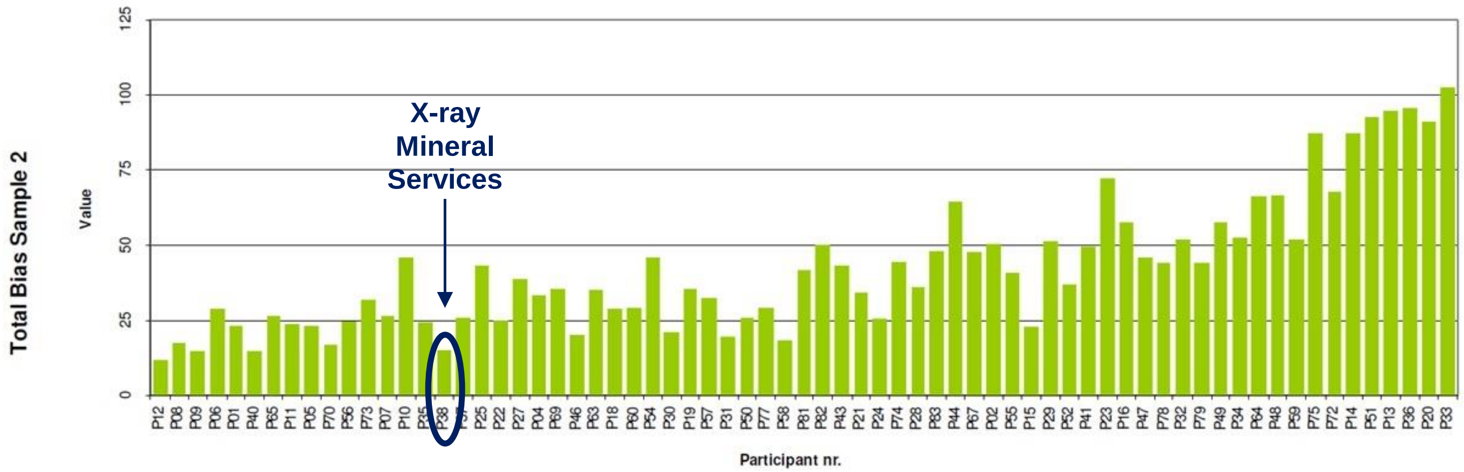
Reynolds Cup 2016 - Carbonate-bearing shale			
Mineral	RC 8-2 (m%)		
	Actual (m%)	Submitted (m%)	Δ
Quartz	15.7	14.2	1.5
K-Feldspar group	3.4	4.0	0.6
Plagioclase group	4.0	3.9	0.1
Calcite	11.4	12.0	0.6
Dolomite/ankerite	6.4	4.5	1.9
Pyrite	2.5	3.1	0.6
Rutile	0.1	0.0	0.1
Anatase	2.3	2.3	0.0
Amorphous group	5.0	3.9	1.1
Total Non-clay	50.8	47.9	6.5
Kaolinite	12.5	10.8	1.7
Mica (dioctahedral)	19.8	18.2	1.6
Smectite (dioctahedral)	11.4	10.5	0.9
Chlorite (trioctahedral)	5.5	9.6	4.1
Total clay/phyllosilicate	49.2	49.1	8.3
Total identified	100.0	97.0	14.8
Bias non-clay	6.5		
Bias clay	8.3		
Total bias	14.8		

12 minerals and amorphous material

The Total Bias chart illustrates the variability of results for a shale sample submitted by all participants in the Reynolds Cup 2016.



The total bias: cumulative absolute error in the quantification of the minerals, compared to the actual composition of the mixture – the lower the better!

In 2016, we achieved 4th position out of 83 participants on the carbonate-bearing shale sample

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Gorffen / Fin / End

Acknowledgements:
My colleague Lorenza whose presentation I have adopted and adapted for this purpose
Images that I have blagged are the property of their source authors
Thank you for the invitation

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