

MAR 20050010: FORT HILLS

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**VALUABLE MINERALS IN THE UTS FORT HILLS
OIL SANDS LEASE CORE SAMPLES**

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**Work performed for:
UTS Energy Corporation**

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EXECUTIVE SUMMARY

This report summarizes the heavy mineral composition of five Fort Hills oil sands core samples in comparison to the composition reported for other Athabasca oil sands leases.

Core sample 17, from the Middle McMurray Formation contains the most heavy mineral concentration: TiO_2 , in the form of rutile and anatase is 0.4 wt% and ZrO_2 in the form of zircon is 0.08 wt%. This amount of valuable minerals is within the range that has been reported for Middle McMurray oil sands ores. The bitumen-free mineral sand sample (core 41) above the Devonian shale and core 28 from the Lower McMurray contain the smallest quantities of valuable minerals (0.05–0.06 wt% TiO_2 and 0.007–0.008 wt% ZrO_2). These concentrations are at the low end of the average in oil sands. Core 41 also contains substantial amounts of pyrite, more than the Upper McMurray core samples.

While the concentration of heavy minerals in the Fort Hills lease core samples is significantly lower than in conventional mineral sands operations around the world, the bitumen extraction process concentrates the heavy minerals in the bitumen froth phase up to approximately 20 wt%, depending on the efficiency of the extraction process.

Oxidation of the samples due to long-term storage at room temperature precluded extraction of bitumen in a batch extraction unit to concentrate the heavy minerals. Severe bitumen oxidation results in a disproportionate increase in the quartz and clay minerals carried over to the froth phase. However, comparisons of the ore data to ore and froth mineral data from other leases suggests that the heavy mineral concentration in the Fort Hills ore samples would reach similar levels in the froth tailings.

The primary challenge to the production of heavy mineral concentrates from froth tailings is the cost of removing hydrocarbon contamination to enable the use of conventional gravity, magnetic, and electrostatic separation techniques commonly used in mineral sands operations.

CONTENTS

EXECUTIVE SUMMARY	III
INTRODUCTION	1
MATERIALS AND METHODS	2
RESULTS AND DISCUSSION	2
PARTICLE SIZE DISTRIBUTION	3
HEAVY MINERALS ANALYSIS	3
CLAY MINERALOGY	4
CONCLUSIONS	5
ACKNOWLEDGEMENTS	5
REFERENCES	5
APPENDIX A:	11
MICROSTRUCTURAL AND MINERALOGICAL DATA	11
Core 1: 1AA-02-30-96-10W4 INT. 39.04-42.78 m	12
Core 17: 1AA-02-30-96-10W4 INT. 79.09-85.51 m	13
Core 21: 1AA-02-30-96-10W4 INT. 91.50-94.42 m	15
Core 28: 1AA-02-30-96-10W4 INT. 110.01-113.16 m	16
Core 41: 1AA-02-30-96-10W4 INT. 151.13-152.20 m	17

TABLES

Table 1 – Particle size distribution of the bitumen-free UTS Fort Hills oil sands core samples.	7
Table 2 – Bulk chemical analysis of the core samples	7
Table 3 – Mineral composition of the core samples.	7
Table A1 – Mineralogy and oxide composition of the core I silt fraction. 12	
Table A2 – Mineralogy and oxide composition of the silt fraction.....	14
Table A3 – Mineralogy and oxide composition of the silt fraction.....	15
Table A4 – Mineralogy and oxide composition of the silt fraction.....	17
Table A 5 – Mineralogy and oxide composition of the silt fraction.....	18

FIGURES

Figure 1 – Particle size distribution of UTS Fort Hills core samples.....	8
Figure 2 – TiO ₂ and ZrO ₂ depth profiles.....	8
Figure 3 – Concentration of titanium oxide minerals in the ores and froth minerals of an Athabasca oil sands lease.	9
Figure 4 – Concentration of zircon in the ores and froth minerals of an Athabasca oil sands lease.	9
Figure 5 – Variation of clay minerals properties with depth.	10
Figure A1 – UTS core #1. SEM image showing the particles with heavy elements brighter than the sand particles.	12

Figure A 2 – X-ray diffraction profile of the silt fraction of core 1 13
Figure A 3 – Backscattered electron image of core 17. 13
Figure A4 – Backscattered electron image of another region of core 17. 14
Figure A5 – X-ray diffraction profile of the silt fraction of core 17 14
Figure A6 – Backscattered electron image of a region of core 17 rich in heavy minerals 15
Figure A7 – X-ray diffraction profile of the silt fraction of core 21 16
Figure A 8 – Backscattered electron image of a region rich in pyrite in core 28. 16
Figure A9 – X-ray diffraction profile of the silt fraction of core 28 17
Figure A 10 – Backscattered electron image of a region rich in pyrite in core 41 18
Figure A11 – X-ray diffraction profile of the silt fraction of core 41 18

INTRODUCTION

In the past 20 years, several studies have been conducted to evaluate the potential for producing heavy mineral concentrates from oil sands. The most detailed study was the Minerals Development Agreement research project on valuable minerals from oil sands (1). A total of 288 samples were examined from various regions in the McMurray Formation including the Syncrude, Suncor, and Gulf leases. Titanium oxides averaged 0.47 wt% in the Lower McMurray fluvial deposits, 0.50 wt% in the Middle McMurray Estuarine deposits and 0.32 wt% in the Upper McMurray marine deposits. Zircon was about 0.024 wt% across the region. Titanium oxides were reported primarily rutile and leucoxene. Ilmenite was also found in minor quantities. Similar speciation was found in other studies except that the mineral reported as leucoxene is primarily anatase with a fine amorphous iron oxide coating, which is ubiquitous in oil sands (2, 3). In addition, the bulk of the heavy minerals were found to be concentrated in the sand fraction (plus 44 μm).

Most of the research efforts focused on froth treatment tailings, where heavy minerals are preferentially concentrated due to their oleophilic surfaces. While heavy mineral concentration in the froth is dependent on extraction process variables, TiO_2 has been reported in the range 4 to 15 wt% and zirconium oxides in the range 1 to 3.6 wt% (1-5). At these concentrations, the froth treatment tails are one of the richest sources of rutile and zircon in the world.

At the current production level of froth treatment tailings, over 1.2 million tonnes of TiO_2 and zircon are produced per year. Despite the abundance, investment in froth treatment heavy minerals production has been slow primarily due to the affinity of heavy minerals for organic components in oil sands. The bound organics affect the magnetic, electrostatic, and sedimentation properties of the heavy minerals, precluding the use of conventional mineral sands technologies to upgrade the tailings. The development of technologies to economically remove the residual hydrocarbons has been challenging. The technologies that have been suggested include: roasting (1, 4, 5) and chemical treatment.

MATERIALS AND METHODS

Five oil sands core samples from the UTS Fort Hills lease were evaluated. The samples had been previously stored at room temperature for an extended period of time prior to receipt at CANMET.

To remove bitumen from the core samples, a mixture of IPA/toluene was used to rinse the solids until all the bitumen had been removed (cold washing). The bitumen-free minerals were thoroughly dispersed in deionized water and sieved through a 44- μm mesh screen. The fines fraction was centrifuged to separate the clay (minus 2- μm) fraction. Heavy media separation was carried out on the sand fraction to separate minerals heavier than 2.9 g/cm³ in cores 1 and 17.

Prior to size separation, microscopic investigation of the minerals was conducted using a Hitachi 4800 high-resolution scanning electron microscope. Images were collected in the backscattered electron mode to enable visual observation of high-atomic-number elements (associated with heavy minerals). Chemical information on the microstructure was obtained using an attached energy-dispersive spectrometer.

Detailed mineralogical analysis was conducted on the sand (plus 44- μm), silt (minus 44- μm to plus 2- μm) and clay (minus 2- μm) fractions using x-ray diffraction and fluorescence techniques.

Diffraction data were collected using a Bruker D8 θ - θ x-ray diffractometer equipped with a CoK α monochromating multilayered mirror on the incident and diffracted beam sides (twin mirrors). The silt fractions were quantified using TOPASTM (a Rietveld refinement software). The basal diffraction pattern (001) of mixed-layer clays (from glycolated slides) was modeled using NEWMOD^R to determine the clay mineral content. Bulk elemental composition was obtained using a wavelength-dispersive x-ray fluorescence spectrometer (Bruker S4 Explorer).

RESULTS AND DISCUSSION

The advantage of oil sands tailings as a source for heavy mineral concentrates over the traditional mineral sand deposits stems from a side-effect of the bitumen extraction process. Bitumen has a greater affinity for heavy mineral surfaces than quartz and clay minerals at the extraction pH (7 to 8.5) and the heavy minerals are therefore

preferentially concentrated in the bitumen froth phase. Subsequent froth treatment generates tailings that are rich in these heavy minerals while the bulk of quartz and clay minerals report to the extraction tailings stream.

The concentration of heavy minerals in the froth treatment tailings is highly dependent on process variables that affect bitumen extraction and flotation efficiency. For example, highly degraded bitumen generates froth of poor quality containing high concentrations of quartz and clay minerals in addition to the heavy minerals. Given that the Fort Hills core samples had most likely been oxidized during prolonged storage at room temperature, standard extraction tests would result in poor froth quality and unusually low heavy-mineral concentrations compared to fresh ores from other commercial leases. Comparisons made to existing oil sands leases are therefore based on the heavy-mineral contents in the ore samples.

PARTICLE SIZE DISTRIBUTION

The particle size distributions are given in Table 1 and Figure 1. All the core samples are low-fines ores (less than 10 wt%). The median fines content in Athabasca oil sands is about 15 wt% and can be as low as 5 wt% and as high as 40 wt%. The clay content (minus 2 μm) is also on the low side of the average usually observed in oil sands. Core 17, has by far the smallest d_{50} (approximately 100- μm) of the five samples.

HEAVY MINERALS ANALYSIS

Bulk chemical analysis given in Table 2 shows that the bitumen-free samples are made up of over 95% quartz and clays. The depth profiles of the titanium oxide and zirconium oxide associated with valuable minerals are illustrated in Figure 2. The TiO_2 and ZrO_2 concentrations range between 0.06 wt% in the mineral sand core 41 and core 28, and 0.5 wt% in the Upper McMurray core 17. These concentrations of heavy minerals are far below the 3–5 wt% typically observed in the traditional mineral sands operations around the world. The heavy mineral levels are, however, within the range typically observed in Athabasca oil sands. To improve detection of the valuable minerals of interest, backscattered electron imaging, sensitive to atomic number was used for microstructural analysis. Mineralogical evaluation was conducted based on size fractions.

Density separation in a heavy medium (SG 2.9) was conducted on cores 1 and 17 but did not offer greater detection of heavy minerals than separation based on size.

The concentration of heavy minerals in the ore and froth tailings of core samples from an oil sands lease spanning the Upper to Lower McMurray Formation is illustrated in Figures 3 and 4. The data, based solely on the contribution of heavy minerals in the fines fraction (minus 44- μm), show a range of total TiO_2 (anatase and rutile) between 0.01 and 0.9 wt% in the ore. The concentration of zircon ranges from less than 10 ppm to 200 ppm. The fines mineralogy of the UTS core samples are within these ranges, albeit at the low end (0.02 to 0.11 wt% for TiO_2 and 7 to 450 ppm for zircon). After bitumen extraction, the froth solids contain TiO_2 minerals between 4 and 11 wt% and the concentration of zircon is approximately 1 wt%. The variation in the froth mineralogy of the samples reported in Figure 3 and 4 is due to varying ore processability potentials. A similar several-hundred-fold increase in heavy mineral concentrations in froth tailings is expected for the UTS lease.

Details of the chemistry and mineralogy of each fraction are presented in Appendix A.

CLAY MINERALOGY

The clay mineralogy of the deposit is essential in terms of both the processability of the oil sand and subsequent tailings behaviour. Any heavy mineral operation seeking to remove organics from the mineral phase has to contend with clay minerals, as these minerals tend to strongly associate with both organics and heavy minerals due to their small size and permanent charge structures. The variation of the clay mineralogy with burial depth could also serve to elucidate the nature of alteration reactions during burial and in situ bitumen chemistry.

The primary clay minerals observed in all five cores are kaolinite, kaolinite-smectite, illite, and illite smectite (Table 3). Minor quantities of smectite and chlorite were observed in core 1. The clay mineralogy is typical of the McMurray Formation (2). It is important to note that the specific surface area of the clay minerals increases with depth within the bitumen-rich formations (cores 1 to 28) primarily due to increasing concentrations of interstratified kaolinite-smectite (Figure 5). Typically, the rate of transformation of smectite to kaolinite via kaolinite-smectite is limited by drainage. The

deviation of core 41 from linearity results directly from the impact of the hydrocarbon components on metamorphic reactions involving clay minerals.

CONCLUSIONS

The heavy mineral concentration in the Fort Hills oil sands core samples is consistent with concentrations previously observed in several other leases across the McMurray Formation. The Middle McMurray sample contains the highest amounts of TiO_2 (0.4 wt%) and ZrO_2 (0.08 wt%). The bitumen-free mineral sand deposit (core 41) contains only 0.06 wt% TiO_2 and 0.008 wt% zircon, about one-tenth the concentration in the Middle McMurray. On average, about 70 wt% of the TiO_2 and 63 wt% of the ZrO_2 are concentrated in the sand fraction.

More work would be required to investigate the variability of the heavy minerals in the bitumen-free core since these hydrocarbon-free parts of the deposit are the most amenable to conventional heavy mineral separation techniques. The single "water sands" or bitumen-free sample studied here had a less than commercial TiO_2 or ZrO_2 concentration. The samples which contained bitumen would be expected to result in a froth treatment tailings similar to other commercial tailings. The other deposits in the McMurray Formation would have to be upgraded from froth treatment tailings in line with the current understanding of the heavy minerals surface chemistry.

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Table 1 – Particle size distribution of the bitumen-free UTS Fort Hills oil sands core samples. The fractions were obtained from a combination of wet sieving and centrifugation.

Size (μm)	Weight per cent less than				
	Core 1	Core 17	Core 21	Core 28	Core 41
250	73.2	99.6	50.8	44.9	47.2
150	36.5	90.4	20.9	22.2	21.4
106	19.4	57.8	13.2	15.3	15.1
75	12.5	24.1	8.5	10.4	11.7
45	9.3	9.2	5.4	6.9	8.7
2	2.0	2.1	0.9	0.8	1.6

Table 2 – Bulk chemical analysis of the core samples

Core #	Oxide in weight per cent											
	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	Cl	K ₂ O	CaO	TiO ₂	Cr ₂ O ₃	Fe ₂ O ₃
1	0.06	0.17	2.3	95.4	0.05	0.5	0.02	0.7	0.19	0.12	0.14	0.46
17		0.08	3.0	94.6	0.03	0.4	0.02	0.8	0.08	0.41	0.03	0.46
21	0.03	0.02	1.5	97.4	0.02	0.2	0.02	0.5	0.03	0.10	0.28	0.16
28	0.05		0.9	97.7		0.7	0.02	0.2	0.02	0.05	0.30	0.32
41	0.19		2.0	93.7	0.02	2.1	0.22	0.7	0.11	0.06	0.00	0.93

Core #	Oxide in ppm							
	CoO	CuO	ZnO	Rb ₂ O	SrO	Y ₂ O ₃	ZrO ₂	CeO ₂
1		51	22	16	40	18	123	
17		44	22	24	54		826	
21		42			33	9	120	
28						11	65	
41	34	49			33		83	220

Table 3 – Mineral composition of the core samples. The sand fraction was assumed to be 100 wt% quartz.

Core #	Mineral in weight per cent of total solids												
	K-S	Kaolinite	I-S	Illite	Chlorite	Smectite	Quartz	Pyrite+ Marcasite	K- Feldspar	Rutile+ Anatase	Zircon	Dolomite+ Ankerite	Siderite
1	0.1	1.5	0.2	1.0	0.2	0.01	95.6	0.20	0.8	0.05	0.00	0.16	0.10
17	0.5	1.7	0.2	0.7	0.0	0.00	96.4	0.00	0.0	0.12	0.05	0.03	0.21
21	0.2	0.7	0.1	0.3	0.0	0.00	98.3	0.00	0.3	0.03	0.00	0.01	0.02
28	0.4	0.3	0.0	0.2	0.0	0.00	98.7	0.12	0.3	0.02	0.00	0.01	0.00
41	0.5	1.0	0.0	0.2	0.0	0.00	96.6	0.62	0.9	0.03	0.01	0.05	0.00

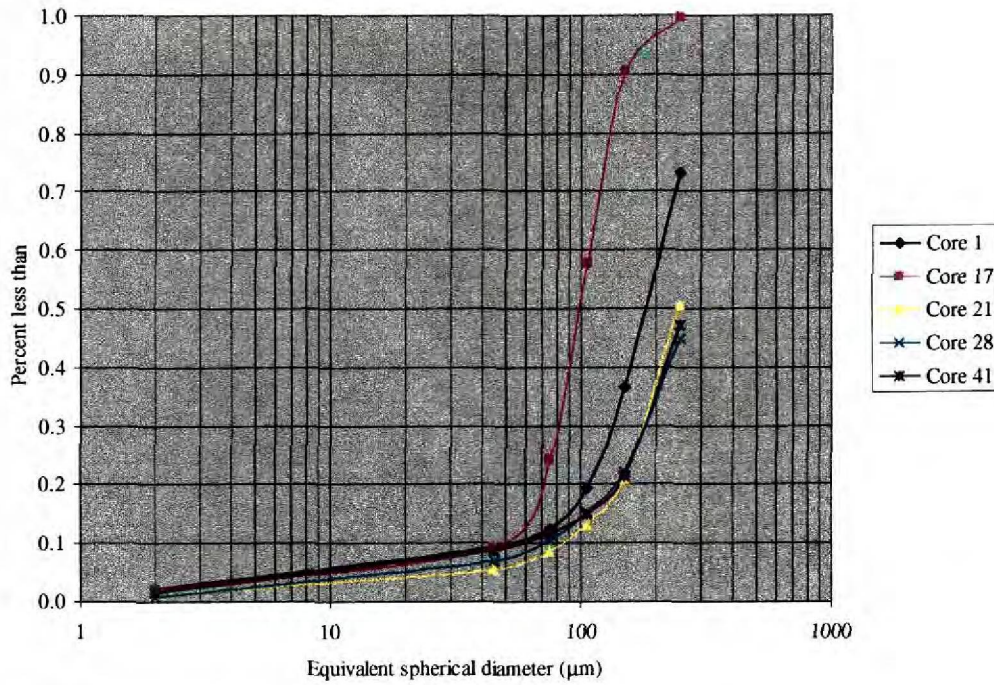


Figure 1 – Particle size distribution of UTS Fort Hills core samples

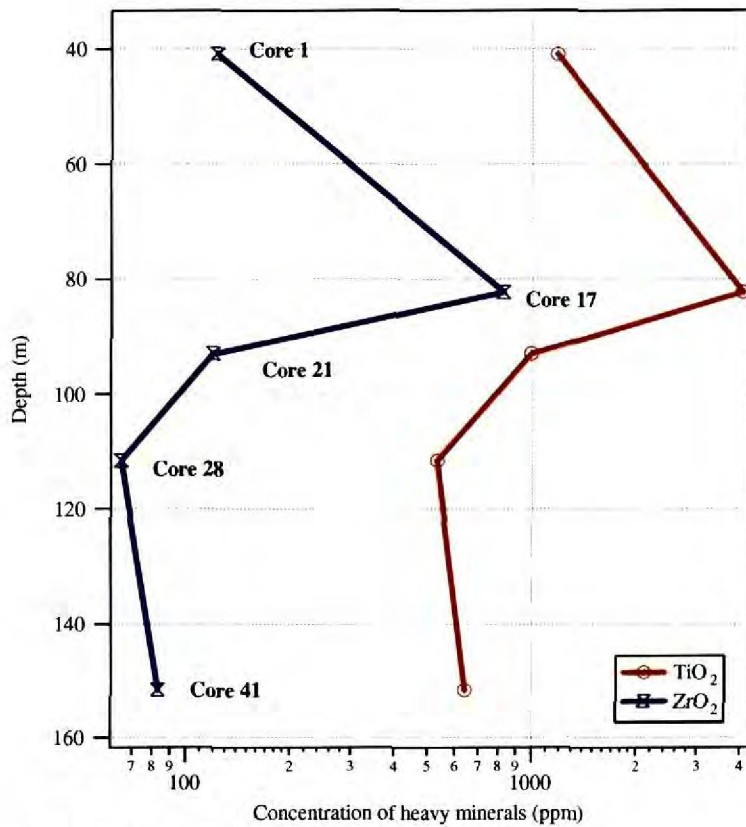


Figure 2 – TiO₂ and ZrO₂ depth profiles

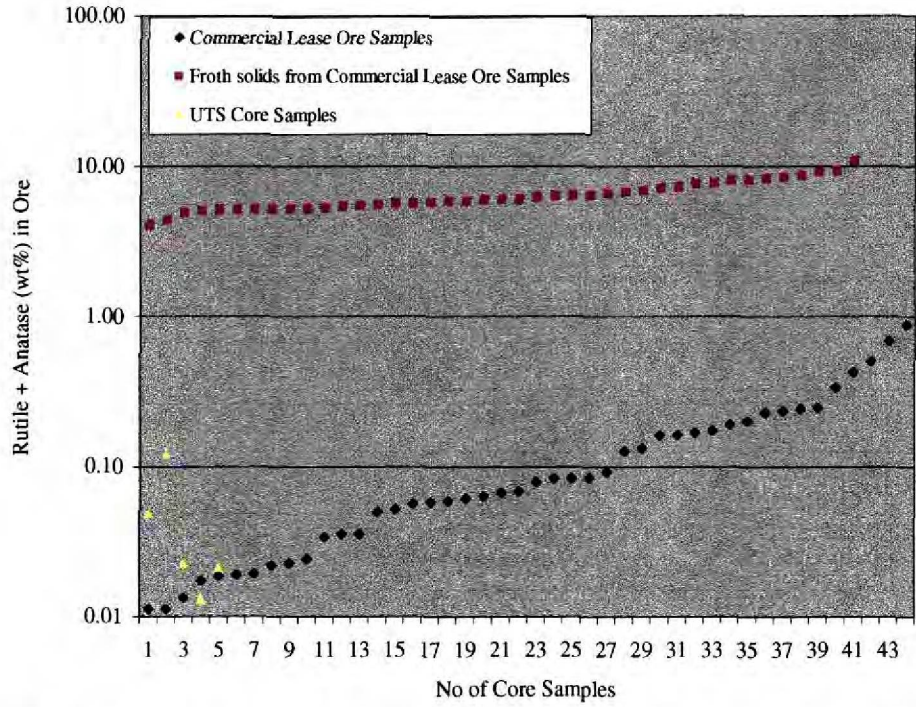


Figure 3 – Concentration of titanium oxide minerals in the ores and froth minerals of an Athabasca oil sands lease. The mineral content is based on contributions from the fines fraction.

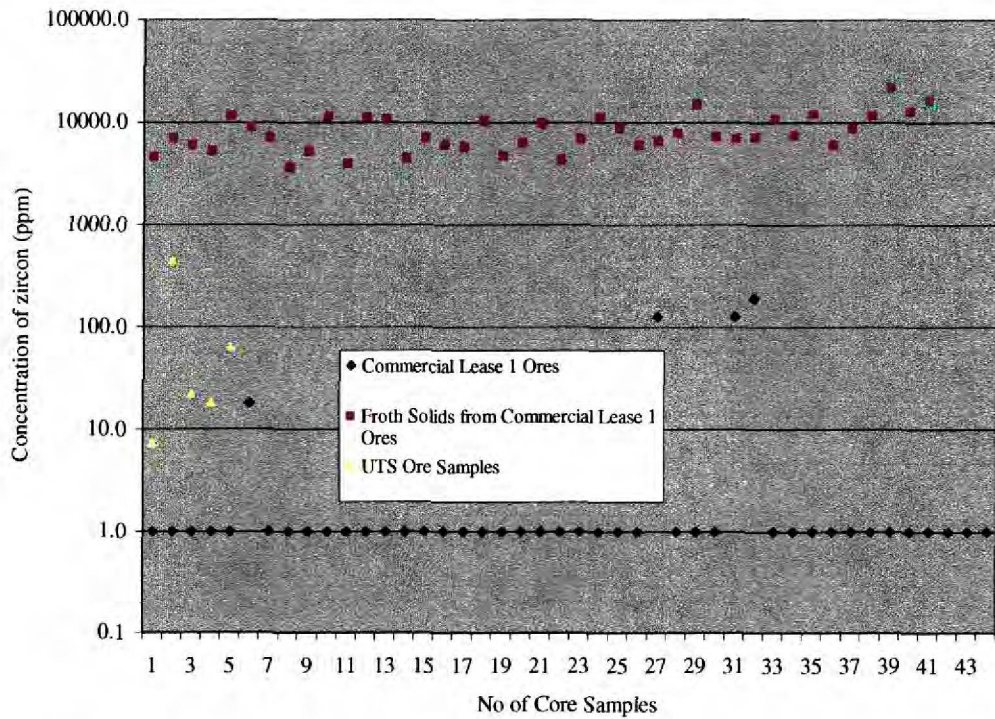


Figure 4 – Concentration of zircon in the ores and froth minerals of an Athabasca oil sands lease. The mineral content is based on contributions from the fines fraction.

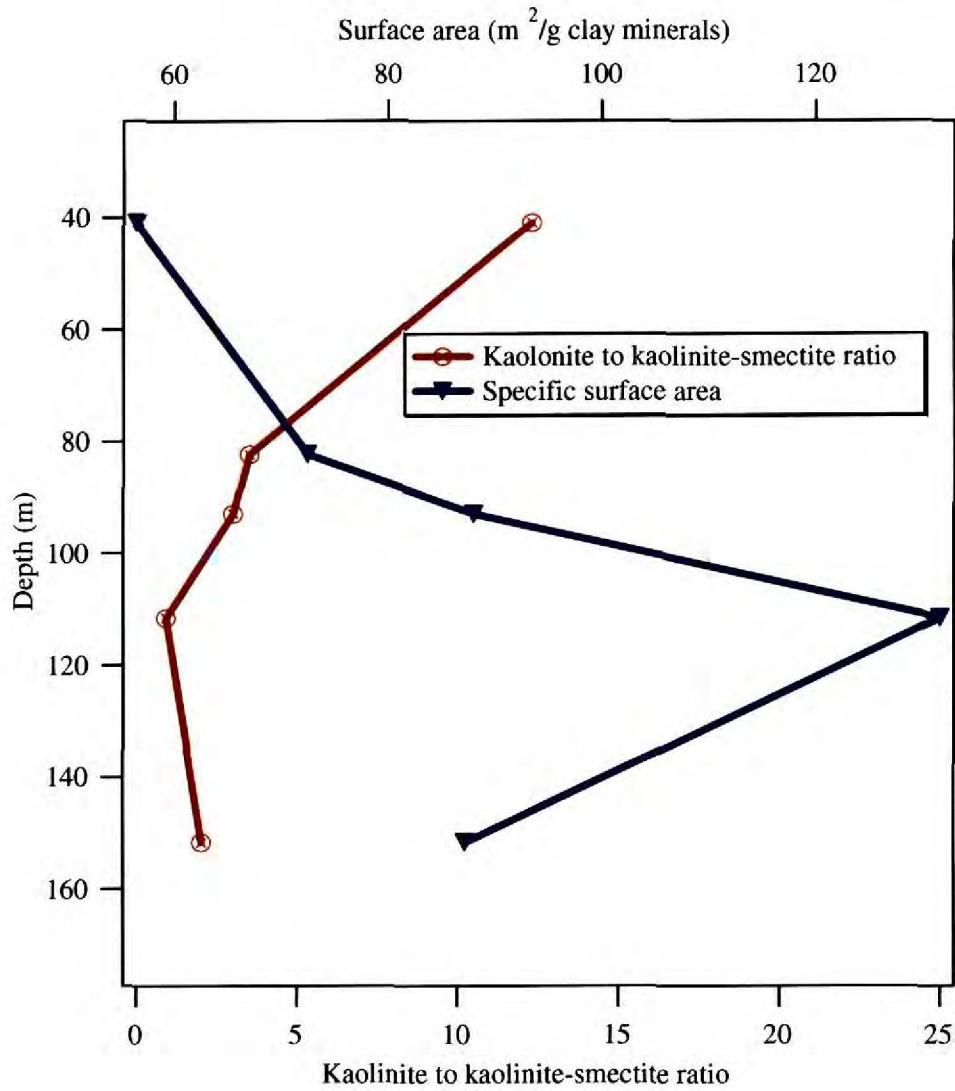


Figure 5 – Variation of clay minerals properties with depth. Both the surface area and interstratified components have implications on clay-organic interactions and tailings behaviour

**APPENDIX A:
MICROSTRUCTURAL AND MINERALOGICAL DATA**

Core 1: 1AA-02-30-96-10W4 INT. 39.04-42.78 m REC. 3.05/3.74 m

Figure A1 shows a backscattered electron image of core 1. Heavy mineral particles are bright on a dark quartz and aluminosilicate matrix. A close-up of a representative bright area comprising framboidal-shaped pyrite is shown in the right micrograph. The bulk elemental analysis in Table 2 shows the presence of minor to trace amounts of titanium and zirconium oxides. X-ray diffraction analysis shows TiO₂ as primarily rutile and ZrO₂ as zircon. The diffraction pattern of the silt-sized fraction is given in Figure A2 where the pattern is not dominated by quartz (as in the sand fraction or bulk) and contains measurable concentrations of heavy minerals. The corresponding mineralogy and oxide composition in the silt fraction are given in Table A1.



Figure A1 – UTS core #1. SEM image showing the particles with heavy elements brighter than the sand particles. The close-up in the lower micrograph highlights pyrite mineral with framboidal shape.

Table A1 – Mineralogy and oxide composition of the core 1 silt fraction.

Core #	Mineral in weight per cent										
	Kaolinite	Mica	Chlorite	Quartz	Pyrite	Marcasite	K-Feldspar	Rutile	Zircon	Dolomite	Siderite
1	10.2	8.2	1.0	63.3	2.3	0.5	10.5	0.6	0.1	2.1	1.4

Core #	Oxide in weight per cent											
	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	Cl	K ₂ O	CaO	TiO ₂	Cr ₂ O ₃	Fe ₂ O ₃
1	0.2	1.0	10.1	76.8	0.2	3.2	0.1	2.4	1.2	0.7	0.2	3.6

Oxide in ppm									
NiO	CuO	ZnO	Rb ₂ O	SrO	Y ₂ O ₃	ZrO ₂	MoO ₂	CeO ₂	
825	120	82	68	110	44	667	272	420	

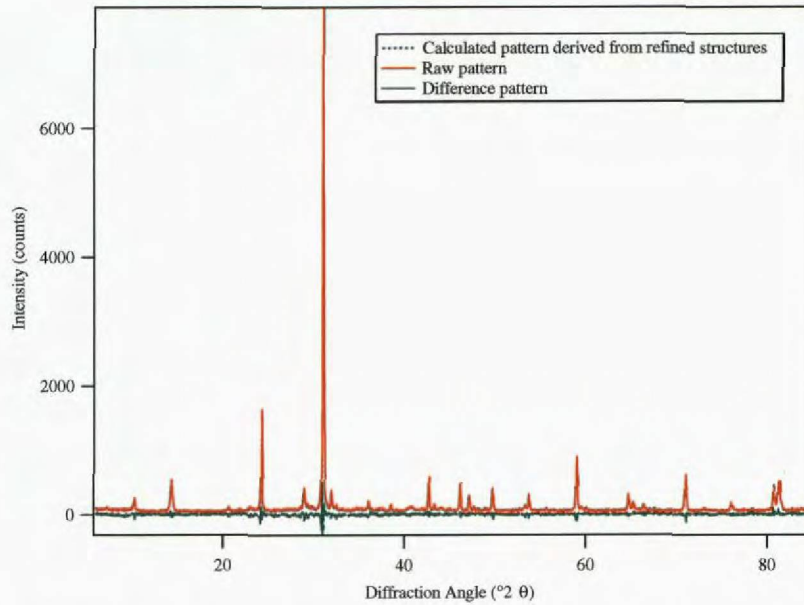


Figure A 2 – X-ray diffraction profile of the silt fraction of core 1

Core 17: 1AA-02-30-96-10W4 INT. 79.09-85.51 m REC. 2.91/3.42 m

Core 17 contains the most titanium and zirconium oxides (Table 2 and Table A2). Typical microstructure and heavy element chemistry are shown in Figures A3 and A4. Several bright areas in the microstructure are rich in Fe, Ti, or Zr or a combination of these elements. The speciation of TiO_2 was confirmed as mostly anatase and rutile from the x-ray diffraction analysis (Figure A5).

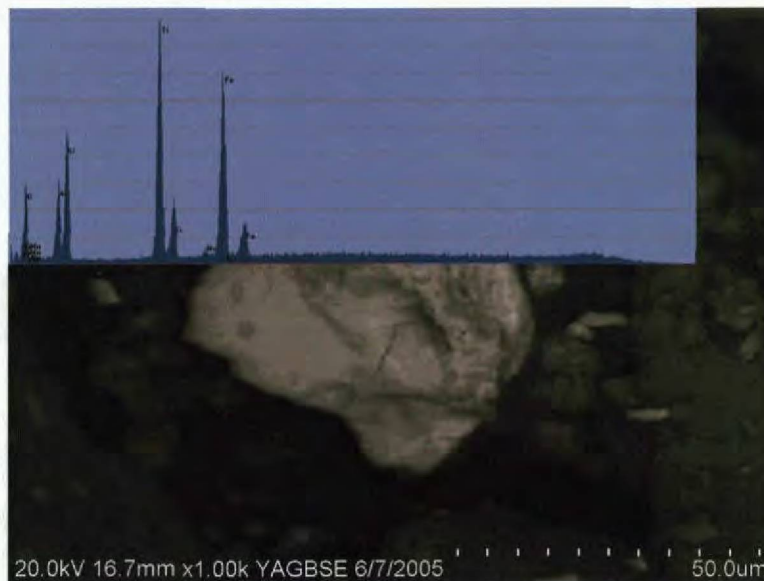


Figure A 3 – Backscattered electron image of core 17. The elemental spectrum (inset) is from the bright region and is predominantly titanium oxide with substantial iron oxide.



Figure A4 – Backscattered electron image of another region of core 17. The elemental spectrum (inset) is from the zircon-rich bright region.

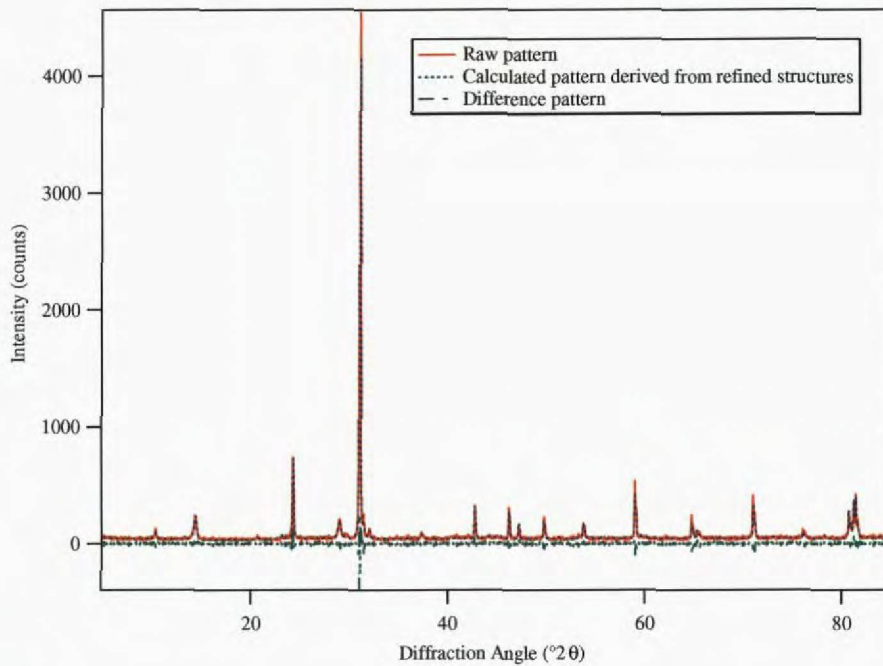


Figure A5 – X-ray diffraction profile of the silt fraction of core 17

Table A2 – Mineralogy and oxide composition of the silt fraction

Core #	Mineral in weight percent											
	Kaolinite	Mica	Chlorite	Quartz	Pyrite	Marcasite	K-Feldspar	Rutile	Anatase	Zircon	Ankerite	Siderite
17	12.2	5.6	0	76.6	0	0	10.5	0.8	0.8	0.6	0.5	3.0

Core #	Oxide in eight percent											
	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	Cl	K ₂ O	CaO	TiO ₂	Cr ₂ O ₃	Fe ₂ O ₃
17	0.2	0.4	9.8	81.2	0.1	0.9	0.1	2.0	0.3	1.6	0.0	2.8
	Oxide in ppm											
	NiO	CuO	ZnO	Rb ₂ O	SrO	Y ₂ O ₃	ZrO ₂	MoO ₂	CeO ₂			
		150		54		120	5348		440			

Core 21: 1AA-02-30-96-10W4 INT. 91.50-94.42 m REC. 2.92/2.92 m

Both titanium and zirconium oxides are present at this depth but at concentrations much lower than core 17. A typical microstructure is shown in Figure A6. Similar to cores 1 and 17, the predominant heavy minerals are rutile, anatase, and zircon (Table A3 and Figure A7).



Figure A6 – Backscattered electron image of a region of core 17 rich in heavy minerals. The elemental spectrum (inset) is primarily from the zircon-rich bright region

Table A3 – Mineralogy and oxide composition of the silt fraction

Core #	Mineral in weight per cent											
	Kaolinite	Mica	Chlorite	Quartz	Pyrite	Marcasite	K-Feldspar	Rutile	Anatase	Zircon	Ankerite	Siderite
21	8.7	3.9	0	79.4	0	0	6.9	0.4	0.03	0.05	0.1	0.5
Core #	Oxide in weight per cent											
	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	Cl	K ₂ O	CaO	TiO ₂	Cr ₂ O ₃	Fe ₂ O ₃
21		0.1	6.2	87.9	0.0	0.8	0.1	1.4	0.1	0.5	0.3	2.0
	Oxide in ppm											
	NiO	CuO	ZnO	Rb ₂ O	SrO	Y ₂ O ₃	ZrO ₂	MoO ₂	CeO ₂			
	1550	130	38	44	86	46	881	554				

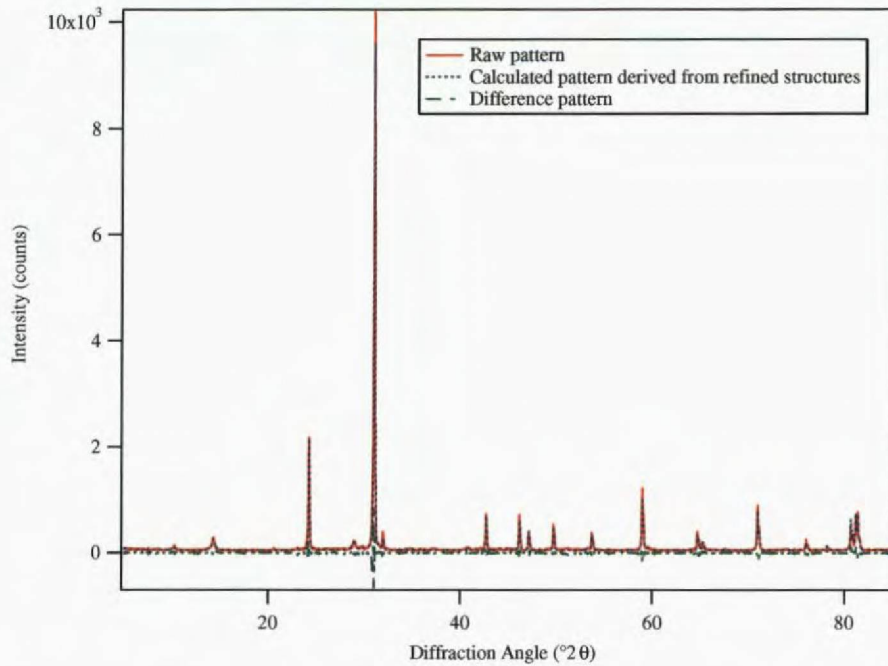


Figure A7 – X-ray diffraction profile of the silt fraction of core 21

Core 28: 1AA-02-30-96-10W4 INT. 110.01-113.16 m REC. 2.84/3.15 m

Core 28 contains the least amount valuable minerals. The only detectable heavy mineral in the SEM is pyrite (Figure A8). X-ray diffraction analysis revealed trace amounts of rutile and zircon (Figure A9 and Table A4).

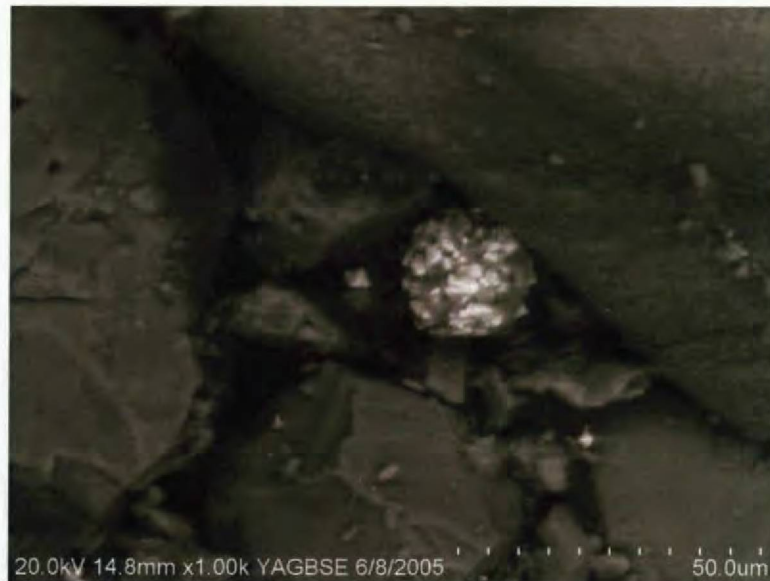


Figure A 8 – Backscattered electron image of a region rich in pyrite in core 28. No zirconium-or titanium-rich areas were identified.

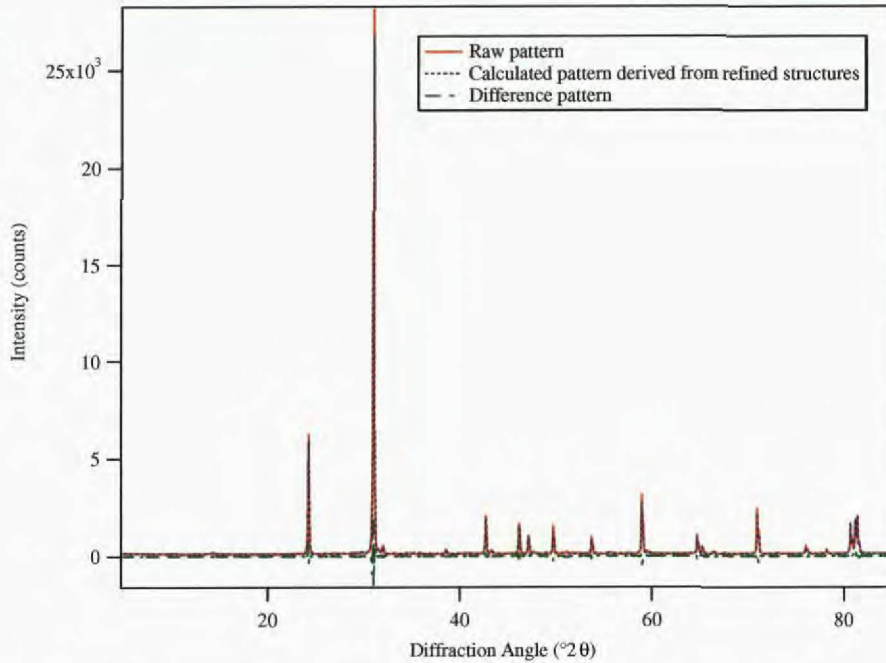


Figure A9 – X-ray diffraction profile of the silt fraction of core 28

Table A4 – Mineralogy and oxide composition of the silt fraction

Core #	Mineral in weight per cent											
	Kaolinite	Mica	Chlorite	Quartz	Pyrite	Marcasite	K-Feldspar	Rutile	Anatase	Zircon	Ankerite	Siderite
28	3.5	1.4	0	87.3	1.9	0.1	5.4	0.13	0.03	0.03	0.1	0.5

Core #	Oxide in weight per cent											
	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	Cl	K ₂ O	CaO	TiO ₂	Cr ₂ O ₃	Fe ₂ O ₃
28			2.5	90.3	0.0	2.6	0.1	1.0	0.1	0.2	0.3	2.5

Oxide in ppm									
NiO	CuO	ZnO	Rb ₂ O	SrO	Y ₂ O ₃	ZrO ₂	MoO ₂	CeO ₂	
1650	180	69		61		291	564		

Core 41: 1AA-02-30-96-10W4 INT. 151.13-152.20 m REC. 1.37/1.57 m

The mineral sands core 41 above the Devonian shale contains trace amounts of valuable minerals similar to core 28. However, there is a high concentration of pyrite in the sample (Figure A10). Large pyrite crystals are fairly commonplace in the microstructure. This was confirmed by both x-ray diffraction analysis and x-ray fluorescence oxide analysis (Figure A11 and Table A5).

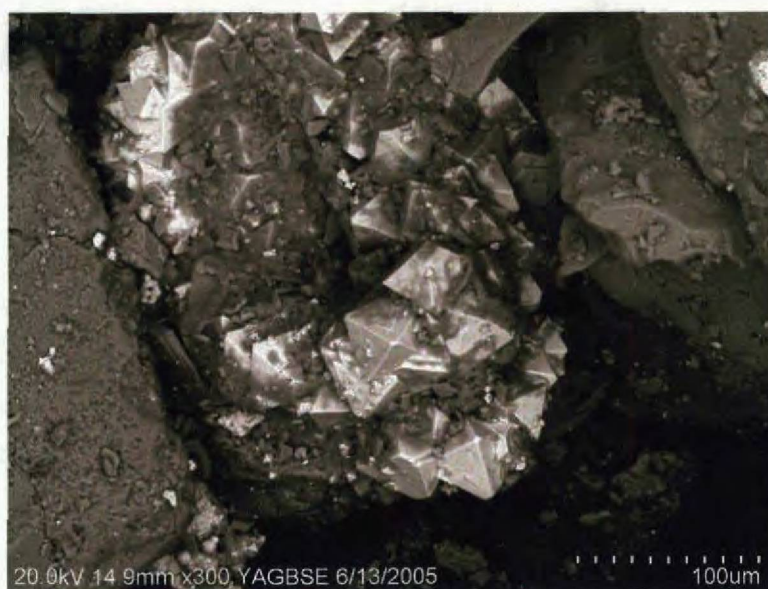


Figure A 10 – Backscattered electron image of a region rich in pyrite in core 41

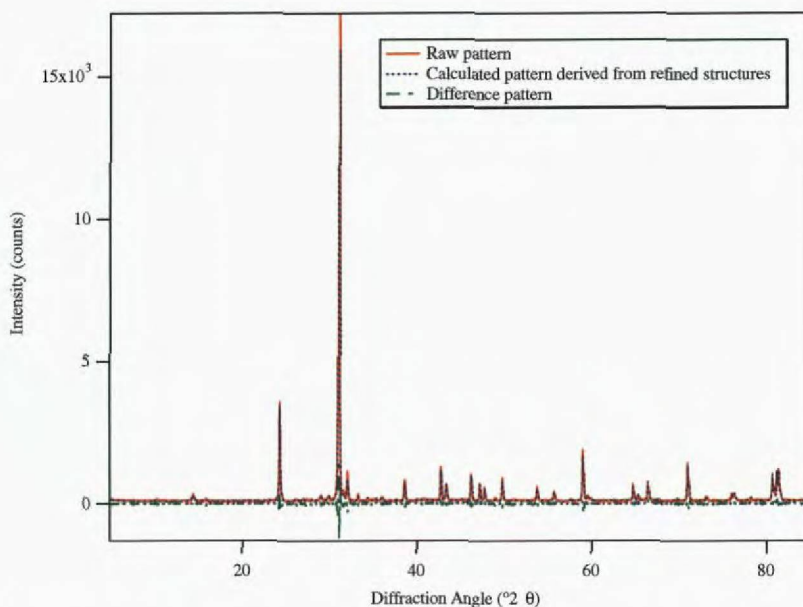


Figure A11 – X-ray diffraction profile of the silt fraction of core 41

Table A 5 – Mineralogy and oxide composition of the silt fraction

Core #	Mineral in weight per cent											
	Kaolinite	Mica	Chlorite	Quartz	Pyrite	Marcasite	K-Feldspar	Rutile	Anatase	Zircon	Ankerite	Siderite
41	3.9	1.9	0	71.6	8.5	0.2	13.3	0.2	0.03	0.09	0.7	0.03
Core #	Oxide in weight per cent											
	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	Cl	K ₂ O	CaO	TiO ₂	Cr ₂ O ₃	Fe ₂ O ₃
41	0.1	0.0	3.8	79.0	0.0	8.3	0.1	2.2	0.2	0.1	0.2	5.7

Oxide in ppm								
NiO	CuO	ZnO	Rb ₂ O	SrO	Y ₂ O ₃	ZrO ₂	MoO ₂	CeO ₂
1010	120	45	53	88		354	405	51

R11

R10W4

111.6516667
 S7 39S2778
 UTM 6361575
 460833 212

T97

T97

57°23'43"
 111°39'6"

Permit #
9303070760

1AA-2-30

T96

T96

57°20'15"
 111°36'18" UTM 6355117
 463581
 111.605
 S7.3375 212

R11