

A Reconnaissance Lithochemical Study of Upper Cretaceous Whitemud Formation and Paleocene Ravenscrag Formation Kaolinitic Clays in Southern Saskatchewan



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Abstract

The results of a multi-element geochemical study are reported from the sampling of kaolinitic core sections from 20 widely spaced drillholes in far southern Saskatchewan. The focus of the sampling was on core that intersected the kaolin-rich Upper Cretaceous Whitemud Formation, with a secondary emphasis on core from the kaolinitic Willow Bunch Member (WBM) of the Paleocene Ravenscrag Formation, plus samples from a small number of other clay deposits.

Of particular interest were the minor element contents of the 29 samples that were taken, especially the contents of the rare earth elements (REE), as residual REE-bearing ion-adsorption kaolinitic clays, which are currently mined in South China, are the world's largest source of the valuable heavy REEs.

The core from all 20 of the drillholes that were sampled are stored in Regina at the Saskatchewan Subsurface Geological Laboratory of the Ministry of Energy and Resources. Individual samples comprised selective spot sampling of available chips through the entire kaolinitic interval in each section of core. In a few of the drillholes with particularly thick kaolinitic sections, two, and in one core three, samples were required. The samples were sent to the Saskatchewan Research Council's Geoanalytical Laboratory for multi-element analysis by inductively coupled plasma-optical emission spectroscopy, which included both total and partial digestion packages.

The arithmetic mean (total digestion) for all 29 samples is 160.9 ppm total (T)REE+Y,Sc, with a tight range from 91.4 ppm to 236.3 ppm, except for a value of 365.1 ppm TREE+Y,Sc from a Ravenscrag WBM stoneware clay location at Rockglen. This sample is considered to be modestly anomalous relative to the background values from the other samples. All of the samples have relatively low heavy REE/light REE ratios (0.38 mean value), which are not economically favourable.

The results of this reconnaissance sampling project do not support the hypothesis that kaolinitic clays of the Upper Cretaceous Whitemud Formation and Paleocene Ravenscrag Formation contain ion-adsorption-type REE mineralization. The results also indirectly support the interpretation that the kaolin was formed in situ at the site of deposition and was not transported from weathered, REE-bearing source rocks. Also, these relatively low REE contents likely reside within the mineral structures of the samples, rather than being adsorbed onto the surfaces of the kaolin, although a study would have to be done to determine this.

This modest project only involved the sampling of 20 widely spaced drillholes in kaolin and minor volcanic ash/bentonite locations from across southern Saskatchewan. Although unlikely, there could be substantial areas of kaolin in local settings with elevated REE values related to ion-adsorption. Of note is that the anomalous sample was taken from a Ravenscrag WBM location at Rockglen and was one of only five drillholes in the WBM that were sampled in this study. This may warrant a further investigation of WBM locations, including Rockglen, in the south-central and southeastern areas of the province.

Keywords: kaolin, geochemistry, rare earth elements, Upper Cretaceous, Whitemud Formation, southern Saskatchewan, ion-adsorption clays

1. Introduction

The purpose of this reconnaissance lithogeochemical study was to develop an understanding of the minor element contents, and more specifically the rare earth element (REE) contents, of widely distributed kaolin clay deposits throughout far southern Saskatchewan, by analysis of samples of drill core (refer to Figure 1). Charles Normand (personal communication, 2018) of the Saskatchewan Geological Survey suggested that this could be a useful project, based on his understanding of ion-adsorption, REE-bearing clay deposits, which are currently mined in South China and are the world's largest source of valuable heavy REEs (HREEs). The South China deposits are associated with residual kaolin developed through chemical weathering of REE-bearing source rocks. Normand (2014) noted similarities between these South China deposits and the Upper Cretaceous Whitemud Formation in Saskatchewan. The latter consists of up to 22 m of kaolinitic sediments, and is widely distributed in the south-central and southwest portions of the province. This study is based on the premise that at least a portion of the kaolin was formed at the source from weathering of REE-bearing bedrock and then transported fluviially to southern Saskatchewan.

The focus of the sampling was on core from the kaolin-rich Whitemud Formation (Fm.), with secondary emphasis on the kaolinitic Willow Bunch Member (WBM) of the Paleocene Ravenscrag Fm., plus a small number of other clay deposits. Twenty drillholes were sampled (Figure 1) and a total of 29 samples collected. These drillholes, along with numerous other drillholes in clay deposits and locations, are available for examination and study at the Saskatchewan Ministry of Energy and Resources Subsurface Geological Laboratory (SGL) in Regina.

2. Ion-adsorption Clay Deposits

The following summary is from Van Gosen *et al.* (2017), who provide a general summary of REE deposit types, including a section on ion-adsorption clay deposits, and from Cocker (2014), who documents lateritic, supergene rare earth element deposit types, of which ion-adsorption clays are a category. Normand (2014) provides a thorough description of the known REE deposit types in Saskatchewan. Rare earth elements comprise the 15 lanthanide series elements, from lanthanum (La, atomic number 57) to lutetium (Lu, 71). The 15 rare earth elements are La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb and Lu. Yttrium (Y) and scandium (Sc) are also generally included. The HREEs comprise terbium (Tb, 65) to lutetium, plus Y, Sc.

Ion-adsorption REE-bearing clay deposits are formed as the result of deep weathering of REE-enriched bedrock in subtropical to tropical environments with moderate to heavy rainfall. Primary source rocks typically consist of granites, rhyolites, carbonatites and various metamorphic rocks. Weathering profiles can extend to depths of 30 to 60 m, and consist of a depleted surface zone (A), enriched zone (B), and partially weathered zone (C) overlying the bedrock protolith. Ion-adsorption REE-bearing clay deposits are secondary, supergene deposits in which REEs have been leached and concentrated by groundwater, and are characterized by kaolinite, halloysite ± gibbsite clays. The mobilized REEs become weakly adsorbed onto the clays. The types of REEs and their concentrations reflect those of the bedrock source. Concentrations of REEs in the clays are generally 3 to 10 times those of the protolith. Mineable grades in South China range from 300 to 5000 ppm REEs. Those with high levels of the valuable HREEs can be mined at lower concentrations. These are large-tonnage, low-grade deposits and the REEs can be readily recovered with saline solutions. World examples include the Zudong mine, South China, and the Dong Pao, Vietnam; Tantalus, Madagascar; and Stromberg, Australia deposits. Li *et al.* (2019) describe the world-class Zudong HREE ion-adsorption clay mine of South China. It is the world's largest single producer of HREEs, with a resource of 17 600 tonnes rare earth oxides (REO) at an ore grade of about 1000 ppm REO. It was formed by the *in situ* weathering of a HREE-enriched (200 to 450 ppm total REEs) A-type granite. The various orebodies range from a few metres to 10 metres in thickness. REEs are adsorbed to kaolinite and halloysite clays.

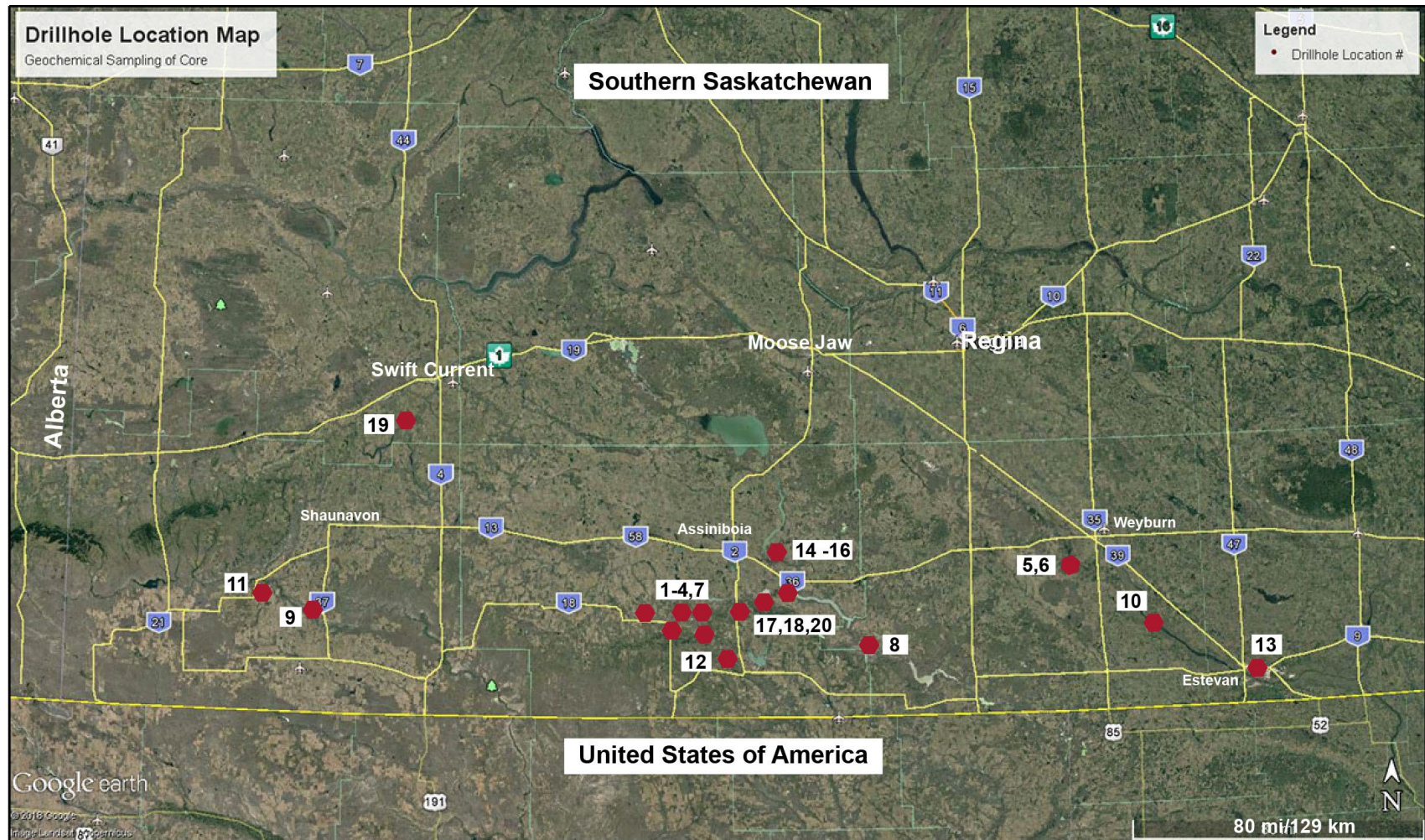


Figure 1 – Locations of the drillholes that were sampled for this study: 1) Ekaton Gollier Creek Kaolin WM87-175; 2) Ekaton Project 12 Kaolin WM87-200; 3) Ekaton Wood Mountain Kaolin WM88-237; 4) Ekaton Wood Mountain Kaolin WM88-224; 5) Ekaton Grassdale Kaolin GD88-08; 6) Ekaton Grassdale Kaolin GD88-04; 7) Ekaton Wood Mountain Kaolin WM88-242; 8) SEM Big Muddy #2; 9) SEM Frenchman CR#1; 10) Goodwater Clay P5 Hole 8; 11) Eastend Stoneware Clay P7 Hole 22; 12) Rockglen Stoneware Clay P53 Hole 4; 13) Estevan Brick Clay P4 Hole 4; 14) Willows Ball Clay P1 Hole 2; 15) Readlyn Ball and Fire Clay P10 Hole 2; 16) Willows Ball and Fire Clay P8 Hole 3; 17) Willowbunch Ball Clay P19 Hole 3; 18) Pickthall Bentonite P16 Hole 3; 19) Duncairn Pumicite P25 Hole 7; and 20) St. Victor Bentonite-Pumicite P17 Hole 3. (Base map modified from Google Earth image, accessed June 26, 2019.)

3. Geological Setting of the Upper Cretaceous and Tertiary Strata in the Study Area

The following description is summarized from Saskatchewan Geological Survey (2003), and Macdonald and Slimmon (1999). Marine shales and siltstones of the Upper Cretaceous Bearpaw Formation form the base of the subcropping rock units in the project area (Figure 2). This is followed by a dominantly continental sequence, initially comprising fine-grained volcanic lithic sandstone with shale interbeds of the <30 m thick Eastend Formation. This is succeeded by the up to 22 m thick Whitemud Formation, comprising white to light grey, kaolinic claystone, siltstone and fine sandstone, with minor mudstone and lignite interbeds. Lastly, this is overlain in the west by ~7.5 m of bentonitic shale of the Battle Formation. Elsewhere the Whitemud Fm. is overlain disconformably by 60 m of fluvial-lacustrine, medium-grained sandstone, bentonitic shale and muddy siltstone of the Frenchman Formation. The Paleocene Ravenscrag Formation lies conformably above the Frenchman Formation, and consists of 160 to 240 m of fluvial-lacustrine sandy claystone, feldspathic sandstone, siltstone and shale, with coal horizons, and kaolinite beds of the Willow Bunch Member.

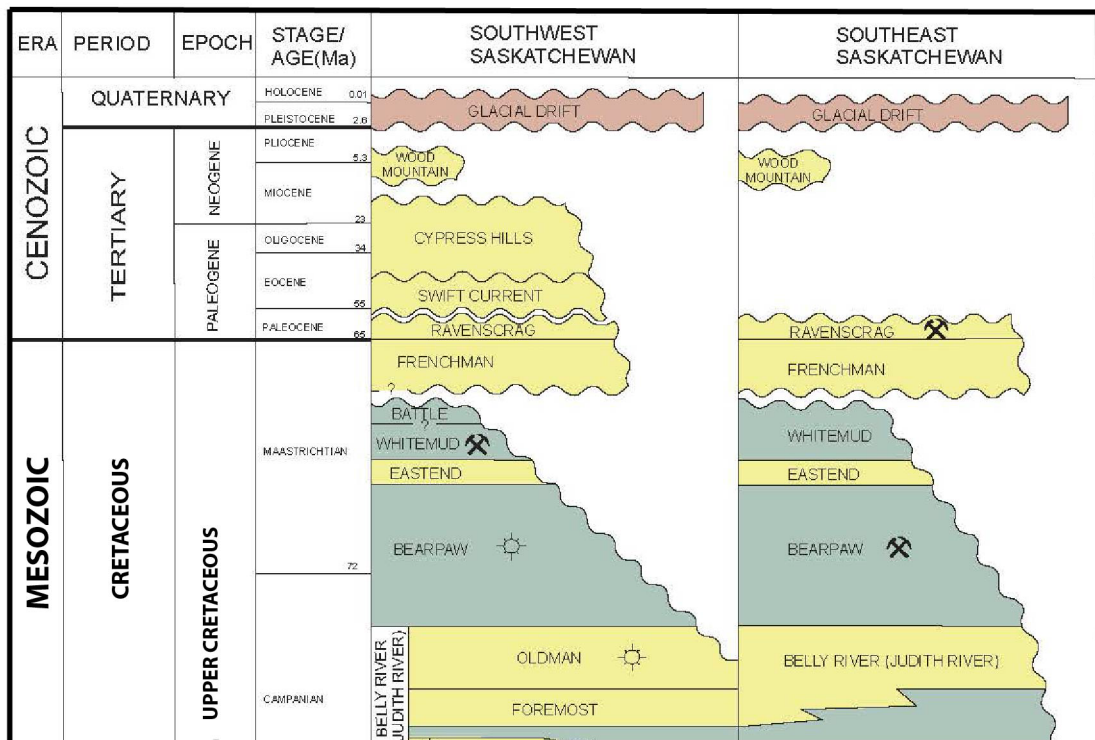


Figure 2 – Stratigraphic columns for southern Saskatchewan, illustrating the Upper Cretaceous and Tertiary formations referenced in this paper, with symbols for mining (crossed hammers) and natural gas production (eight-point framed circle) noted. (Derived from the Stratigraphic Correlation Chart, Saskatchewan Ministry of Energy and Resources website, <https://publications.saskatchewan.ca/#/products/81737>).

The study area lies at the northwest margin of the Williston Basin. Throughout the late Cretaceous and early Tertiary periods, increasing amounts of continental clastic sediments were derived from the west, related to tectonic activity and uplift from the Laramide Orogen (ca. 80 to 50 Ma). This occurred as a series of tectonically induced pulses, following the regression of the Bearpaw Sea (Pruett and Murray, 1991). Sediments that formed the Eastend, Whitemud, Frenchman and Ravenscrag formations were deposited in basins resulting from the salt dissolution of Devonian Prairie Evaporite sequences and related subsidence by eastward-directed subsurface fluid flow from the Laramide orogenic activity (Broughton, 1977). Current Whitemud Fm. localities, from west to east, include Eastend – Ravenscrag, Fir Mountain, Wood Mountain – Gollier Creek, Willows – Readlyn, and Claybank. These localities are

associated with the Cypress, Wood Mountain and Willow Bunch basins, which are believed to be erosional remnants of a once much more widely distributed formation (Broughton, 1977).

a) Whitemud Formation

The Whitemud Formation was the principal unit sampled in this study. It is Upper Cretaceous (Maastrichtian) in age and, according to Lerbekmo (1987), has a magnetostratigraphic age of 68 Ma, based on samples taken in the Cypress Hills region. As described by Pruett and Murray (1991), the Whitemud Fm. can be divided into lower, middle and upper units. The lower unit is the main focus of economic interest and consists of massive or weakly bedded to cross-bedded, white, to light- to medium-grey, fine- to coarse-grained, kaolinitic quartzose sandstone that can locally contain up to 60% kaolin. The middle unit contains thin (≤ 0.3 m) lignite seams interbedded with brown, grey and green claystones, siltstones and shales. The upper unit contains interbedded variously hued grey kaolinitic siltstones and claystones. The Whitemud Fm. is interpreted to have been deposited as alluvium in low-gradient river channels and streams, and in shallow lakes and ponds on a low-lying coastal plain. Broughton (1977) suggested that the Whitemud sediments were deposited in the upper deltaic plains of delta complexes prograding into the depositional basins.

Guillet and Guliov (2014) provide a comprehensive description of Whitemud Fm. geology, distribution and uses. The Whitemud clays have been commercially extracted as white-burning ball, high-duty refractory, plastic stoneware, and structural clays. This includes, notably, a once thriving brick manufacturing industry, comprising numerous companies, over a period of nearly a century (Worcester, 1950; Guillet and Guliov, 2014). Whitemud kaolin in the Wood Mountain area was extensively evaluated as a filler for high-quality paper in the mid to late 1980s (Master, 1987). There was a brief period in the late 2000s during which value-added metakaolin was produced at the Gollier Creek mine. Metakaolin is used as an additive in cement production to increase concrete strength and durability. Pruett and Pickering (2006) provide a thorough general summary of the world kaolin industry, including geology and commercial aspects.

Byers (1969) conducted a detailed petrographic study of the upper Eastend and Whitemud formations in southern Saskatchewan. The Whitemud sediments were found to have metamorphic lithic grains as the major constituent, with lesser kaolinitic clay and vermicular kaolin, minor clear quartz, chert, muscovite, volcanic lithic grains and feldspar, and trace magnetite, ilmenite (weathered to leucoxene), zircon, tourmaline and garnet. Resistant grains are angular to subangular. Byers (1969) interpreted both the upper Eastend and Whitemud formations as being derived from Upper Cretaceous volcanic rocks, Precambrian and Paleozoic metamorphic rocks, and Paleozoic carbonate rocks from western Montana. The upper Eastend sediments were described as volcanic lithic sandstones and interpreted to largely represent mechanical weathering of extensive, freshly extruded volcanic rocks. Byers (1969) suggested the Elkhorn Mountain volcanic rocks (EMV) as a potential source. The EMV consist of a ca. 84 Ma, extensive, thick succession of mainly dacitic to rhyolitic ignimbrites that erupted over a period of about one million years (Horton, 2016). The ca. 80 Ma Boulder granite batholith intruded up through the EMV. The Whitemud kaolinitic sediments were interpreted to have formed from relatively slow chemical weathering and leaching of the altered volcanic tuffs and flows at the source, converting the feldspars to kaolin, followed by transport. Golovneva (2000) interpreted the climate during the Maastrichtian age as warm, humid and temperate with high rainfall, which would have been conducive to chemical weathering.

In their petrographic study of the Frenchman and Ravenscrag formations in Saskatchewan, Misko and Hendry (1979) also found a significant component of volcanic clasts, with up to 30% in the sand fraction of the Frenchman Formation and lesser content in the Ravenscrag. The bulk of the material was also thought to have been derived from western Montana, with contributions from both the Elkhorn Mountain volcanic and Adel Mountain volcanic rocks (AMV). The AMV extend over 900 km² and consist of about 1000 m of potassium-rich basalt and andesite flows, breccias and volcanoclastic sediments, which have been extensively intruded by coeval mafic plugs, sills, irregular bodies and thousands of dykes, all dated at 76 to 73 Ma (Harlan *et al.*, 2005).

Pruett and Murray (1991) carried out a detailed mineralogical and textural study of the Whitemud sediments. Kaolinite content was found to increase upwards through this formation from the Eastend Fm. The abundance of smectite clays, micas and feldspars decreases from the Eastend Fm. into the Whitemud Fm. Vermiform kaolinite

crystals are in close spatial association with feldspar and mica grains. Etched quartz and feldspar grains, and gibbsite ($\text{Al}(\text{OH})_3$) near the top of the Whitemud Fm. indicate the *in situ* presence of a strong chemical leaching environment. The conclusion from mineral distributions, root remains, pedologic mottles, clay grain coatings and soil horizons was that the Whitemud sediments were modified by intense weathering in a paleosol environment.

Master (1987) also concluded that the Whitemud kaolin developed *in situ* from the alteration of feldspar. Although unaltered feldspar is a relatively minor component, the original feldspar content was determined to be up to 30 to 40%, based on the abundance of feldspar pseudomorphs. Due to the fragility of the pseudomorphs, the conclusion was that they would not have survived any significant transport and must have formed in place. Up to 20% unidentified clay-fill material was recognized and Master (1987) stated that, if this is kaolin, it could have originated at the source area.

Guillet and Guliov (2014) support the interpretation that the Whitemud clays were formed *in situ* by chemical weathering of feldspar at the site of deposition. No evidence was presented other than a statement that this is the typical interpreted setting of large secondary kaolin occurrences on a world scale.

b) Willow Bunch Member of the Ravenscrag Formation

Of secondary importance in the sampling program was the Willow Bunch Member (WBM) of the Ravenscrag Formation. As described by Guillet and Guliov (2014), the Willow Bunch Member typically consists of 3 to 6 m of brownish grey- to pale grey-weathering, sandy, kaolinized mudstone that is carbonaceous and iron stained, and contains several thin beds of carbonaceous shale. Due to the generally lower quality and abundance of kaolin in comparison to the Whitemud Formation, these clays have not been well explored on a regional basis. Significant deposits, however, are known to occur in the Harptree and Willow Bunch areas and in the Rockglen and Strathallen areas south of Wood Mountain.

4. Project Methodology

The main purpose of this reconnaissance geochemical project was to gain a broad perspective on the minor element contents, and specifically the REE contents, primarily of Whitemud Fm. kaolin and secondarily Ravenscrag Fm. kaolin deposits, from selected widely spaced drillholes in the far southern part of the province. The drillhole locations are shown on Figure 1 and the drillhole and sample descriptions are summarized in Table 1.

The cores for the 20 drillholes that were sampled are available for viewing and study in the Saskatchewan Subsurface Geological Laboratory in Regina. Of the 29 samples taken, 20 were of Whitemud Fm. kaolinitic sedimentary rock from 12 drillholes, and 6 were of Ravenscrag WBM kaolinitic sedimentary rock from 5 drillholes. Three samples were also taken of pumicite (volcanic ash)/bentonite, one each from three drillholes, two of which are from the Ravenscrag Fm.; the other is from the Upper Eocene to Lower Miocene Cypress Hills Fm.

Many of the sampled drillholes are from an extensive drill program carried out by Ekaton Industries Inc. mainly in the Wood Mountain and Gollier Creek area from 1987 to 1988. Drill logs for these holes are available in the Saskatchewan assessment file database (Ekaton Industries Inc., 1992), and the ongoing program at that time was summarized by Master (1987). Two of the holes were drilled by the Saskatchewan Ministry of Energy and Mines (SEM) in 1991 in the Big Muddy and Frenchman River areas. The SEM also drilled the Rockglen hole in 1974. Documentation for these drillholes could not be found. These more recent holes have 7 cm diameter core and recovery was generally good (Figure 3). The remaining holes were drilled by the Industrial Minerals Branch of the Saskatchewan Geological Survey from 1948 to 1953 (Crawford and Carlson, 1953; Carlson and Babey, 1955). A small portable rig was used to drill shallow holes with 3.5 cm diameter core recovered (Figure 4). Core recovery was often poor due to the wet semiconsolidated nature of the material, and a significant quantity of core is missing.

Table 1 – Summary of drillholes sampled and sample descriptions.

Drillhole Name (Location No. on Figure 1)	NTS_50 ¹	UTM ² 83_13_E	UTM ² 83_13_N	Year Drilled	Sample Number	Sample Interval (m)	Sample Description
Ekaton Gollier Creek Kaolin WM87-175 ^A (1)	72G08	409637	5470717	1987	CL-1-2019	10.98 to 21.65 ³	Whitemud Fm. medium (m)-grey, thin-bedded, impure kaolin clay with silty sections.
Ekaton Project 12 Kaolin WM87-200 ^A (2)	72G08	391335	5470607	1987	CL-2-2019	3.05 to 6.71 ³	Whitemud Fm. light (l)-grey, pure kaolin matrix with fine sand-sized quartz, feldspar and lithic grains. Two consecutive samples.
Ekaton Project 12 Kaolin WM87-200 ^A (2)	72G08	391355	5470607	1987	CL-3-2019	6.71 to 10.18 ³	
Ekaton Wood Mountain Kaolin WM88-237 ^A (3)	72G08	413642	5467399	1988	CL-4-2019	22.26 to 23.78 ³	Ravenscrag Fm. (Willow Bunch Member) l-m grey, impure kaolin matrix with fine sand-sized quartz, feldspar and lithic grains. Two consecutive samples.
Ekaton Wood Mountain Kaolin WM88-237 ^A (3)	72G08	413642	5467399	1988	CL-5-2019	23.78 to 25.30 ³	
Ekaton Wood Mountain Kaolin WM88-224 ^A (4)	72G08	403591	5473217	1988	CL-6-2019	6.1 to 9.91 ³	Whitemud Fm. l. grey, high purity kaolin matrix with fine sand-sized quartz, feldspar and lithic grains. Two consecutive samples. Lost core section in second sample.
Ekaton Wood Mountain Kaolin WM88-224 ^A (4)	72G08	403591	5473217	1988	CL-7-2019	9.91 to 12.2 13.72 to 15.24 ³	
Ekaton Grassdale Kaolin GD88-08 (5)	72H09	567635	5492888	1988	CL-8-2019	3.05 to 11.28 ³	Whitemud Fm. white-l. grey, pure kaolin matrix with residual fine sand quartz, feldspar, and lithic grains.
Ekaton Grassdale Kaolin GD88-08 (5)	72H09	567635	5492888	1988	CL-9-2019	15.55 to 21.34 ³	Whitemud Fm., similar to previous sample, more impure towards the end of section.
Ekaton Grassdale Kaolin GD88-04 (6)	72H09	561101	5492808	1988	CL-10-2019	3.96 to 5.49 ³	Whitemud Fm. white, relatively pure matrix kaolin with residual fine sand grains; 5.49-12.2 m missing core.
Ekaton Grassdale Kaolin GD88-04 (6)	72H09	561101	5492808	1988	CL-11-2019	12.2 to 16.52 ³	Whitemud Fm. white, relatively pure kaolin matrix with residual fine sand-sized quartz, feldspar and lithic grains.
Ekaton Wood Mountain Kaolin WM88-242 ^A (7)	72G08	401905	5470008	1988	CL-12-2019	12.8 to 16.31 ³	Whitemud Fm. white-l. grey kaolin matrix with residual silt – sand quartz, feldspar and lithic grains.
Ekaton Wood Mountain Kaolin WM88-242 ^A (7)	72G08	401905	5470008	1988	CL-13-2019	16.31 to 21.34 ³	Whitemud Fm., similar to previous sample, becomes more impure and unconsolidated towards bottom.
SEM Big Muddy #2 (8)	72H06	490089	5457288	1991	CL-14-2019	31.15 to 33.85	Whitemud Fm. white-l. grey, pure kaolin matrix with residual quartz, feldspar and lithic fine sand grains. Two consecutive samples.
SEM Big Muddy #2 (8)	72H06	490089	5457288	1991	CL-15-2019	33.85 to 36.7	
SEM Frenchman CR#1 (9)	72F07	239553	5481758	1991	CL-16-2019	15.9 to 24.1	Whitemud Fm., 3 consecutive samples from a thick section of white-l. grey, good quality kaolin as matrix with feldspar and minor quartz and lithic fine sand.
SEM Frenchman CR#1 (9)	72F07	239553	5481758	1991	CL-17-2019	24.1 to 31.5	
SEM Frenchman CR#1 (9)	72F07	239553	5481758	1991	CL-18-2019	31.5 to 42.3	
Goodwater Clay P5 Hole 8 ^B (10)	62E05	600731	5470311	1953	CL-19-2019	3.35 to 22.87 ³	Ravenscrag Fm. (WBM) light to dark grey, v. fine-grained clay sample. Local lignite sections excluded.

Drillhole Name (Location No. on Figure 1)	NTS_50 ¹	UTM ² 83_13_E	UTM ² 83_13_N	Year Drilled	Sample Number	Sample Interval (m)	Sample Description
Eastend Stoneware Clay P7 Hole 22 ^B (11)	72F07	221849	5487083	1953	CL-20-2019	8.54 to 17.68 ³	Whitemud Fm. variable l.-m. tan and grey clays, l. grey, fine clay bottom half. 6.1 m sampled in interval.
Rockglen Stoneware Clay P53 Hole 4 (12)	72G01	422429	5451482	1974	CL-21-2019	7.47 to 8.84 ³	Ravenscrag Fm. (WBM) white, pure, massive kaolin.
Estevan Brick Clay P4 Hole 4 ^C (13)	62E02	649203	5443364	1948	CL-22-2019	1.83 to 8.99 ³	Ravenscrag Fm. (WBM) l-m grey clay mudstone in upper part gradational to fine sandstone with clay matrix.
Willows Ball Clay P1 Hole 2 ^C (14)	72H12	440742	5497236	1948	CL-23-2019	11.43 to 13.26 ³	Whitemud Fm. l. grey, fine-grained sandstone with kaolin matrix and quartz, feldspar and lithic grains.
Readlyn Ball and Fire Clay P10 Hole 2 ^C (15)	72H12	446802	5491898	1949	CL-24-2019	6.4 to 10.06 ³	Whitemud Fm. l. grey to white, relatively pure kaolin with some fine sand sections.
Willows Ball and Fire Clay P8 Hole 3 ^C (16)	72H12	446838	5495136	1948	CL-25-2019	4.57 to 16.77 ³	Whitemud Fm. l. grey to white, good quality kaolin becoming sandy near to the end.
Willowbunch Ball Clay P19 Hole 3 ^C (17)	72H05	452793	5478917	1951	CL-26-2019	9.6 to 18.81 ³	Ravenscrag Fm. (WBM) l. grey, relatively uniform clay with minor lignite sections excluded from sampling.
Pickthall Bentonite P16 Hole 3 ^C (18)	72H05	427973	5476443	1950	CL-27-2019	3.05 to 10.06 ³	Ravenscrag Fm. with ~ 3 m of bentonite in sampled sections within this interval.
Duncairn Pumicite P25 Hole 7 ^C (19)	72K01	281956	5554881	1952	CL-28-2019	6.71 to 9.6 ³	Cypress Hills Fm. brownish bentonite often intermixed with white pumicite.
St. Victor Bentonite-Pumicite P17 Hole 3 ^C (20)	72H05	437744	5477848	1950	CL-29-2019	Sections of 3.81 to 17.68 ³	Ravenscrag Fm. l. grey to white, fine-grained pumicite with minor bentonite, more impure downhole.

¹ National Topographic Survey 1:50 000-scale map sheet

² UTM coordinates in NAD83, Zone 13, E = easting, N = northing; converted from original Lsd-Section-Township-Range-Meridian locations (Saskatchewan Mining and Petroleum GeoAtlas)

³ Converted from feet

References: ^A Ekaton Industries Inc. (1992), ^B Carlson and Babey (1955), ^C Crawford and Carlson (1953).



Figure 3 – Drillhole Ekaton Project 12 Kaolin WM87-200, large diameter (7 cm) core of an Upper Cretaceous Whitemud Formation kaolin interval. Note the sample intervals for this project, marked by yellow, labelled paper. This core was previously split and sampled during the original drilling program in 1987 by Ekaton Industries Inc.

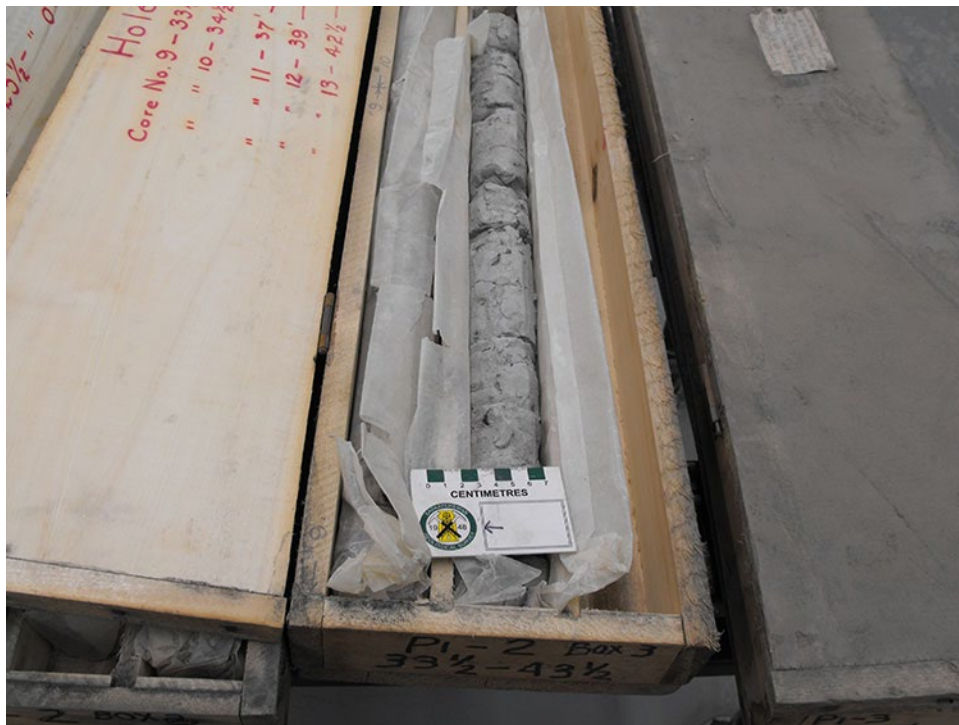


Figure 4 – Drillhole Willows Ball Clay P1 Hole 2, small diameter (3.5 cm) core of a Whitemud Formation kaolin interval drilled in 1948 by the Saskatchewan Geological Survey. Note the wax paper used in these holes to contain the original wet, semiconsolidated core.

As the known ion-adsorption REE clay operations are large-tonnage, bulk-mining operations, the purpose of the sampling program was to estimate the overall contents of the kaolin-bearing core sections in the drillholes. Selective grab samples of chips were taken, where available, throughout each section and placed in a clear plastic sample bag. Larger chips were split with a geological hammer and a portion was placed in the sample bags while the remainder was returned to the core box. In some holes with particularly competent core, some chips had to be removed using a hammer. Some of the more recent holes with thick kaolin intervals required two, and in one hole three, samples to be taken. The intervals were marked at the ends with yellow, labeled paper. For all of the samples, the sampled intervals were recorded as well as a concise description of the core. Photographs were taken of the sample intervals. The sample number was recorded in black marker on the plastic sample bag and on a piece of orange flagging tape that was put in the bag. The bags were sealed with fibre tape and placed in plastic pails that were labeled and delivered to the Saskatchewan Research Council's Geoanalytical Laboratory in Saskatoon for multi-element analysis.

5. Discussion of Analytical Results

The multi-element analyses were conducted using inductively coupled plasma-optical emission spectroscopy (ICP-OES), with separate partial and total digestion sample runs. The complete laboratory procedures, including sample preparation, detection limits and quality control, as well as analytical results, are described in Data File 47, which is referenced at the beginning of this paper.

Arithmetic means and ranges for the major elements and selected minor elements determined by total digestion ICP-OES are presented, respectively, in tables 2 and 3. (See Data File 47 for results of the partial digestion analyses.)

Table 2 – Summary of the arithmetic means and ranges for the major oxides in the 29 samples in this study, determined by ICP-OES total digestion analysis.

Oxide	Al ₂ O ₃	CaO	Fe ₂ O ₃	K ₂ O	MgO	MnO	Na ₂ O	P ₂ O ₅	TiO ₂
Arithmetic Mean (wt.%)	16.5	1.06	2.74	1.78	1.0	0.023	0.84	0.06	0.66
Range (wt.%)	10.7 to 27.2	0.05 to 9.93	0.48 to 5.96	0.34 to 2.92	0.23 to 4.06	<0.01 to 0.09	0.05 to 2.80	0.02 to 0.22	0.32 to 0.96

Table 3 – Summary of the arithmetic means and ranges for selected trace and minor elements in the 29 samples in this study, determined by ICP-OES total digestion analysis.

Element	Ba	Ce	Li	S	Sr	V	Y	Zr
Arithmetic Mean (ppm)	613	53	42	1014	114	131	21	121
Range (ppm)	145 to 1260	36 to 136	18 to 136	32 to 6640	21 to 335	56 to 204	10 to 37	64 to 241

The Al₂O₃ content is a potentially commercial product. In this study, the arithmetic mean for this oxide for all samples is 16.5%, with a high of 27.2% in Whitemud Fm. sample CL-24-2019 from Readlyn. The Fe₂O₃, TiO₂ and Zr contents can be mainly attributed, respectively, to contained detrital magnetite and authigenic siderite, ilmenite intergrown with magnetite and partially altered to leucoxene, and zircon as described by Byers (1969). The variable sulfur (S) content is likely attributable to contained authigenic gypsum, which occurs as selenite (Byers, 1969). The total digestion and partial digestion results for this element are almost identical, indicating that the sulfur is held in a highly soluble mineral(s). Ba, Li, Sr and V are all within general background for clays. The mean for V by total digestion (131 ppm) is much higher than that by partial digestion (17.7 ppm), indicating that the vanadium is mainly held in insoluble minerals such as oxides.

A summary of the rare earth element analytical results is presented in Table 4.

Table 4 – Summary of analytical results for the rare earth elements in the 29 samples for this study, determined by ICP-OES total digestion analysis. Abbreviations: TREE - total rare earth elements; LREE - light rare earth elements; HREE - heavy rare earth elements; ppm - parts per million.

Sample Number	TREE+Y,Sc ¹ (ppm)	LREE ² (ppm)	HREE+Y,Sc ³ (ppm)	% HREE+Y,Sc ⁴	HREE+Y,Sc / LREE
CL-1-2019	109.5	66.3	43.2	39	0.65
CL-2-2019	213.9	155.1	58.8	27	0.38
CL-3-2019	169.4	113.6	55.8	33	0.49
CL-4-2019	134.0	95.3	38.7	29	0.41
CL-5-2019	129.0	89.2	39.8	31	0.45
CL-6-2019	147.9	114.3	33.6	23	0.31
CL-7-2019	134.9	93.3	41.6	31	0.45
CL-8-2019	210.8	162.6	48.2	23	0.30
CL-9-2019	136.3	97.3	39.0	29	0.40
CL-10-2019	91.4	63.5	27.9	31	0.44
CL-11-2019	118.0	85.0	33.0	28	0.39
CL-12-2019	169.1	129.4	39.7	23	0.31
CL-13-2019	168.1	125.6	42.5	25	0.34
CL-14-2019	143.8	104.1	39.7	28	0.38
CL-15-2019	236.3	181.4	54.9	23	0.30
CL-16-2019	108.2	77.6	30.6	28	0.39
CL-17-2019	158.2	122.1	36.1	23	0.30
CL-18-2019	168.3	131.1	37.2	22	0.28
CL-19-2019	161.7	117.3	44.4	27	0.38
CL-20-2019	154.6	118.4	36.2	23	0.31
CL-21-2019	365.1	310.2	54.9	15	0.18
CL-22-2019	154.3	112.2	42.1	27	0.38
CL-23-2019	123.9	89.8	34.1	28	0.38
CL-24-2019	115.8	80.8	35.0	30	0.43
CL-25-2019	193.2	143.4	49.8	26	0.35
CL-26-2019	138.1	99.0	39.1	28	0.39
CL-27-2019	164.6	119.3	45.3	28	0.38
CL-28-2019	185.6	124.3	61.3	33	0.49
CL-29-2019	162.0	124.9	37.1	23	0.30

¹ TREE+Y,Sc: total REE+Y,Sc (La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Yb, Y, Sc)

² LREE : light REE (La, Ce, Pr, Nd, Sm, Eu, Gd)

³ HREE+Y,Sc: heavy REE+Y,Sc (Tb, Dy, Ho, Er, Yb, Y, Sc)

⁴ % HREE+Y,Sc: the percentage that the HREE+Y,Sc comprise of the TREE+Y,Sc

Rare earth element contents were consistent in all samples, and are considered background, with one exception. The arithmetic mean for all 29 samples was 160.9 ppm TREE+Y,Sc, with little variation among samples, perhaps indicating similar source areas. TREE+Y,Sc ranged from a low of 91.4 ppm in sample CL-10-2019, to a high of 365.1 ppm in sample CL-21-2019. This latter value is considered modestly anomalous, in comparison to the other samples, and was taken from Ravenscrag WBM kaolin drill core from Rockglen (Figure 5). The other five WBM samples (CL-4, -5, -19, -22, -26-2019) from four drillholes, however, are not anomalous, with an arithmetic mean of 144.5 ppm TREE+Y,Sc. The 20 Whitemud Fm. samples have a very similar arithmetic mean of 153.3 ppm TREE+Y,Sc. The three pumicite/bentonite samples had slightly higher TREE+Y,Sc contents: CL-27-2019 (Ravenscrag Fm.) – 164.6 ppm; CL-28-2019 (Cypress Hills Fm.) – 185.6 ppm; and CL-29-2019 (Ravenscrag Fm.) – 162.0 ppm. These three pumicite/bentonite samples would have formed from primary volcanic ash deposits that had altered *in situ*, therefore their REE contents would also be expected to be primary constituents. As the values are similar to those in the 26 kaolin samples, it can be surmised that the REE contents of those samples likely also reflect primary compositions. In all of these samples the less valuable LREEs are dominant, with a range of 63.5 to 310.2 ppm and a mean of 118.8 ppm. The HREE+Y,Sc values have a tight range, from 27.9 to 61.3 ppm, with a mean of 42.1 ppm. The percentage of HREE+Y,Sc comprising the TREE+Y,Sc in each of the samples ranges from 15 to 39%, with a mean of 27%. Anomalous sample CL-21-2019 had the lowest HREE+Y,Sc component of 15% of TREE+Y,Sc at 54.9 ppm, with the remainder as LREE. The HREE+Y,Sc / LREE ratios (Table 4) range from 0.18 to 0.65, with a mean of 0.38. Sample CL-1-2019 is a geochemical outlier, with the highest relative HREE+Y,Sc content

of 39% of TREE+Y,Sc and a HREE+Y,Sc / LREE ratio of 0.65. Yttrium and scandium comprise the majority of the HREE+Y,Sc content in all of the samples.



Figure 5 – Drillhole Rockglen Stoneware Clay P53 Hole 4 core, showing the anomalous REE-bearing sample CL-21-2019 taken from white, relatively pure, massive kaolin clay in interval 7.47 to 8.84 m of the Ravenscrag Formation Willow Bunch Member.

6. Conclusions

The results of this reconnaissance sampling project do not support the hypothesis that kaolinitic clays of the Upper Cretaceous Whitemud Formation and Paleocene Ravenscrag Formation contain ion-adsorption-type REE mineralization. The results indirectly support the interpretation that the kaolin was formed *in situ* at the site of deposition, and was not transported from a weathered REE-bearing source. The arithmetic mean TREE+Y,Sc for all samples is 160.9 ppm, and values fall within a fairly tight range. The one exception is sample CL-21-2019, which contains 365.1 ppm TREE+Y,Sc and is considered to be modestly anomalous in relation to the overall dataset. The relatively low HREE+Y,Sc / LREE ratios in these samples is a negative economic factor. These modest REE contents are likely primary constituents that reside within the mineral structures of the samples, rather than being adsorbed onto the surfaces of the kaolin, although a separate study would be necessary to determine this.

This project involved the sampling of only 20 widely spaced drillholes that intersected kaolin-rich strata from across southern Saskatchewan. Large areas of known kaolin locations were not sampled in this study. Although unlikely, there could be substantial areas of kaolin in local settings with elevated REE values related to ion-adsorption. Of note is that the anomalous sample CL-21-2019 was taken from a Ravenscrag WBM location at Rockglen, and was one of only five drillholes that sampled the WBM in this study. This may warrant a further investigation of WBM locations, including Rockglen, in the south-central and southeastern areas of the province.

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