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SUMMARY

In this paper, we present new pyrolysis and bulk geochemical results obtained using a cored section of the Husky Little Bear N-09 exploration well, drilled in 2012. A total of 326 samples were collected at 0.5m spacing. This is the highest resolution of lithogeochemical logs for discerning stratigraphic units of black shale in the Canol shale gas zone This information can be found in public documents. Samples were collected from the Hume, Hare Indian and Canol formations. The contact between the Canol and Imperial formations is not encountered in this core. This report is concerned with specific major and trace elements associated with proximal siliciclastic flux, bioproductivity and anoxic paleoconditions of the basin. Rock Eval data from the Canol Formation indicates the presence of high-quality Type II kerogen, indicating a hydrocarbon production potential within the peak oil, and marginally, the condensation zones.

INTRODUCTION

The black shales of the Horn River Group, identified a decade ago to host the largest unconventional hydrocarbon resource in the region (Hamblin, 2006), have recently seen investment into the exploration of the central Mackenzie Plain (Hayes, 2011; Pyle et al., 2014) with over 1.2 million hectares to the south and northwest of Norman Wells covered by exploration licenses (AANDC, 2014). This prospect consists of the thickest (up to 180 m) Canol Formation with median TOC values of 4.6% and the Bluefish Member of the Hare Indian Formation that is thinner (up to 23 m) and has 5.6% median value and maximum 10% TOC (Pyle et al., 2014). The highest reported TOC for the Canol Shale is 16.9% (Kabanov, 2015). The two organic-rich units are divided by a gray to black shale interval of variable thickness with 1.5% TOC (median value), that was recently named the Bell Creek Member (Fig. 1; Pyle et al., 2014). Available data indicate that the Horn River Group contains predominantly type II kerogen (offset towards type III in the Bell Creek Member) which, in the Mackenzie Plain, occurs within and locally beyond the oil window. The thermal maturity gradient generally increases to the west and south (Snowdon et al., 1987; Gal and Pyle, 2012; Pyle et al., 2014).

The Husky Little Bear N-09 well was completed on March 21, 2012 and is the first out of a set of cored wells targeting the Canol shale play in the exploration license areas of the Mackenzie Plain (AANDC, 2014). Before this drilling campaign, the Horn River Group in the subsurface had few and mostly short cores. The lithogeochemical, mineralogical, and organic-matter database for the Horn River Group has expanded dramatically in recent years due to the Mackenzie Plain Petroleum Project of the NWT Geoscience Office (NTGO/NTGS). New data for this unconventional prospect were obtained from adjacent outcrops and drill cuttings (Gal et al., 2009; Gal and Pyle, 2011, 2012; Hayes, 2011; Pyle and Gal, 2012, 2013; Pyle et al., 2011, 2014). The Mountain River Tributary section (MR) has been proposed as a regional reference section for its stratigraphic completeness and short-distance helicopter accessibility from Norman Wells (Pyle et al., 2014). The North American Stratigraphic Code (2005, p.1566) also supports the development of subsurface reference sections for lithostratigraphic units originally defined in outcrops.

New continuously cored sections with up-to-date wirelog characterization offer an opportunity to study fine patterns that cannot be picked with cuttings resolution. These sections will be used to improve the surface-subsurface correlation and the stratigraphic framework (including the reference section) for tight-reservoir studies and academic research (e.g., Hemmesch et al., 2014).

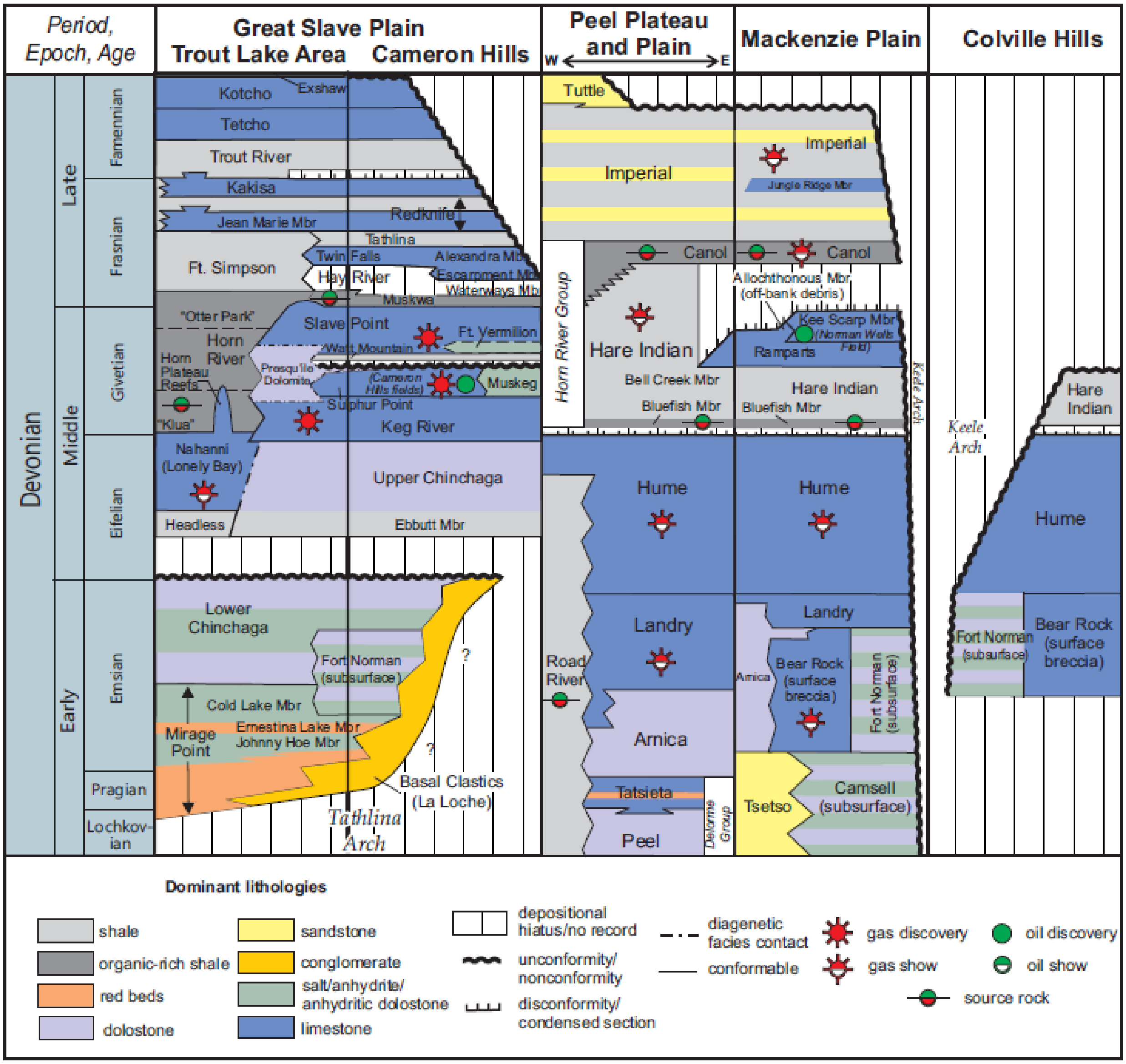

Figure 1: Table of Formations for the Devonian System of Northwest Territories (modified from Rocheleau and Fiess, 2014). Note that northern and southern extremities of the study region (Beaufort-Mackenzie Basin and Liard Basin) are not included. Correlation of stratigraphic units is retained from the source (Rocheleau and Fiess, 2014).

WELL HISTORY

The Husky Little Bear N-09 well (UWI: 300/N-09-6500-12630) is located in the Mackenzie River Corridor, Northwest Territories, approximately 30 km southeast of Norman Wells (Fig. 2). The well was drilled by Husky Energy in January 2012 as an exploration well. It was cored from the middle of the upper Canol Formation down to the upper 7 m of the Hume Formation. The top of the Canol has not been intersected in this core (Fig. 3).

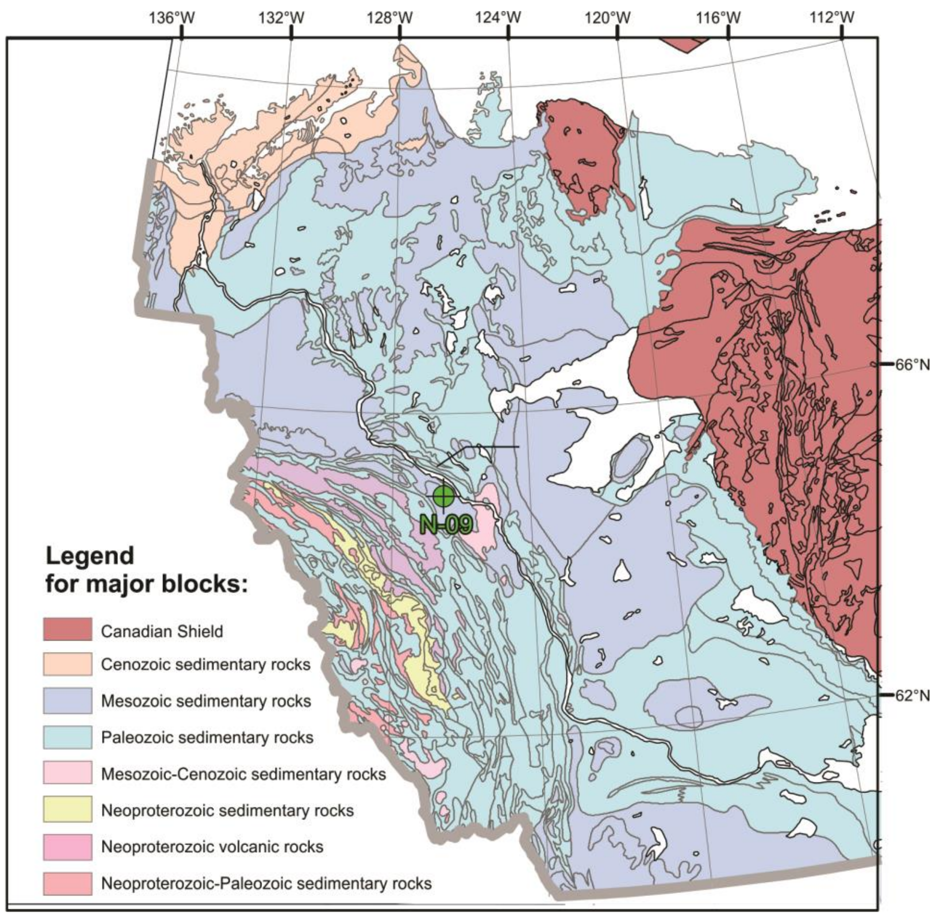

Figure 2: Geological map of northwestern Canada showing location of Husky Little Bear N-09 well (based on Wheeler et al., 1996)

METHODOLOGY

Cores from Devonian strata of the Mackenzie River Corridor were examined and sampled at the NEB Core and Sample Repository at the Geological Survey of Canada in Calgary. A total of 326 samples were collected from core sides and loose chips. The core was sampled approximately every 50 cm, except for the lower carbonates at the bottom of the core which were sampled approximately every meter. Each sample represents an averaged material collected from a stratigraphic interval exceeding 1 cm (typically 2-5 cm) to ensure that none of the collected samples represented a single sedimentary lamina. Drilling mud from the core side was removed prior to collecting each sample.

Bulk-element geochemistry

The samples were analyzed for bulk elemental concentration at Acme Analytical Laboratories in Vancouver, BC using the inductively coupled plasma-mass spectrometry (ICP-MS) instrumentation technique. Two sample preparation techniques were used before running through the ICP-MS in order to analyze for specific elements/compounds.

The first technique was the lithogeochemical whole rock fusion technique (see method LF200 in Appendix 1) which involves mixing a 200 mg sample with a lithium metaborate (LiBO2)/ lithium tetraborate ($Li_2B_4O_7$) flux in a crucible. The crucibles are then placed in a furnace and heated in order to fuse the sample. The bead that develops is cooled, then dissolved in nitric acid and run for ICP-MS in order to analyze the concentrations of rare earth and refractory elements (11 compounds, 33 elements) (see Appendix 1). LOI is also measured by heating and then weighing an aliquot of the sample in order to determine weight loss. This is used as a rough approximation of organic matter (carbon) minus water and light hydrocarbons that were burned off.

The second technique used was geochemical aqua regia digestion (see method AQ200 in Appendix 1), whereby 500 mg of sample was digested in a 1:1:1 solution of hydrochloric acid (HCl), nitric acid (HNO3) and de-ionized water while placed in a heated water bath for one hour. Dilute HCl is then added to the residue in order to top up the sample to the required analysis volume. Samples were split into 0.5g subsamples and run through the ICP-MS instrument to determine the concentration of 14 elements that were not detected in the above technique, including Au and volatile elements, at the ppm level (see Appendix 1).

In addition, total carbon and total sulphur were measured using the Leco instrument (see method TC000 in Appendix 1). In this procedure, the induction flux is added to the crushed sample and ignited in an induction furnace. A carrier gas sweeps up the released carbon and sulphur dioxide which are measured by adsorption in an infrared spectrometric cell. The results represent all forms of carbon and sulphur that are present in the sample. The detection limit for this procedure is 0.02%.

Rock-Eval 6 pyrolysis

The pyrolysis-combustion tests (Appendices 2 and 3) were conducted at the Organic Petrology and Geochemistry Laboratory in GSC (Calgary) using the Rock-Eval 6 instrument. Approximately 1g of the unwashed core sample was crushed to powder form using a mortar and pestle. A 70mg aliquot of the sample was then inserted into a stainless steel crucible and heated in an open pyrolysis system. Initially, the samples are heated at 300°C for 3 minutes to volatilize any free hydrocarbons (HC), which are represented by the S1 peak on the pyrograms. The S1 value (mg HC/g of rock) corresponds to the amount of free and adsorbed hydrocarbons generated naturally over time in the rock (Behar et al., 2001).

The next step in the procedure is to heat the samples from 300°C to 650°C at a rate of 25°C/minute, which yields the S2 peak. The S2 value (mg HC/g of rock) represents the amount of hydrocarbon released due to thermal cracking of kerogen present in the sample. This is the remaining potential of the sample to generate hydrocarbons if conditions had allowed it. Although drilling mud contamination and hydrocarbon migration can affect both the S1 and S2 values (Issler et al., 2012), in the collected core samples, the possibility of drilling mud contamination is negligible due to pre-sampling surface cleaning and the extremely low permeability of shales precluding mud cake formation.

The S3 peak is a measure of the total amount of CO_2 (mg CO_2/g of initial rock) generated over the entire pyrolysis measurement. The S3 curve accounts for the CO_2 measured during the first stage (0-300^{o}C) and second stage (from 300^{o}C to 400^{o}C), which corresponds to CO_2 generated from organic matter. The S3' curve accounts for the CO_2 generated between 400°C and 650°C, which corresponds to mineral decomposition. The S3 peak is the combination of S3 and S3' (Behar et al., 2001).

The final step is the oxidation of the samples, which measures the total amount of organic carbon generated during this stage. Here, the samples are heated from 300°C to 850°C and the CO and CO_2 are detected by IR cells, producing the S4 curve which measures the residual organic carbon (RC). The S5 curve corresponds to the mineral carbon from CO and CO_2 generated during mineral oxidation (Behar et al., 2001), mainly calcination of carbonate salts.

The sum of the pyrolysable organic carbon (PC) and residual organic carbon (RC) is the total organic carbon (TOC; wt%) in the sample. Tmax is measured at the maximum of the S2 peak and indicates the maturity of the samples, which is dependent on the kerogen type (see Tissot et al., 1987). Other calculated parameters include: HI (S2*100/TOC), OI (S3*100/TOC) and PI (S1/S2+S3). These parameters aid in identifying the kerogen type (I-IV) and whether the organic matter in the sample is oil vs gas prone. All pyrograms can be found in Appendix 3.

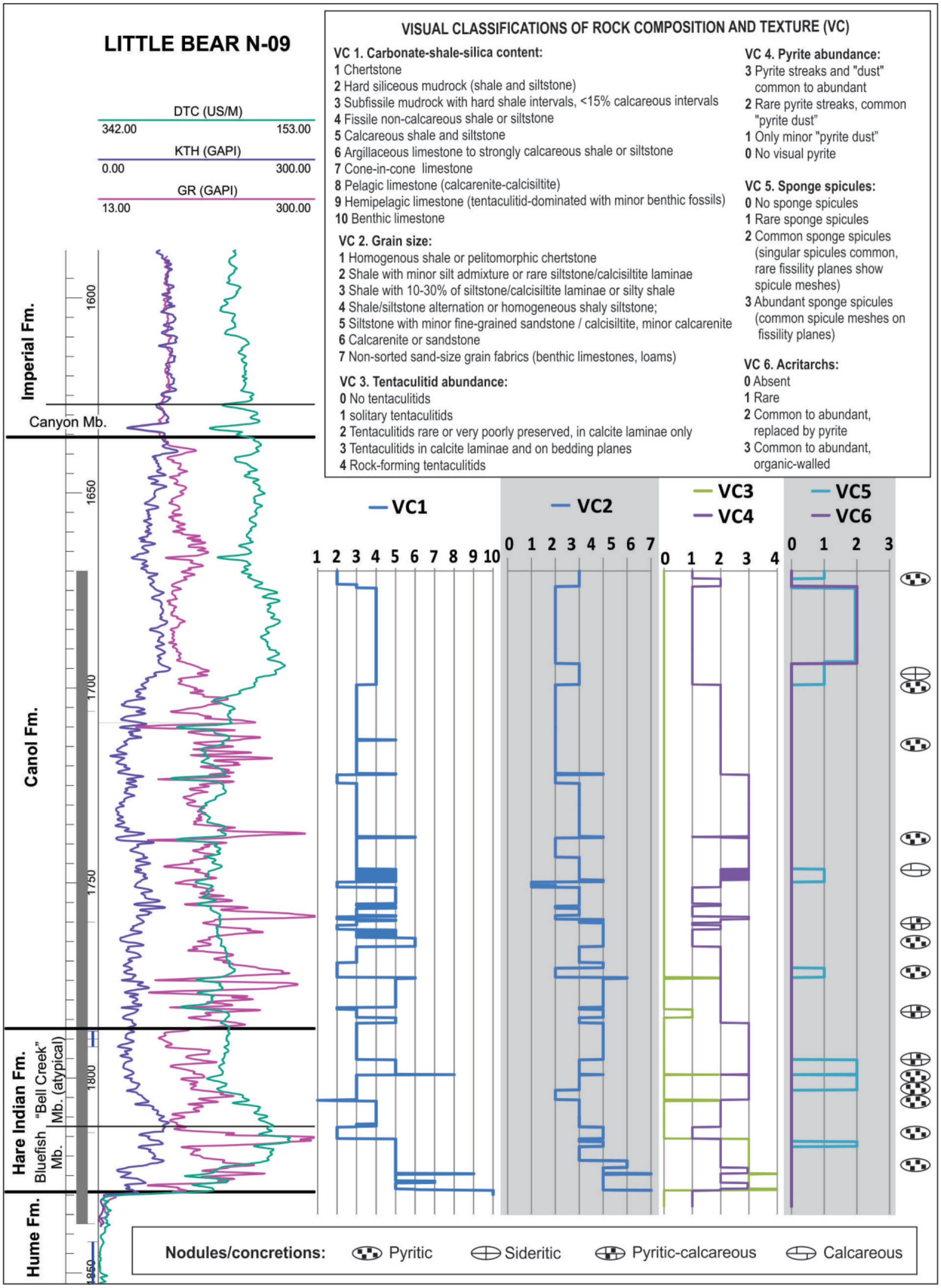

Figure 3: Litholog of the Horn River Group section in Little Bear N-09 well based on lithofacies observations in Kabanov (2015). Spectral GR and sonic logs are included.

RESULTS

Lithogeochemical proxies

Analytical data reported in Appendices 1-3 provide the new resolution for the chemostratigraphy of the Horn River Group (Figs. 4-6) augmented by similarly high-resolution pyrolysis parameters (Fig. 7). The proxies shown on Figures 4-6 include rock-forming elements (here expressed as SiO_2, CaO and Al_2O_3), degree of pyritization (Fe and S), siliciclastic vs. pelagic silica source (SiO_2/Zr), Enrichment Factor for phosphorus (EFP) as a proxy to higher-metazoan (consumer) productivity, Ba (primary bioproductivity vs. early diagenesis), selected proxies for siliciclastic input (here K_2O, TiO_2, and Th/U), redox and anoxia trace-element proxies such as U, V, Mo, Mo/TOC, Ni/Co, and the enrichment factors for Mo and V (EFMo and EFV, correspondingly, shown on logarithmic scale). Most of these proxies are widely used in black shale sedimentology, stratigraphy, and hydrocarbon potential characterization (Calvert and Pedersen, 1993; Tribovillard et al., 2006; Algeo and Rowe, 2012) and have been applied to the studied black shale succession (Pyle et al., 2014).

The enrichment factor for phosphorus (EFP) is defined here as PEF=P_2O_5(X)/ P_2O_5(NASC), where P_2O_5(X) is the phosphate content in a sample, and P_2O_5(NASC) is the average P_2O_5 for North American non-metamorphosed shales referred to as North American Shale Composite (NASC) estimated at 0.13% (Gromet et al., 1984). Phosphorus is vigorously involved in the biogeochemical cycle and precipitates mostly as authigenic and skeletal Ca-phosphates. In phosphorus-lean marine settings, fragments of fish, conodonts, and inarticulate brachiopods are often the only detectable mineral forms of P.

Enrichment factors for trace metals (EFMo and EFV in this work) are defined as EF(element X)=$(X/Al_{sample})/(X/Al_{average\ shale})$ (Tribovillard et al., 2006). If EFX is greater than 1, then element X shows enrichment, and if it is less than 1, then it is considered depleted. As an average value, Tribovillard et al. (2006) recommend the “average shale” concentrations of Wedepohl (1971, 1991), which is followed in (Pyle et al., 2014) and herewith. The average shale concentration of molybdenum is accepted as 1.3 ppm, and vanadium as 130 ppm (Wedepohl, 1991; Tribovillard et al., 2006).

Barium in the form of barite ($BaSO_4$) is known to associate closely with planctogenic organic matter and therefore is frequently used to reconstruct marine primary productivity. However, in permanently anoxic settings barite becomes involved in methanogenic sulphate reduction to be released into aquatic cycle or migrate inside sediment to form concretions (Henkel et al., 2012).

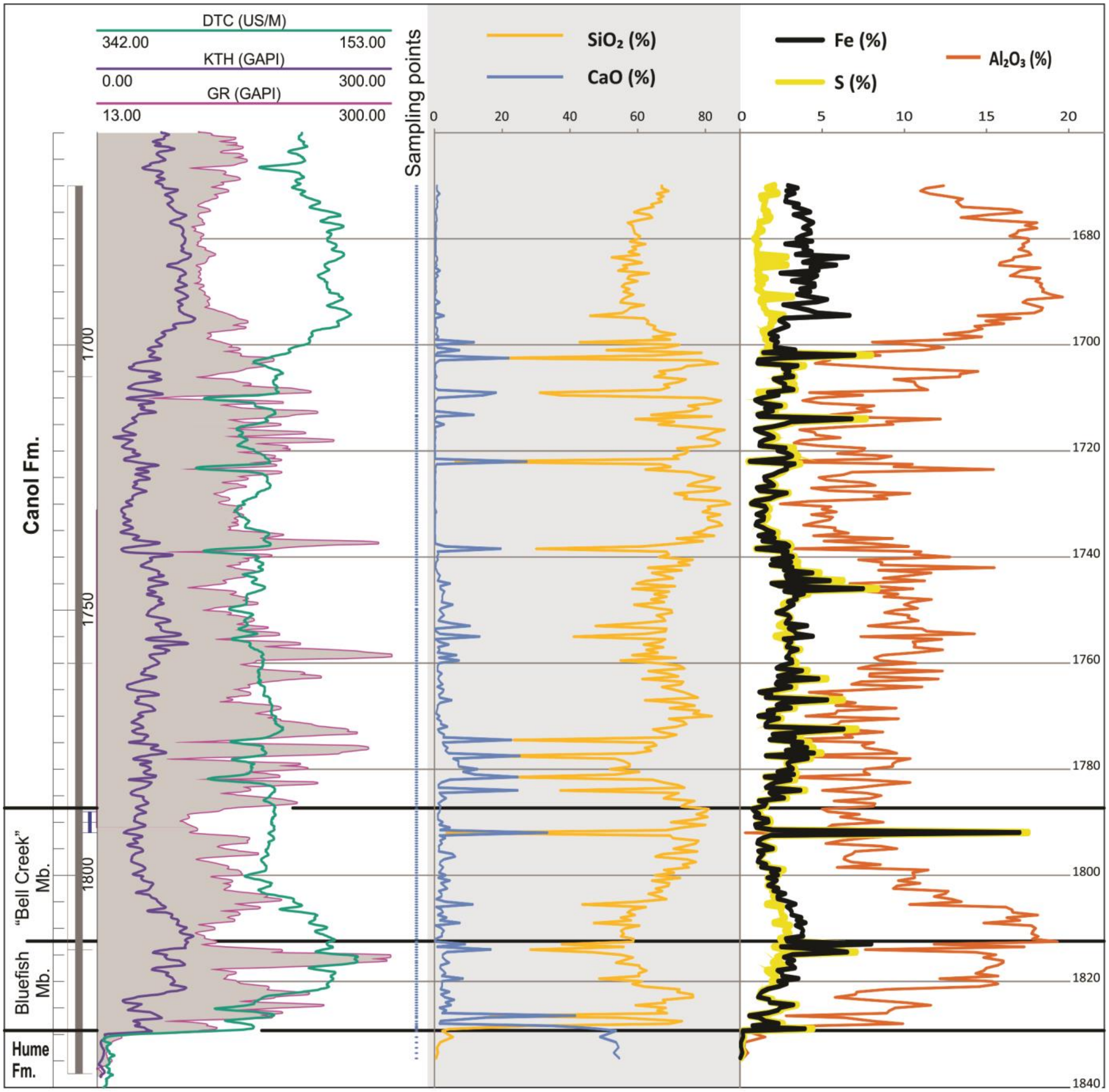

Figure 4: Lithogeochemical logs for the cored section of Husky Little Bear N-09 well compared against geophysical logs: main rock-forming elements (Si, Ca, and Al) in oxide notation, Fe and S as proxies for pyrite abundance. Sulphur refers to Total S in the LECO combustion method.

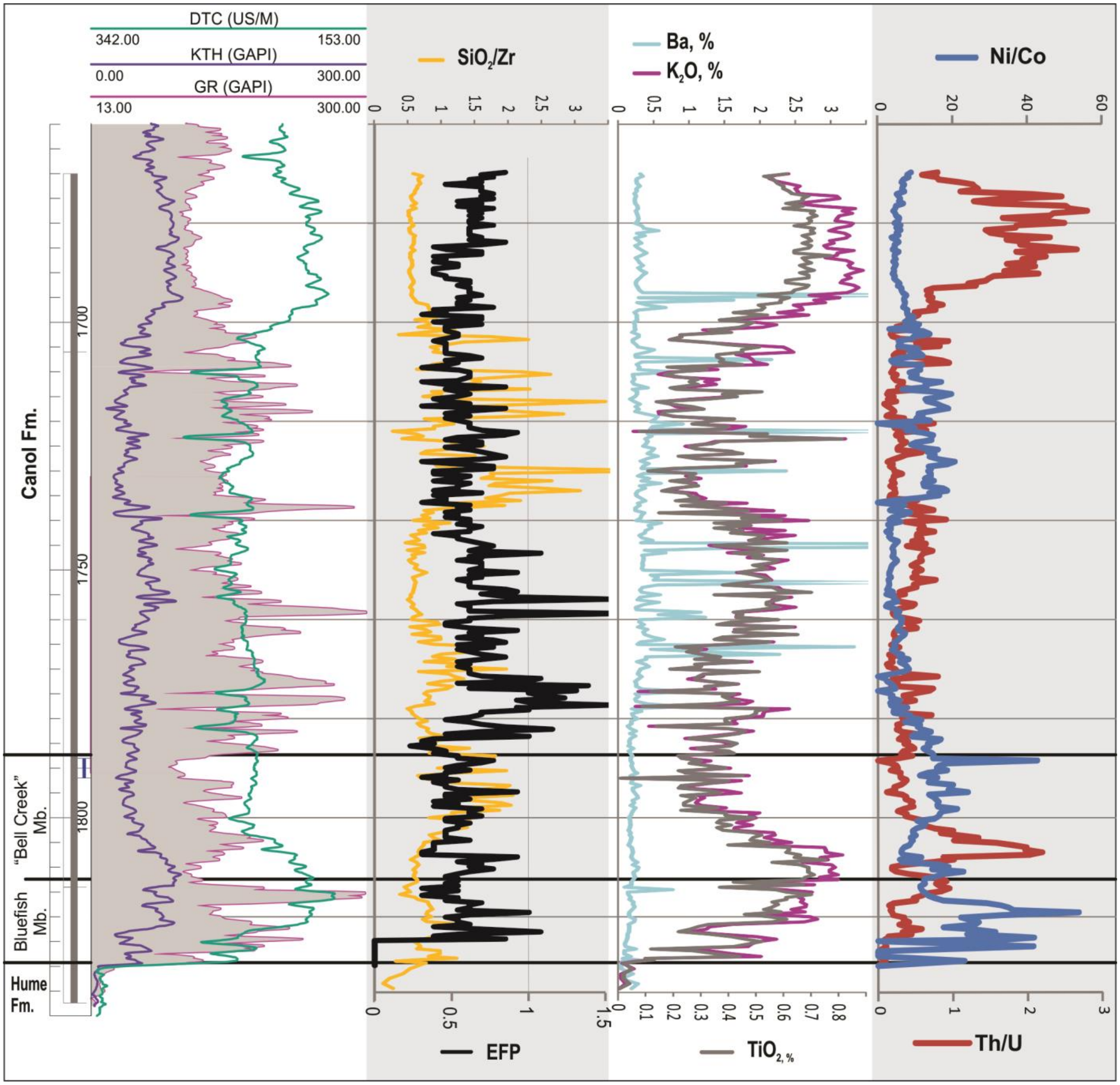

Figure 5: Lithogeochemical logs for the cored section of Little Bear N-09 well compared against geophysical logs. Depositional environment proxies: SiO_2/Zr (siliciclastic vs. basinal biogenic silica, SiO_2 in % and Zr in ppm), Enrichment Factor for phosphorus (EFP), Ba (primary bioproductivity vs. early diagenesis), K_2O, TiO_2, and Th/U (siliciclastic input), and Ni/Co (redox proxy)

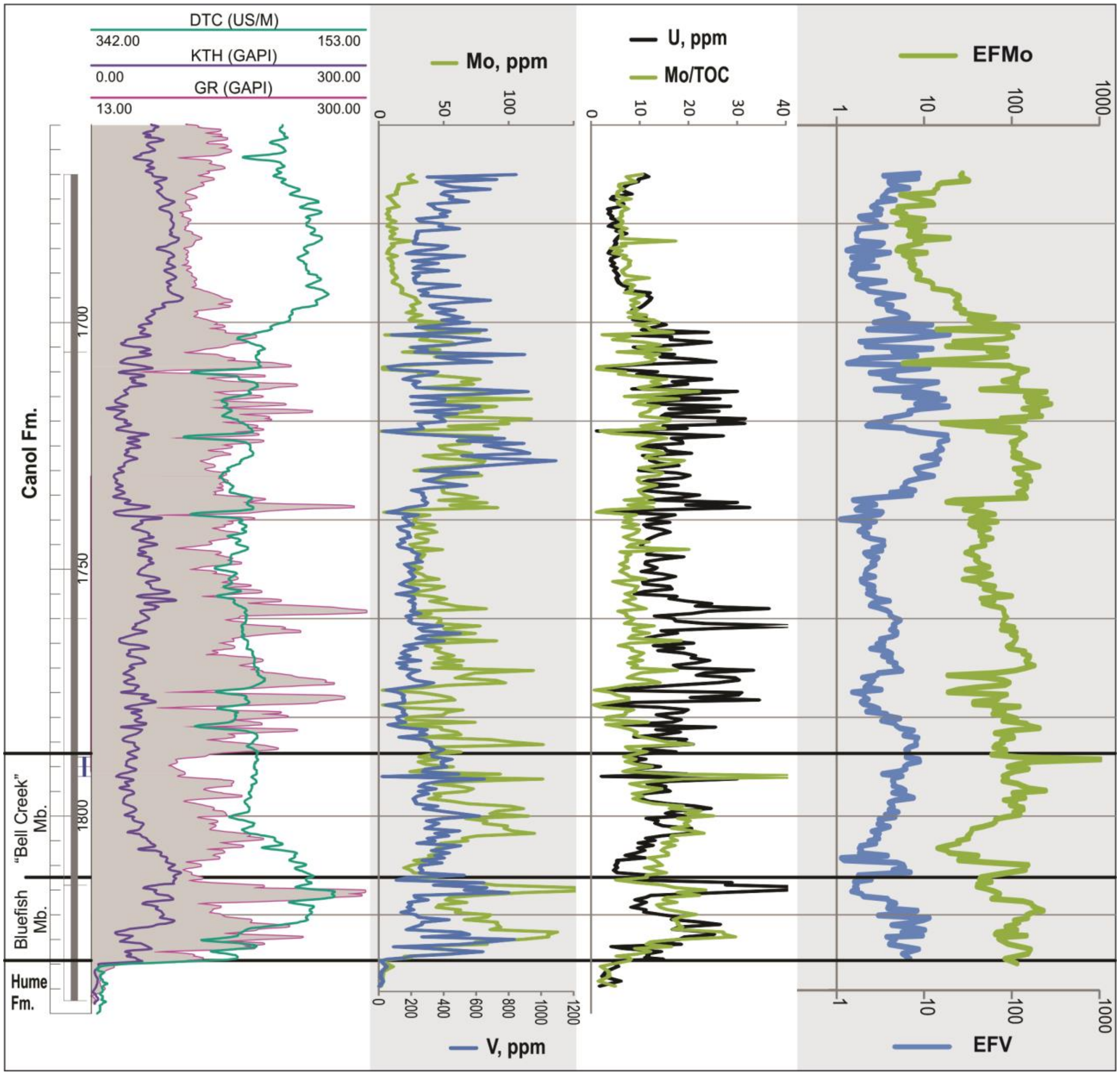

Figure 6: Lithogeochemical logs for the cored section of Little Bear N-09 well. Seafloor anoxia proxies: U, V, Mo, Mo/TOC, and the Enrichment Factors for Mo and V (EFMo and EFV on logarithmic scale).

Chemostratigraphic results

In the Horn River Group of the study area, the basinal-type (off-bank) sections refer to sections composed completely of black shales with the Canol Formation reaching its maximum thickness and no gray bioturbated shales of the upper Hare Indian Formation or carbonate banks of the Ramparts Formation developed. The Carcajou River outcrop section #2 has received detailed multiproxy characteristics and been proposed as the reference basinal section for the Horn River Group (Pyle et al., 2011, 2014).

In Little Bear N-09, the basal high-gamma, low-thorium and TOC-rich unit at 1828.9-1811.8 m is correlated to the Bluefish Member. This unit is also distinct by preservation of mass styliolinids (pelagic calcareous fossils) and the marker cone-in-cone limestone bed at 1826.9-1826.45 m (Fig. 3; Kabanov, 2015). The proportion of terrigenous material in the Bluefish Member is characteristically high as seen from the peaks in Al_2O_3 (Fig. 4), K_2O, and TiO_2 (Fig. 5). The Bell Creek Member in basinal facies is poorly differentiated from the Bluefish and Canol shales and is picked at 1826.45-1787.4 m by characteristic log section, slightly depressed total GR and TOC (Figs. 3 and 6).

The interval equivalent to Basal and Middle Canol units of outcrops (Pyle et al., 2014) is found on 1787.4-1700.0 m. Based on GR, EFV and EFMo signatures, the interval equivalent to the Middle Canol in Carcajou River section 2 (Pyle et al., 2014) is recognized at 1740-1700 m MD (Fig. 6). Iron in the Bell Creek Member and Basal-Middle Canol interval is almost entirely bound in pyrite as seen from the perfect match of S and Fe values (Fig. 4). The "basal Canol" interval at 1787.4-1740.0 m is weakly calcareous with the distinct calcisiltite-rich interval at 1782.0-1774.2 m MD (Figs. 3 and 4). The Middle Canol interval is dominated by non-calcareous siliceous shale punctuated by discrete calcite-rich beds. The median values of CaO are 1.7% for the Hare Indian, 1.3% for the Basal Canol, 0.17% for the Middle Canol, and 0.34% for the available incomplete section of the Upper Canol.

The interval 1670.0-1700.0 m in Little Bear N-09 is correlated to the "Upper Canol" unit of the reference basinal section (Carcajou River 2; Pyle et al., 2014) based on SGR, Ni/Co, Th/U, and siliciclastic proxies (Figs. 4-6). In studied core this interval is composed of the "Imperial-type" fissile shale (1674.0-1699.2 m) grading at the top (1674.0-1670.0m) to harder, more siliceous and pyritic "Canol-type" shale (Kabanov, 2015). The "Imperial-type" fissile shale in the Upper Canol is also manifested by decoupling Fe and S, indicating that iron is partly bound in detrital terrigenous minerals and probably some siderite. Significant amounts of siderite (5.1% in average) have been indicated for the Upper Canol interval of Bear Rock O-20 well (Gal and Pyle, 2012). Acquisition of new data from new cores and interpretation of already available results are underway.

Pyrolysis results

Rock-Eval pyrolysis/TOC results for 326 samples are given in Appendices 2 and 3. In the Horn River Group part of the section. The most notable difference in pyrolysis signatures occur between the Upper Canol (1670-1699.3 m MD) and the underlying section (Fig. 7). The Upper Canol sample set is greatly influenced by the fissile "Imperial-type facies" at 1674.0-1699.2 m and shows the median TOC values of only 1.9% and maximum 3.5% TOC. The Tmax values are markedly low at that interval (Fig. 7). The Basal and Middle Canol intervals (1785.5-1699.2 m MD) show very similar properties of organic matter with TOC values 5.1% (median) and 8.8% (maximum).

The black shale interval at 1811.8-1784.5 m MD, correlating to the Bell Creek Member (Pyle et al., 2014), retrieves TOC values of 4.4% (median) and 6.9% (maximum). The Bluefish Member shows variable TOC with 3.9% (median) and maximum value of 8.9% (Appendix 2). Considering these rocks are in post-peak oil generation, the initial TOC could have been substantially higher.

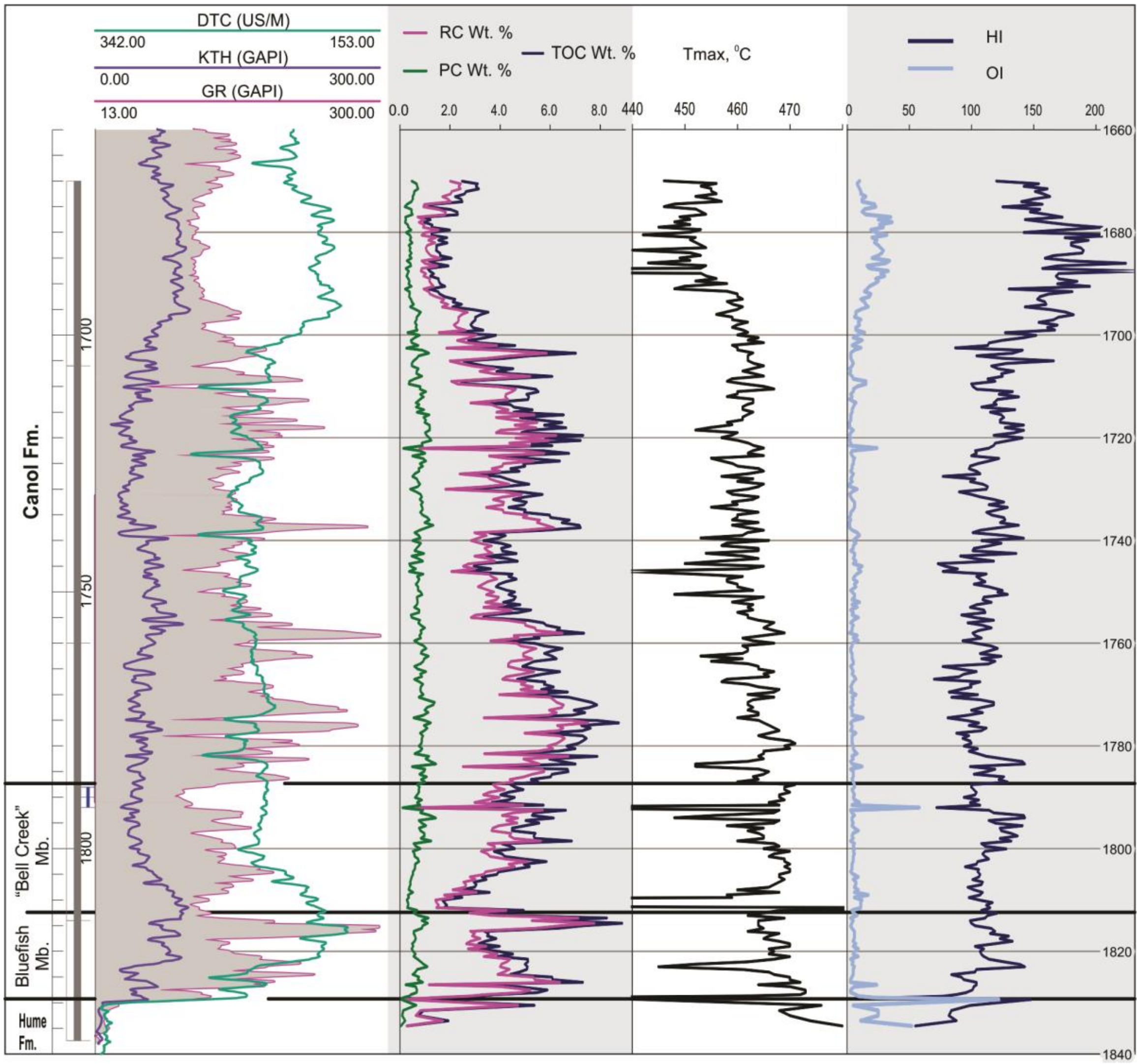

Figure 7: Rock-Eval results for 326 samples from Little Bear N-09 well; SGR and sonic logs are also shown.

The hydrocarbon is of high quality with very high S2 vs. TOC, which is characteristic of Canol play kerogens offsetting them from Type II towards Type I (Figs. 8 and 9). The pseudo van Krevelen cross-plots show overall type II kerogen (Figs. 10 and 11). The production index (Fig. 12) and T_{max} vs. HI cross-plot (Fig. 13) show the post peak oil generation and marginally into condensate zone demonstrating the very attractive prospect for the Canol shale play in exploration areas.

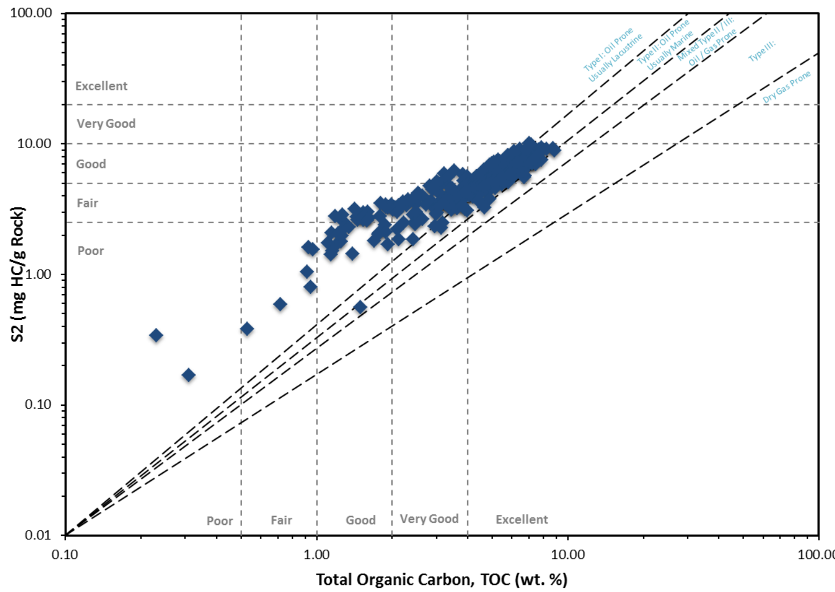

Figure 8: Plot of S2 vs TOC for 326 samples from Little Bear N-09 well. Dashed lines indicating poor to excellent boundaries from Peters, 1986; Peters and Cassa, 1994.

The Upper Canol also stands out of the underlying Horn River Group by pronouncedly high OI and HI (Figs. 7 and 11A). Given the recent core recovery (2012), natural sealing of organic matter in poorly permeable siliceous shale, and stratigraphically controlled distribution, the possiblility of post-drilling oil oxidation to produce elevated OI is discarded. There is a distinct difference in the oxygen index (OI) of the upper part of the core (1665-1705 m) from the underlying section. These Upper Canol spikes suggest distinct change in kerogen type, probably the surge of siliciclastic influx and enrichment in coaly detritus. Discussed above trace-metal signatures (Figs. 5 and 6) and presence of siderites also suggest shallowing and/or transfer the seafloor to suboxic depositional environment.

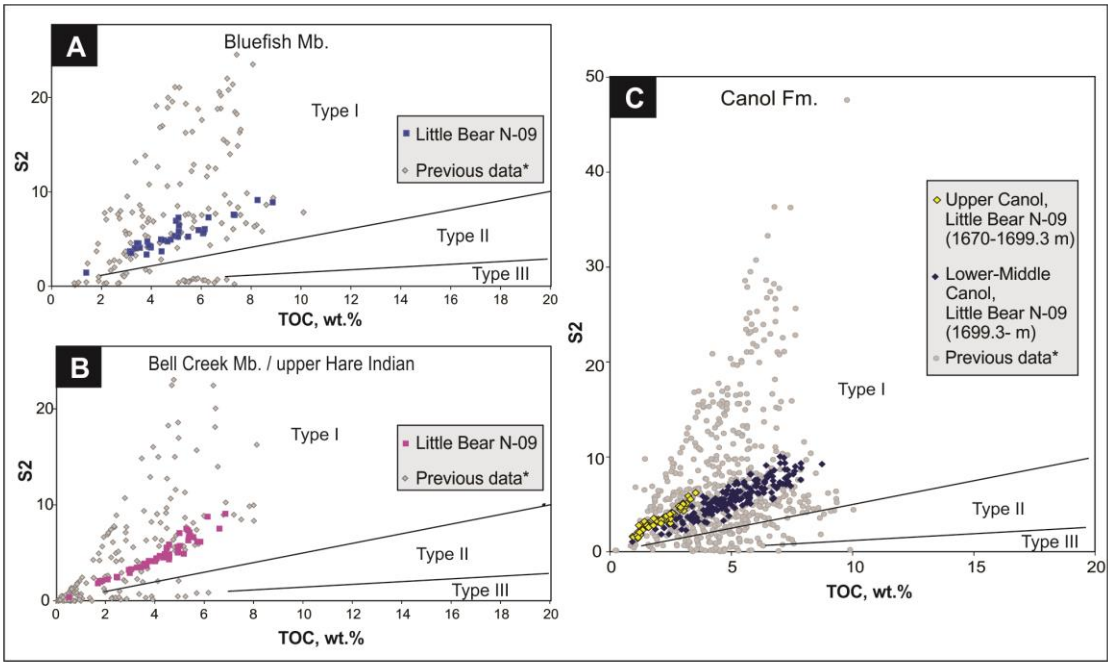

Figure 9: S2 vs TOC plotted against previous data for correspondent stratigraphic units: (A) Bluefish Mb, (B) Bell Creek Mb (in earlier works approximating upper Hare Indian or "grey shale member"; (C) Canol Fm. *Source of previous data: Pyle et al., 2011; Pyle and Gal, 2012,2013; Gal and Pyle, 2012; unpublished data).

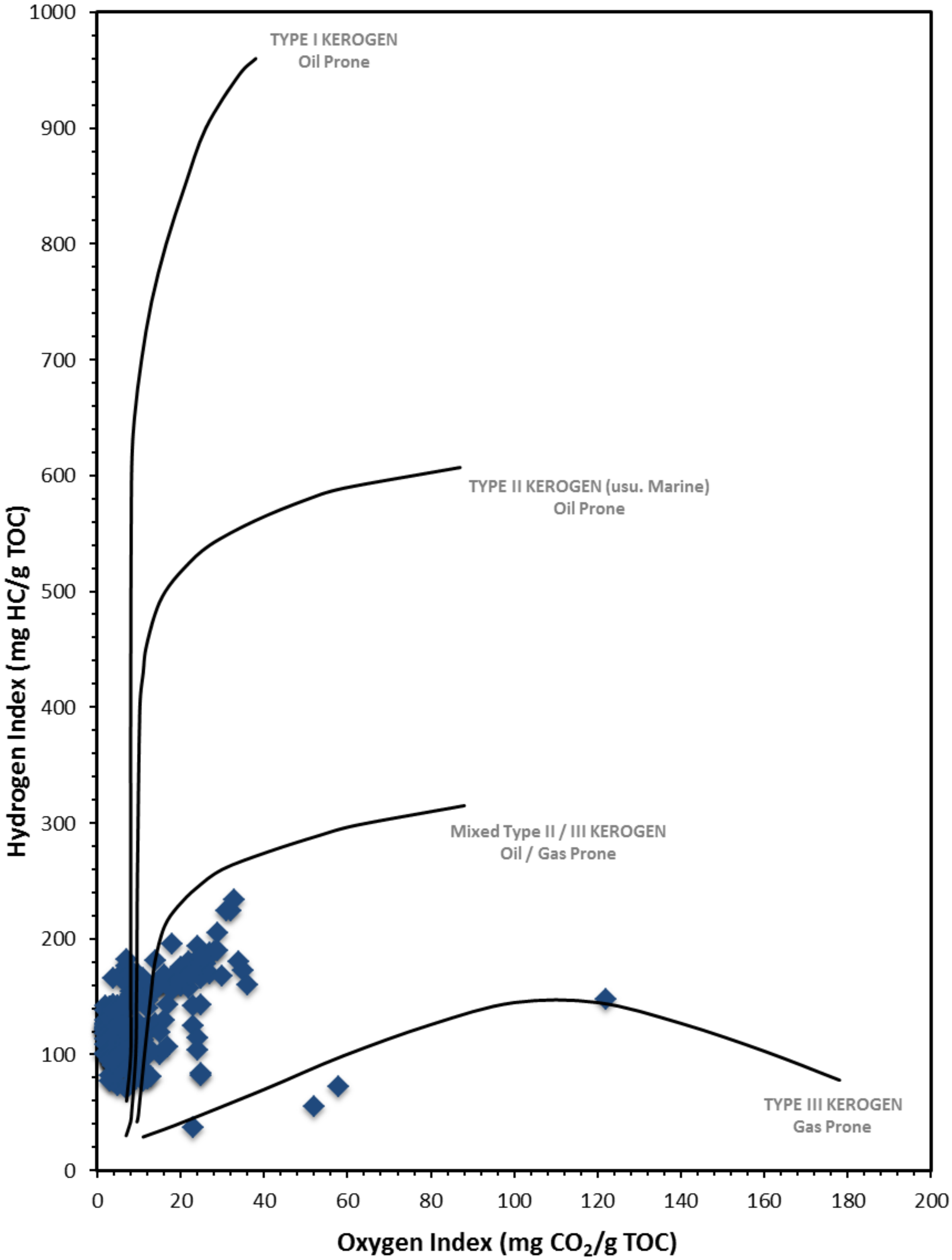

Figure 10: Pseudo van Krevelen (HI vs OI) diagram for 326 samples from Little Bear N-09 well

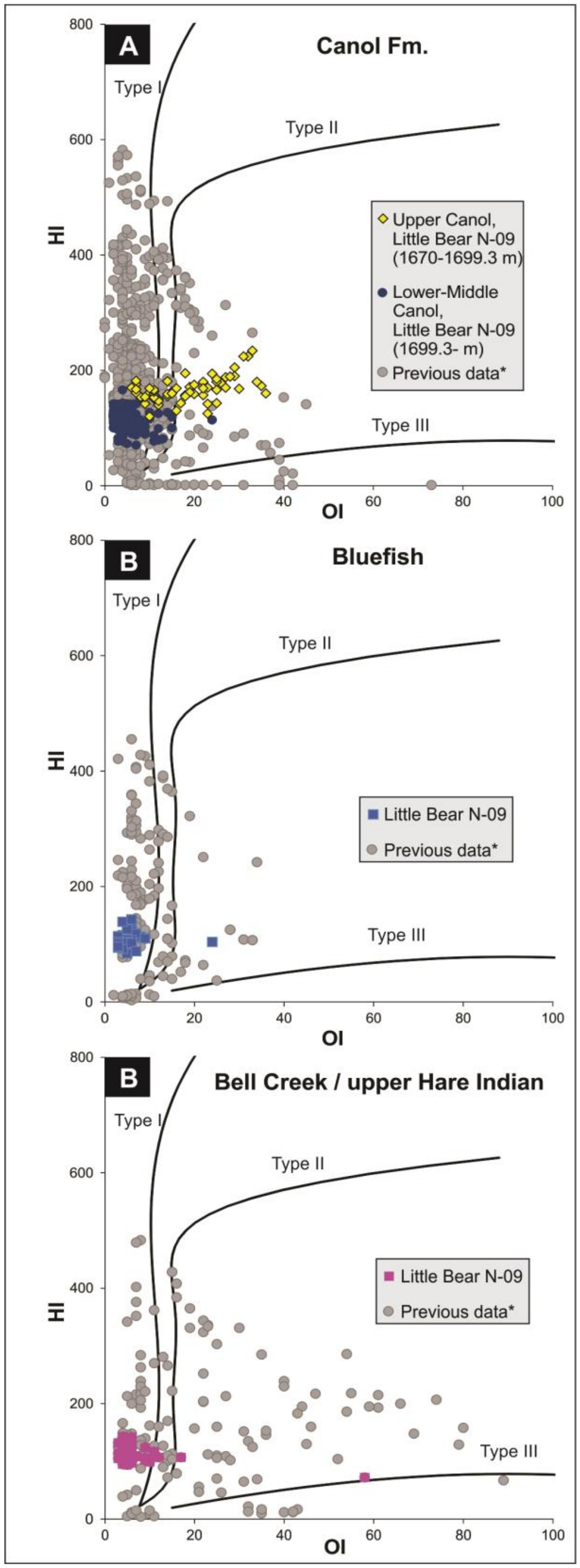

Figure 11: Pseudo van Krevelen diagrams (HI vs. OI) comparing Upper Canol, Basal-Middle Canol, Bell Creek, and Bluefish lithostratigraphic units of Little Bear N-09 with previous data for correspondent units in the Mackenzie River Corridor. See Figure 9 caption for data sources.

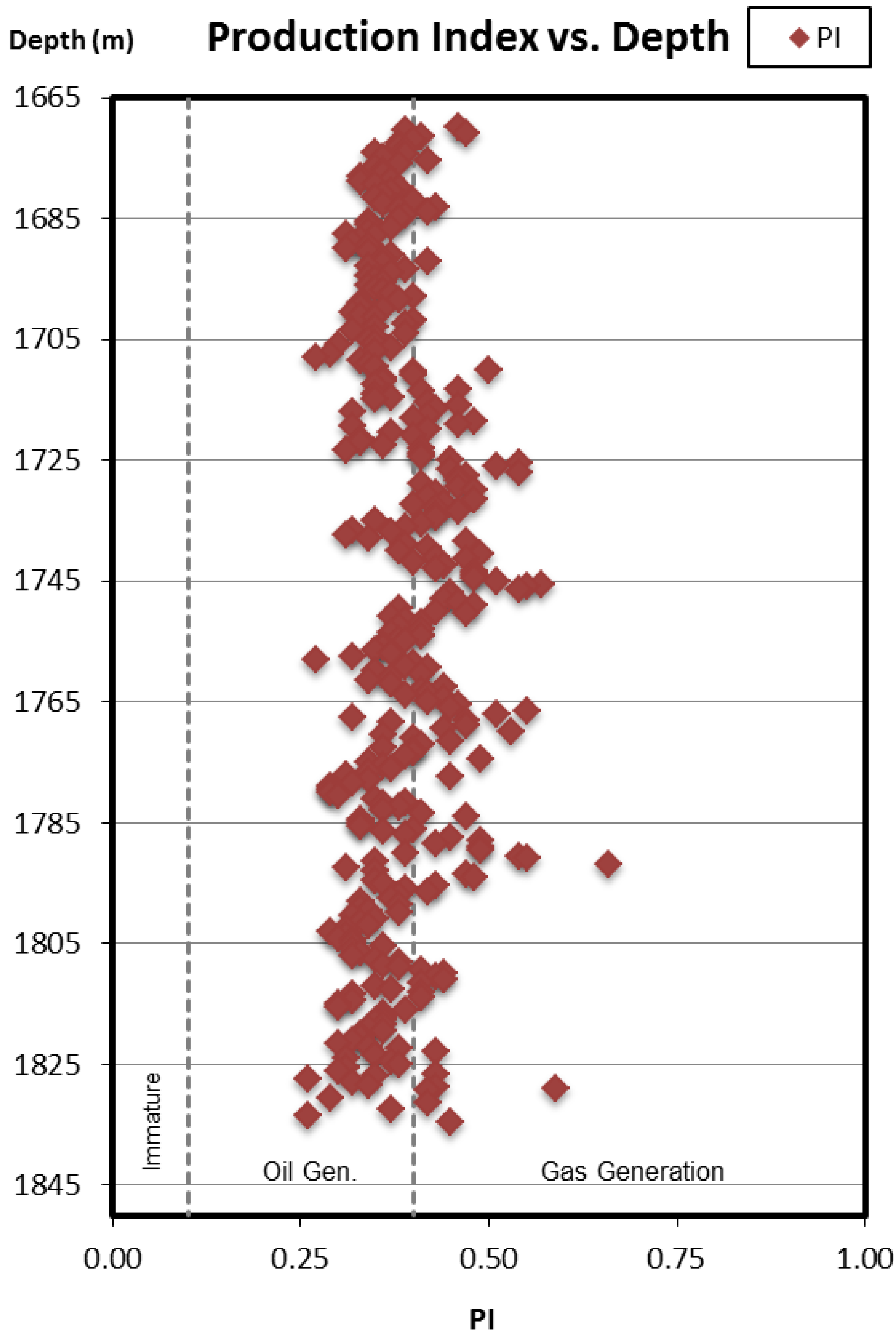

Figure 12: Plot of Production Index (PI) with depth for 326 samples from Little Bear N-09 well

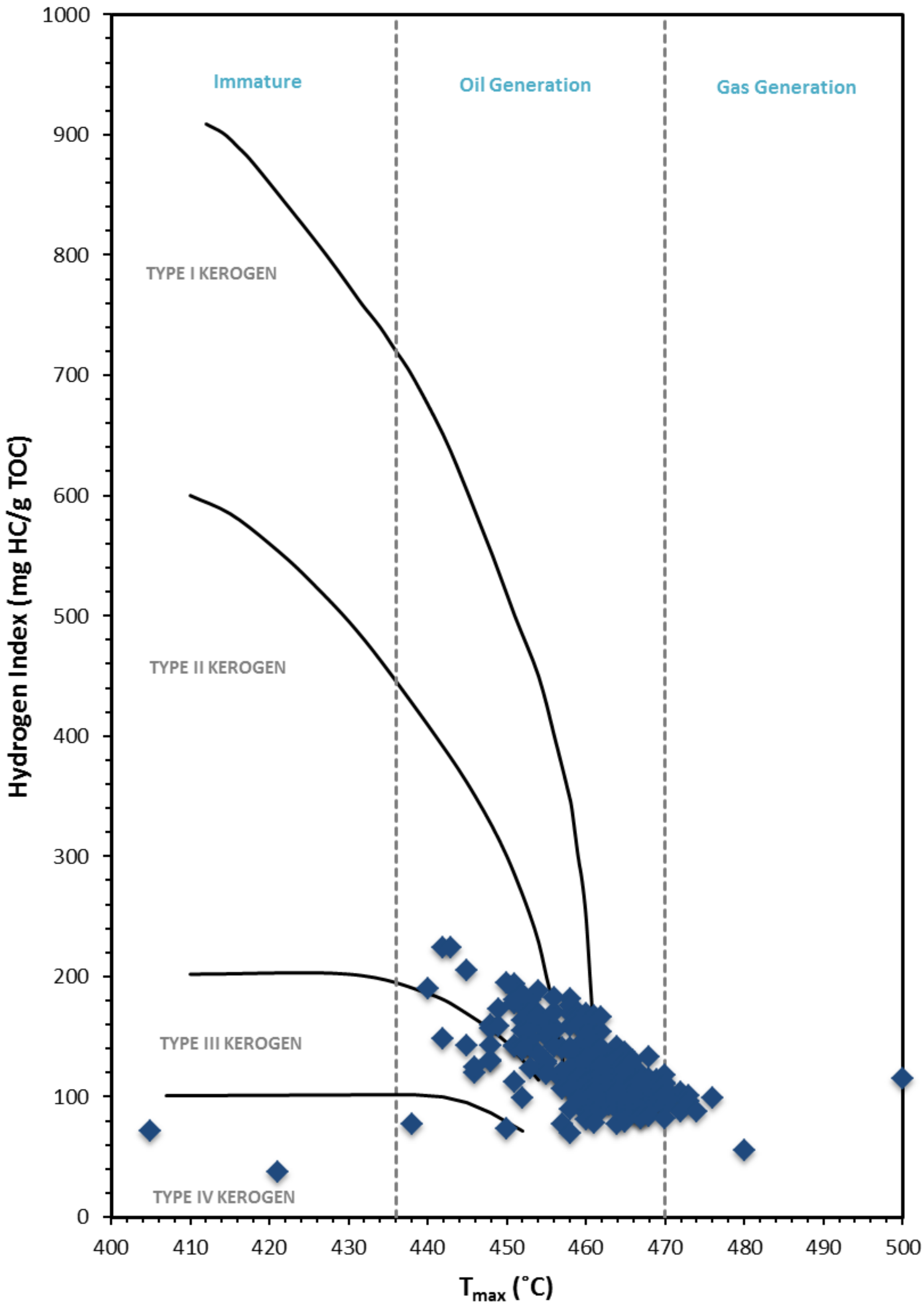

Figure 13: Plot of Tmax vs Hydrogen Index (HI) for 326 samples from Little Bear N-09 well

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