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Publications in this series have not been edited; they are released as submitted by the author

Contents

Appendix 1.

New XRD data from Kugaluk N-02, Little Bear N-09, and Norman Wells P32X wells.

RESUMÉ

Les levés minéralogiques représentent une partie importante des études sur les réservoirs de schiste. De nouveaux résultats, fondés sur des analyses par diffraction des rayons X (diffraction X), sont rapportés dans le présent document pour 101 carottes prélevées dans trois puits, provenant en majeure partie de mudrocks riches en matière organique du Groupe de Horn River (GHR), dans la vallée du Mackenzie. La base de données de diffraction X combinée de la NTGS-CGC offre une résolution suffisante pour une caractérisation minéralogique robuste des unités stratigraphiques. La Formation de Canol présente la plus haute teneur en quartz (médiane de 82 %) et la plus faible teneur en argile (médiane de 7 %). L'illite domine dans les argiles du GHR. La Formation de Hare Indian et la Formation de Canol inférieure sont calcaires et dolomitiques à des degrés variables; la Formation de Canol supérieure est dolomitique, mais non calcaire; les schistes de la Formation d'Imperial basale sont sidéritiques-dolomitiques et non calcaires. Nous pourrons arriver à une caractérisation plus précise après la résolution de questions de corrélation des unités stratigraphiques entre la CGC et la NTGS. Les nouvelles données de diffraction X sont également utilisées pour caractériser le contact entre la Formation de Hume et le GHR (une discordance ennoyée) dans le puits Kugaluk N-02 de la partie nord de la plaine Anderson.

ABSTRACT

Mineralogical surveys are an important part of shale reservoir studies. New results, based on X-ray diffraction analyses (XRD), are reported here for 101 core samples from three wells, mostly from organic-rich mudrocks of the Horn River Group (HRG) in Mackenzie Plain area. The combined NTGS-GSC XRD database has sufficient resolution for robust mineralogical characterization of stratigraphic units. The Canol Formation has the highest quartz (82% median) and the lowest clay content (7% median). Clays in the HRG are dominated by illite. The Hare Indian and lower Canol formations are variably calcareous and dolomitic; the upper Canol is dolomitic but non-calcareous; basal Imperial shales are sideritic-dolomitic and non-calcareous. More accurate characterization can be achieved by resolving issues between GSC and NTGS correlation of stratigraphic units. New XRD data are also used to characterize the Hume-HRG contact (a drowning unconformity) in Kugaluk N-02 well of northern Anderson Plain.

INTRODUCTION

In the Mackenzie Project area, the Devonian is a thick (locally over 1 km) stratal succession composed of carbonate, evaporite, and siliciclastic rocks (Fig. 1). These record the evolution of depositional environments from passive-margin shallow-water carbonate platform through sediment-starved epiplatform anoxic basin to siliciclastics-filled foreland basin (Hadlari et al., 2009; Morrow, 2012; Kabanov and Gouwy, in press). The Devonian Stratigraphic Framework activity is part of the Mackenzie Project of GEM-2 focusing on sub-Imperial Devonian strata. The succession under study is notably rich in hydrocarbons occurring in an oil to wet gas maturity window. Most significant assets are the giant conventional oilfield at Norman Wells and the giant prospect of non-conventional gas and light oil locked in Middle-Upper Devonian mudrocks of the Horn River Group (Pyle and Gal 2016; Kabanov and Gouwy, in press). This frontier shale prospect is also known as the Canol shale play (AANDC 2014) after its thickest, most organic-rich, and most favorable for fracking stratigraphic unit.

Mineralogical surveys are an important part of non-conventional tight reservoir science as mineral species within the rock matrix control brittleness/frackability and other key properties such as permeability, wettability, and shale imbibition (Ross and Bustin, 2009; Dehghanpour et al., 2015; Peters et al., 2016). Previously, the mineral composition of the Horn River Group in the study area was surveyed by the Northwest Territories Geological Survey (NTGS). The NTGS data include 182 well samples (cuttings) and 209 outcrop samples from the Horn River Group and the basal beds of the Imperial Formation. Of these, mudrock units (excluding Ramparts Formation) were tested by 204 outcrop and 179 well samples. The NTGS mineralogical data was published in full by Pyle et al. (2014) and Pyle and Gal (2016).

New mineralogical data include X-ray diffraction (XRD) results from 101 core samples from wells Kugaluk N-02, Norman Wells P32X, and Little Bear N-09 (Fig. 2). The well Kugaluk N-02 is located in northern Anderson Plain and the other two wells in the central Mackenzie Plain. Most samples were taken from the Horn River Group and across stratigraphic contacts between formations. The well Little Bear N-09 was drilled during the recent exploration campaign in the Mackenzie Valley (2012-2014). Husky Little Bear N-09 was one of the first wells with representative cored sections through Horn River Group mudrocks, completed in 2012 and released to the public in 2014. The exploration well Kugaluk N-02 was drilled in 1965 with continuous core recovery throughout the Devonian succession. Norman Wells P32X well is one of deviated production wells at Norman Wells Oilfield drilled in 1997 and cored across the Canol – Kee Scarp contact. Lithological descriptions, geochemical and pyrolysis data for these three wells were published earlier (Kabanov, 2015; Kabanov et al., 2015). This paper offers a preliminary mineralogical survey of mudrock units in the Horn River Group based on NTGS and new data. Samples from Kugaluk N-02 well were analysed to characterize the Hume/Hare Indian contact (889.1-896.3 m) and specific facies within the Landry Formation (977.8-1054.0 m) with results to be discussed in future publications.

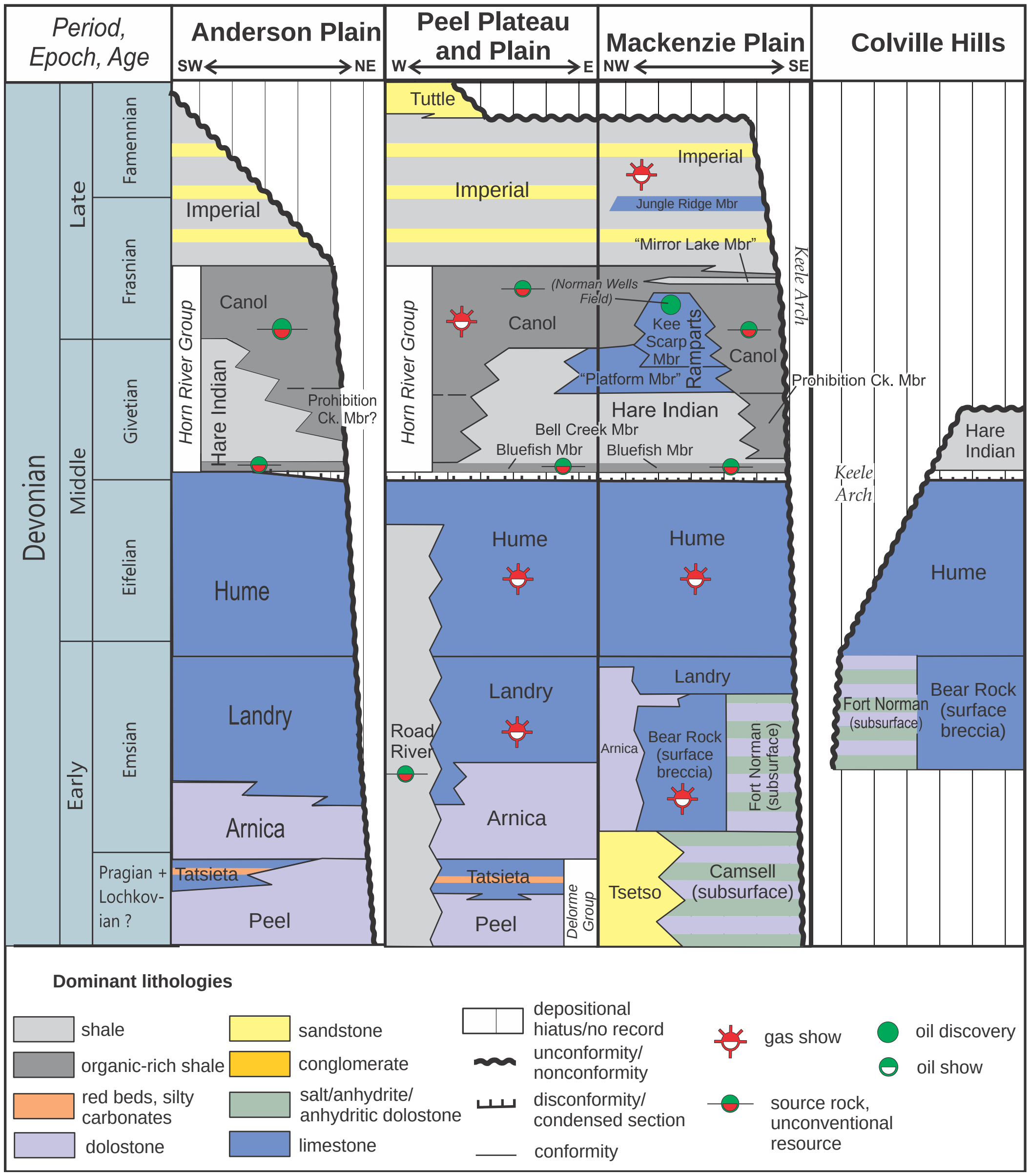

Figure 1: Table of Formations for the Devonian System of central and northern Mackenzie Corridor. Formation charts are taken from the following sources, with modifications: Anderson Plain based on Morrow (2012). Peel area and Colville Hills based on Rocheleau and Fiess (2014); Mackenzie Plain from Kabanov et al. (2016) for the Horn River Group to basal Imperial Formation and Rocheleau and Fiess (2014) for other Devonian units.

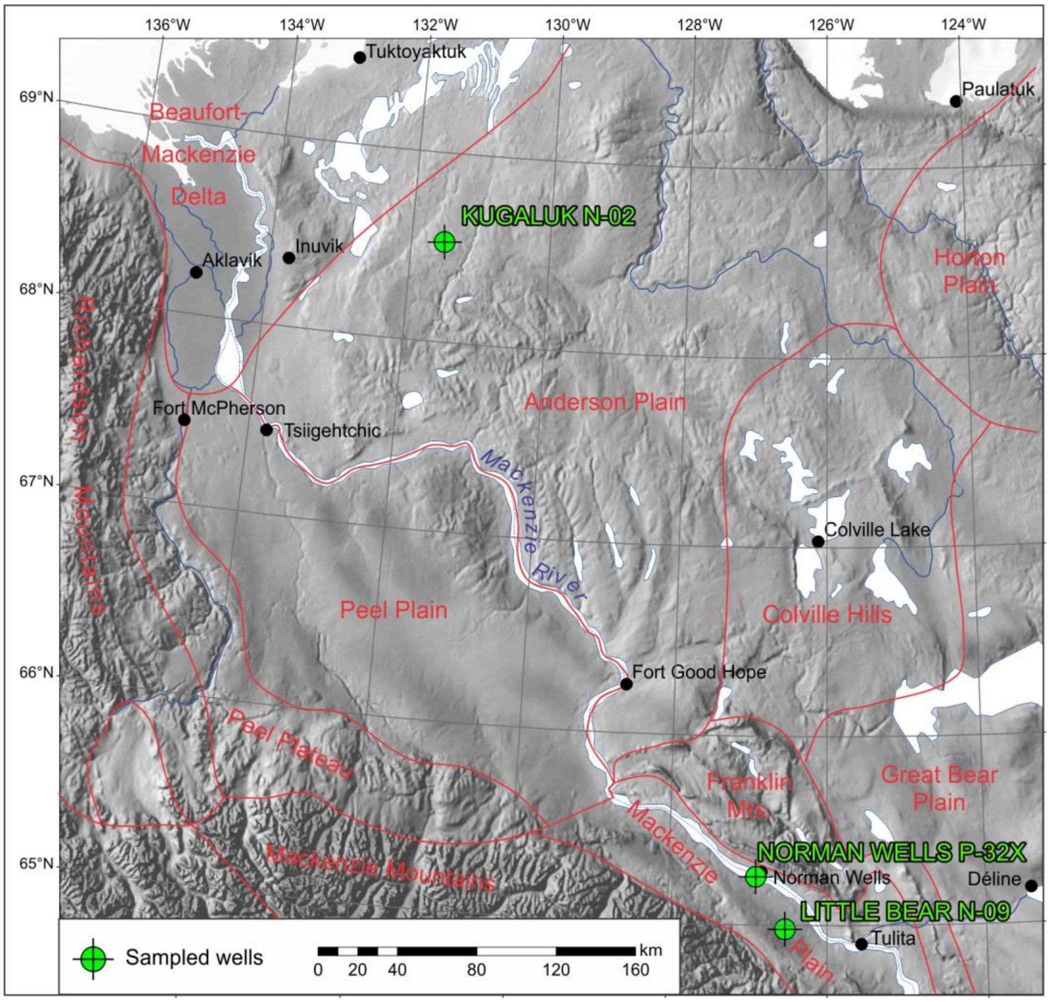

Figure 2: Location of studied materials on a topographic basemap with main physiographic regions outlined

METHODOLOGY

The mineralogy of bulk materials was determined by X-ray powder diffraction analysis (XRD). The bulk samples were pulverized in isopropyl alcohol using a McCrone mill. The samples were air-dried and then back pressed into an aluminum holder to produce randomlyoriented specimens. X-ray patterns were recorded on a Bruker D8 Advance Powder Diffractometer equipped with a Lynx-Eye Detector, Co Kα radiation set at 40 kV and 40 mA.

Initial identification of minerals was made using EVA (Bruker AXS Inc.) software with comparison to reference mineral patterns using Powder Diffraction Files (PDF) of the International Centre for Diffraction Data (ICDD). Quantitative analysis was carried out using TOPAS (Bruker AXS Inc.), a PC-based program that performs Rietveld refinement (RR) of XRD spectra, based on a whole pattern fitting algorithm. To ensure the quality of results, synthetic mixtures using reference minerals in variable amounts were prepared and analyzed. The fidelity of the result was also checked with elemental ICP-MS for Ca and Mg in carbonate notation (Fig. 3). Samples were selected from black-shale units (Bluefish, Prohibition Creek, Vermillion Creek, and Dodo Canyon members) where Ca^{2+} and Mg^{2+} are strongly bound in calcite and dolomite with the least contribution from clay-bound Mg^{2+}.

Three samples with barytocalcite were also included. This test showed a good R^2 (square of the Pearson product moment correlation) of 0.9 (Fig. 3).

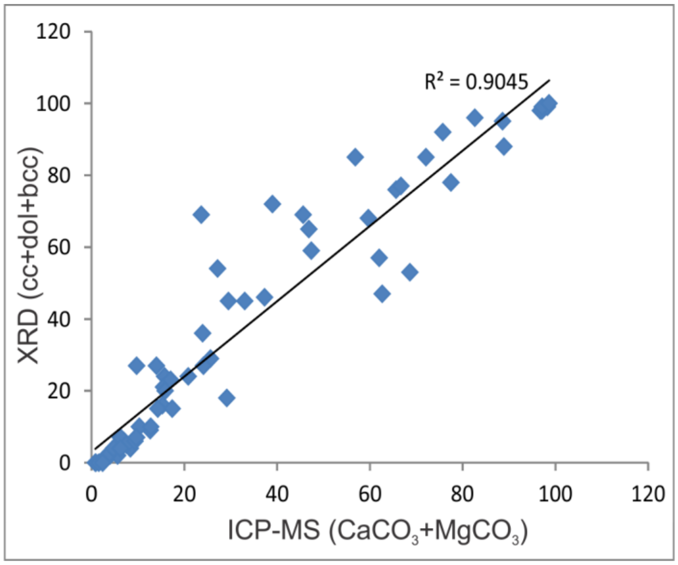

Figure 3: XRD vs. ICP-MS cross-plot with samples from black-shale units of Little Bear N-09 well; *cc* is calcite, *dol* is dolomite, *bcc* is barytocalcite.

XRD RESULTS

New data from Little Bear N-09 well

Our new data include 67 samples from Little Bear N-09 collected from the upper Hume - Mirror Lake stratigraphic interval (Fig. 4). The section is dominated by SiO_2 in the form of crystalline quartz. Our XRD samples were collected preferentially from high-carbonate intervals as seen on geochemical logs on left side of Figure 3 and therefore are not considered representative for overall silica content. More credible evaluation of siliceousness vs. shaliness is conveyed by ICP-MS data from Little Bear N-09 and Loon Creek wells (706 samples in total; Kabanov et al., 2016; Kabanov and Gouwy, in press). Based on these data, the succession is subdivided into three main packages: the alumina-rich Bluefish-Francis Creek interval with 10-18% median Al_2O_3 and 57-60% median SiO_2, the clay-lean siliceous Prohibition Creek - Dodo Canyon interval with 5.8-9.0% median Al_2O_3 and 68-78.5% median SiO_2, and the generally clay-rich mudrocks above with 13-17% median Al_2O_3 and 60-66% median SiO_2. Inside the overall clay-rich interval, the black shale of the lower Loon Creek Member is a prominent clay-lean and siliceous bed with 12.5% median Al_2O_3 and 67-69% median SiO_2. The Dodo Canyon Member stands out by its extremely high matrix silica (75-79% median SiO_2) and lowest Al_2O_3 for the whole succession (5.7-6.7%).

The XRD results show generally minor content of clay minerals which are dominated by illite with trace amounts of kaolinite and mixed-layer clay minerals. The clay mineral content is higher in Bluefish – Francis Creek interval (8.5% median) and Mirror Lake Member (13-24% in three available samples) than in the Prohibition Creek – Dodo Canyon black-shale interval. The latter shows only 3% median values of illite, and the Dodo Canyon Member is the cleanest cherty unit with only 2% median illite.

Carbonate minerals are represented by a mixture of calcite and dolomite at the Bluefish – Vermillion Creek interval and dolomite in Dodo Canyon member (Fig. 4), which is consistent with ICP-MS data (Kabanov et al., 2016). Three samples showed presence of barytocalcite (Fig. 4). The Mirror Lake Member stands out due to the presence of siderite in the carbonate fraction.

The "Other" on normalized XRD logs mostly includes plagioclase and Fe sulphides (pyrite and some marcasite). Strong covariation of Fe and S at the Bluefish – Dodo Canyon interval (left part of Fig. 4) indicates that both elements are bound in Fe sulphides (Fig. 4). In grey-shale units, most obviously in the Mirror Lake Member, Fe and S decouple with the surplus of iron apparently bound in siderite, detrital minerals, and illite (Fig. 4).

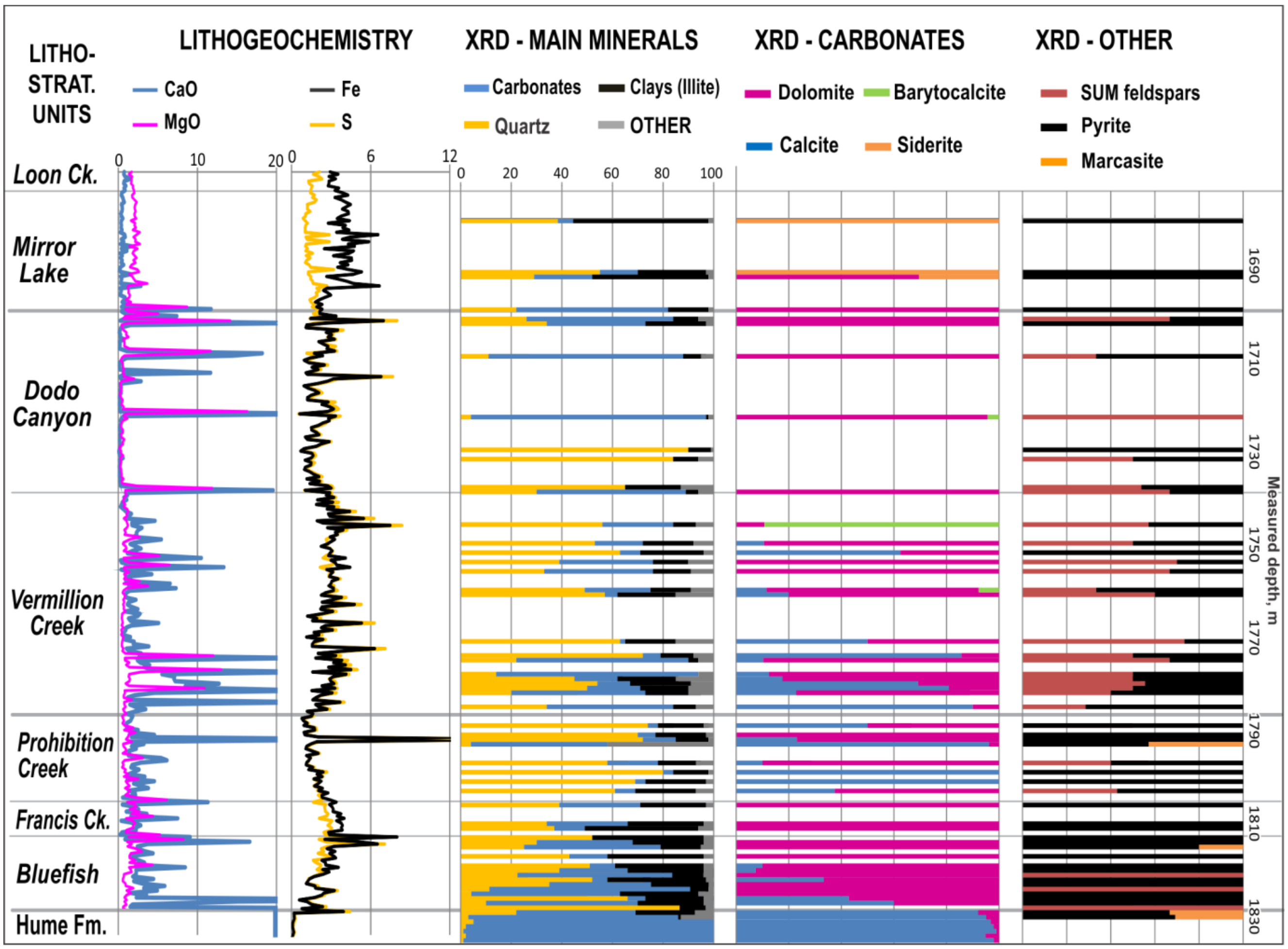

Figure 4: XRD and selected geochemical logs for the Horn River Group of Little Bear N-09 well. Lithofacies and geochemical data are available in (Kabanov et al., 2015).

Mineralogy of the Horn River Group in Mackenzie Plain area

XRD data collected to date (445 samples from Pyle et al., 2014; Pyle and Gal, 2016, and this work) allow for robust mineralogical characterization of the Horn River Group at the formation level (Fig. 5 and Table 1). The Ramparts and Hume formations and the sample set from Silvan Plateau G-51 well are here excluded from analysis. The latter is located at the southern margin of the Mackenzie Plain study area of NTGS, and most samples from the interval correlated by Pyle et al. (2014) to the Canol

Formation (17 out of 18 samples) contain siderite. However, siderite is not a feature of the Canol Formation in all other sections (Table 1), and the data from this well are set aside until better correlation is available. Samples with barytocalcite are rare (only 3 samples from Little Bear N-09), and they are also excluded. Non-quantifiable trace amounts of minerals were zeroed. In total, 396 samples were used in this analysis (Table 1). Selected diffractograms from new measurements are given in Figure 6.

The Bluefish Member is the only sub-formational unit traced unequivocally across the Mackenzie Plain and southern Peel area, therefore its sample subset is compared with subsets from lithostratrigraphic units of higher rank. The upper Hare Indian, including Bell Creek, Francis Creek, and Prohibition Creek members, changes laterally from thick grey shale – siltstone facies to thinner dark colored mudrocks superficially indistinguishable from the Canol Formation. Its correlation and nomenclature is currently under discussion (Pyle and Gal, 2016; Kabanov and Gouwy, in press).

Figure 5 depicts main mineral components together constituting over 90% of mineral matrix in most samples. The NTGS data blend grey-shale and dark-shale XRD samples into the Hare Indian category. Out of the upper Hare Indian sample set, a distinct subset of 19 dark mudrock samples from 3 wells of central Mackenzie Plain is analyzed as the Prohibition Creek Member of Kabanov and Gouwy (in press) replacing the informal black-shale unit of the upper Hare Indian Formation of Pugh (1993). This black-shale subset combines cutting samples from Bear Rock O-20 and Bluefish K-71 wells (Pyle et al., 2014) and the new core samples from Little Bear N-09 well.

The Canol Formation has the largest number of mineralogical samples (Table 1). It is overall the most cherty and least argillaceous unit in the succession. Carbonate material is strongly dominated by dolomite and calcite, and few samples with carbonate content over 10% are dominated by calcite. However, known calcareous samples reside in the lower Canol (Vermillion Creek Member of Kabanov and Gouwy, in press), whereas the upper Canol (Dodo Canyon Member of Kabanov and Gouwy, in press) is dominated by dolomite. Separate mineralogical characterization of these two members will be possible upon more accurate correlation of the Vermillion Creek and Dodo Canyon members to outcrops measured and sampled by. Twenty-two samples from the basal part of the Imperial Formation include those from verified Mirror Lake and Loon Creek members of Kabanov and Gouwy (in press) and undifferentiated Imperial samples of NTGS (Pyle et al., 2014). The Imperial subset shows higher overall clay content, complete absence of calcite, and common occurrence of siderites or a mixture of siderite and dolomite and/or ankerite. Clay minerals are dominated by illite in all stratigraphic subsets, but in difference to new data from Little Bear N-09, chlorite and kaolinite occur in substantial quantity in NTGS samples (Table 1).

Sulphate (gypsum and jarosite), sulphide (pyrite and marcasite), and feldspar minerals occur in trace to minor amounts and were excluded from analysis of the main components (Fig. 5). Feldspars are likely detrital and dominated by plagioclase with minor K-feldspar. Gypsum occurs in all mudrock units in both outcrop and subsurface samples with median values 1-5%. Approximately one-third of samples in both outcrop and subsurface subsets contain gypsum. Jarosite is absent in subsurface samples but common in outcrops (2% median and 3% at 90 percentile). No anhydrite is reported. Sulphides are almost entirely represented by pyrite. Outcrop samples are somewhat depleted with pyrite (2% median and 4% at 90 percentile) compared to subsurface samples (3% median and 6% at 90 percentile), which can be attributed to weathering.

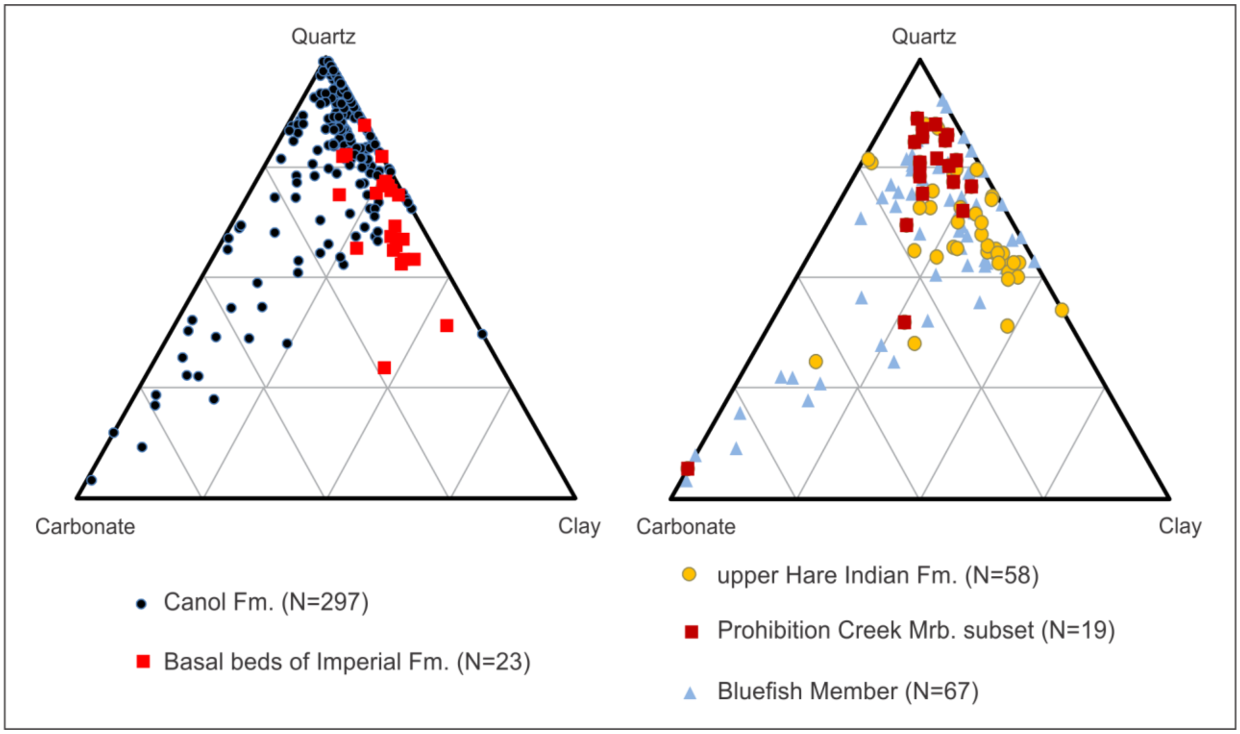

Fig. 5: Mineral composition of mudrock units of the Horn River Group of Mackenzie Plain and adjacent outcrops based on NTGS XRD data and the new dataset reported herewith.

		Quartz	Carbonate	Clay	Carbonate			Clays		
					Calcite	Dolomite	Siderite	Illite / Muscovite	Kaolinite	Chlorite
basal Imperial	10 percentile	51	1	16	n/d	n/d	n/d	13	n/d	n/d
basal Imperial	Median	62	5	28	n/d	2	0	16	2	10
basal Imperial	90 percentile	69	8	39	n/d	5	7	26	5	13
basal Imperial	Maximum	82	23	53	1	16	15	53	5	14
basal Imperial	StDev	11	5	10		3	4	10	2	5
basal Imperial	n(N)	22(22)	20(22)	22(22)	1(22)	17(22)	9(22)	22(22)	14(22)	18(22)
Canol	10 percentile	53	n/d	2	n/d	n/d	n/d	1	n/d	n/d
Canol	Median	82	n/d	7	n/d	n/d	n/d	6	n/d	n/d
Canol	90 percentile	95	17	19	8	6	n/d	15	n/d	4
Canol	Maximum	99	80	54	51	77	2	49	5	9
Canol	StDev	19	14	7	7	12		6	0.5	2
Canol	n(N)	263(263)	126(263)	261(263)	73(263)	90(263)	1(263)	260(263)	10(263)	131(263)
U. Hare Indian	10 percentile	44	3	8	n/d	n/d	n/d	6	n/d	n/d
U. Hare Indian	Median	58	7	26	3	3	n/d	14	1	5
U. Hare Indian	90 percentile	80	21	39	13	12	n/d	26	7	14
U. Hare Indian	Maximum	84	54	57	52	32	1	45	22	16
U. Hare Indian	StDev	15	11	13	10	7		9	5	5
U. Hare Indian	n(N)	55(55)	54(55)	54(55)	43(55)	37(55)	1(55)	53(55)	30(55)	37(55)
Bluefish	10 percentile	26	n/d	8	n/d	n/d	n/d	7	n/d	n/d
Bluefish	Median	59	12	19	6	2	n/d	12	n/d	n/d
Bluefish	90 percentile	75	59	36	16	45	n/d	30	4	11
Bluefish	Maximum	90	90	44	90	89	n/d	44	25	16
Bluefish	StDev	19	23	11	16	21		10	4	4
Bluefish	n(N)	57(57)	49(57)	56(57)	39(57)	44(57)	0(57)	55(57)	15(57)	22(57)

Table 1: Statistical parameters for stratigraphic subsets of XRD data; pyrite, feldspars and sulphates are not included; *n/d* stands for non-detectable with XRD method; *n(N)* denotes number of occurrences of a mineral (excluding trace amounts) in total number of samples.

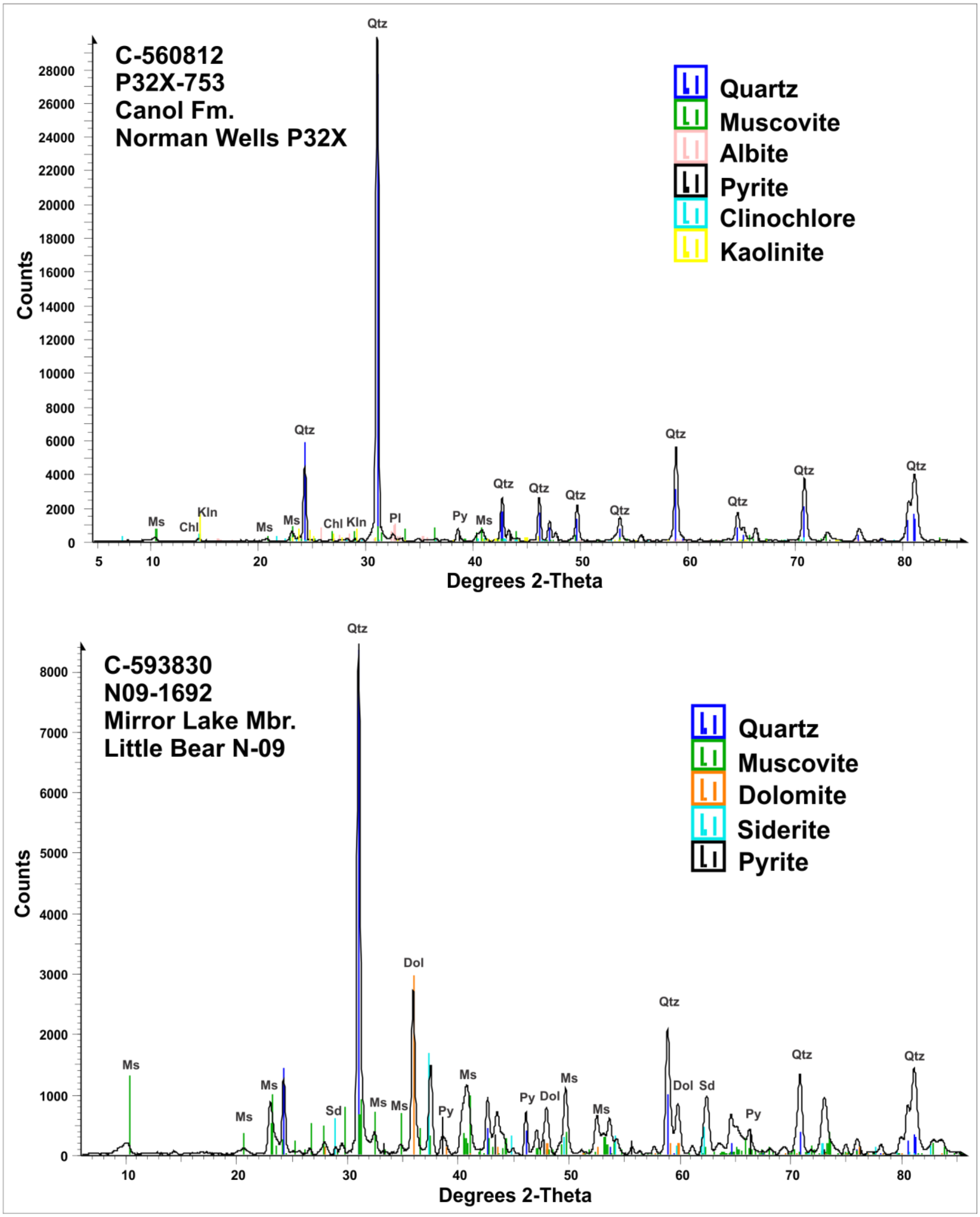

Figure 6: X-ray diffractograms of selected samples

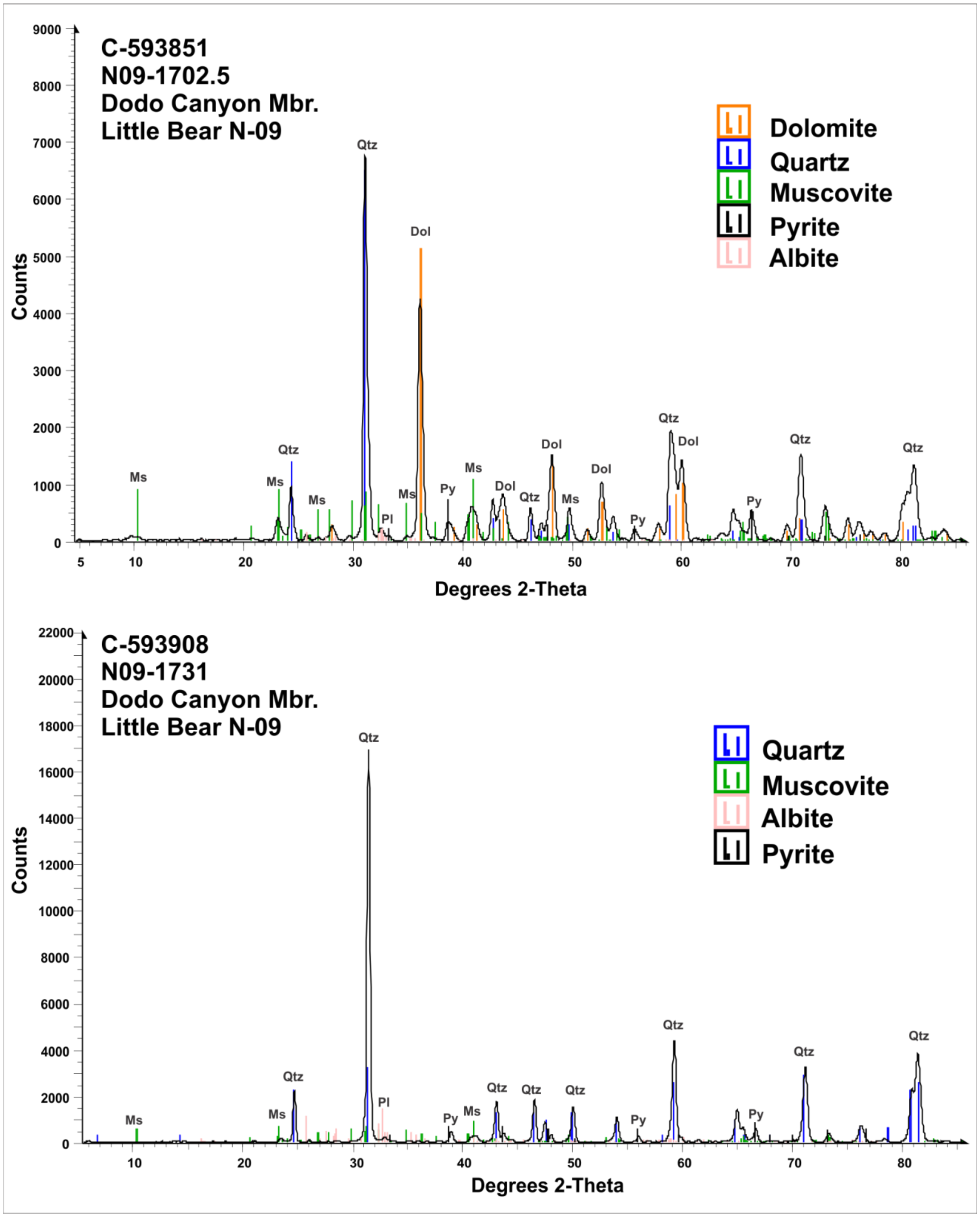

Figure 6 (continued): X-ray diffractograms of selected samples

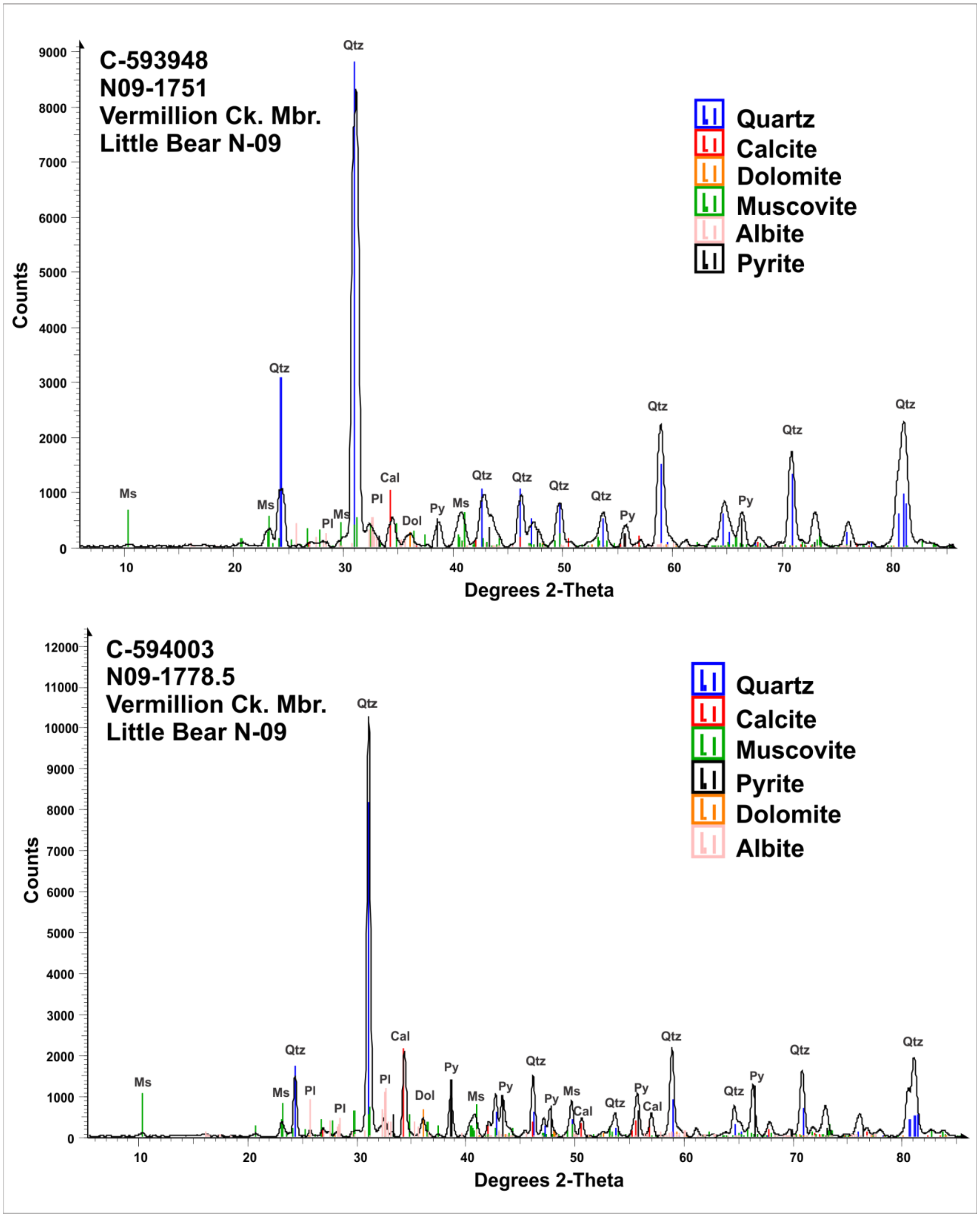

Figure 6 (continued): X-ray diffractograms of selected samples

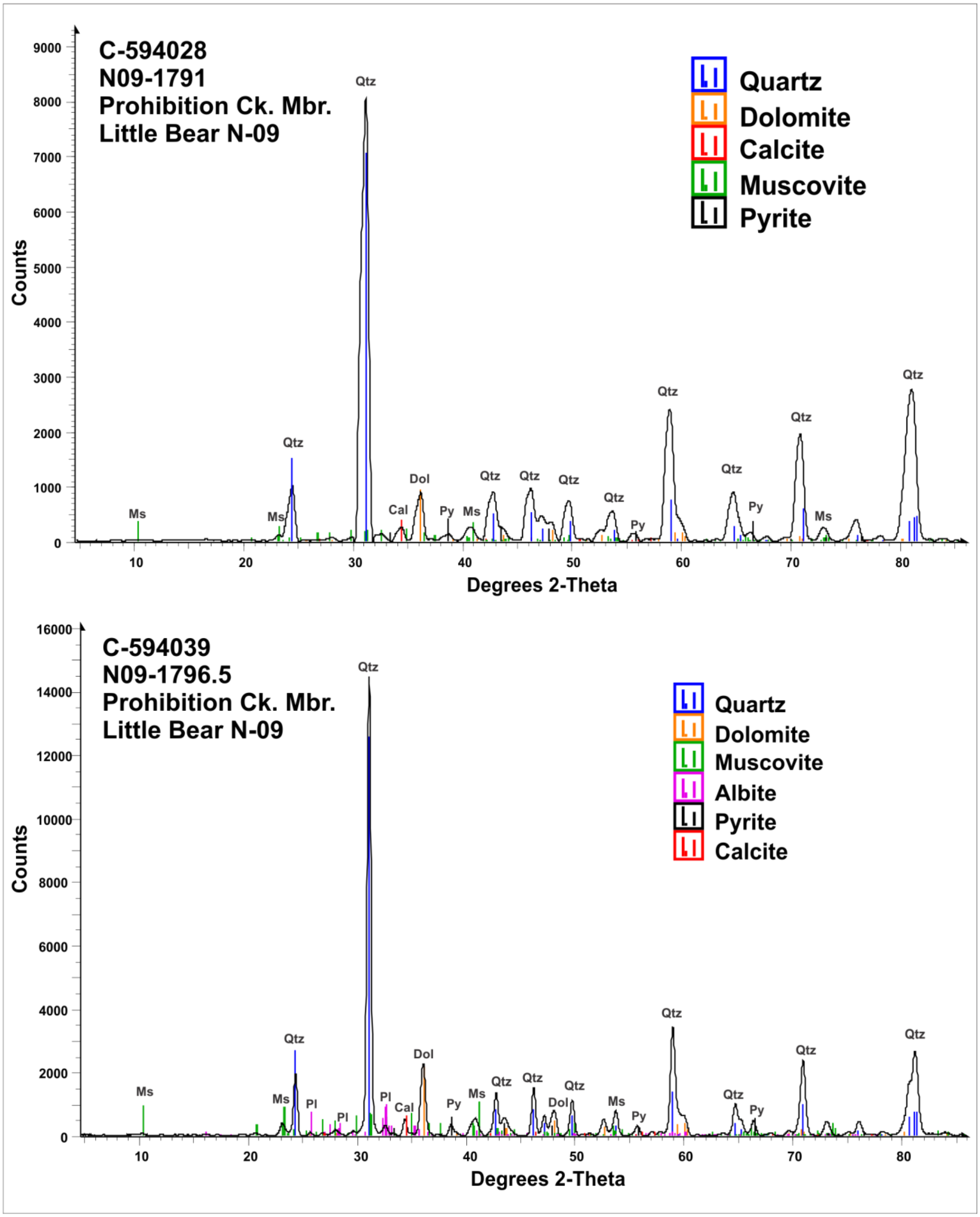

Figure 6 (continued): X-ray diffractograms of selected samples

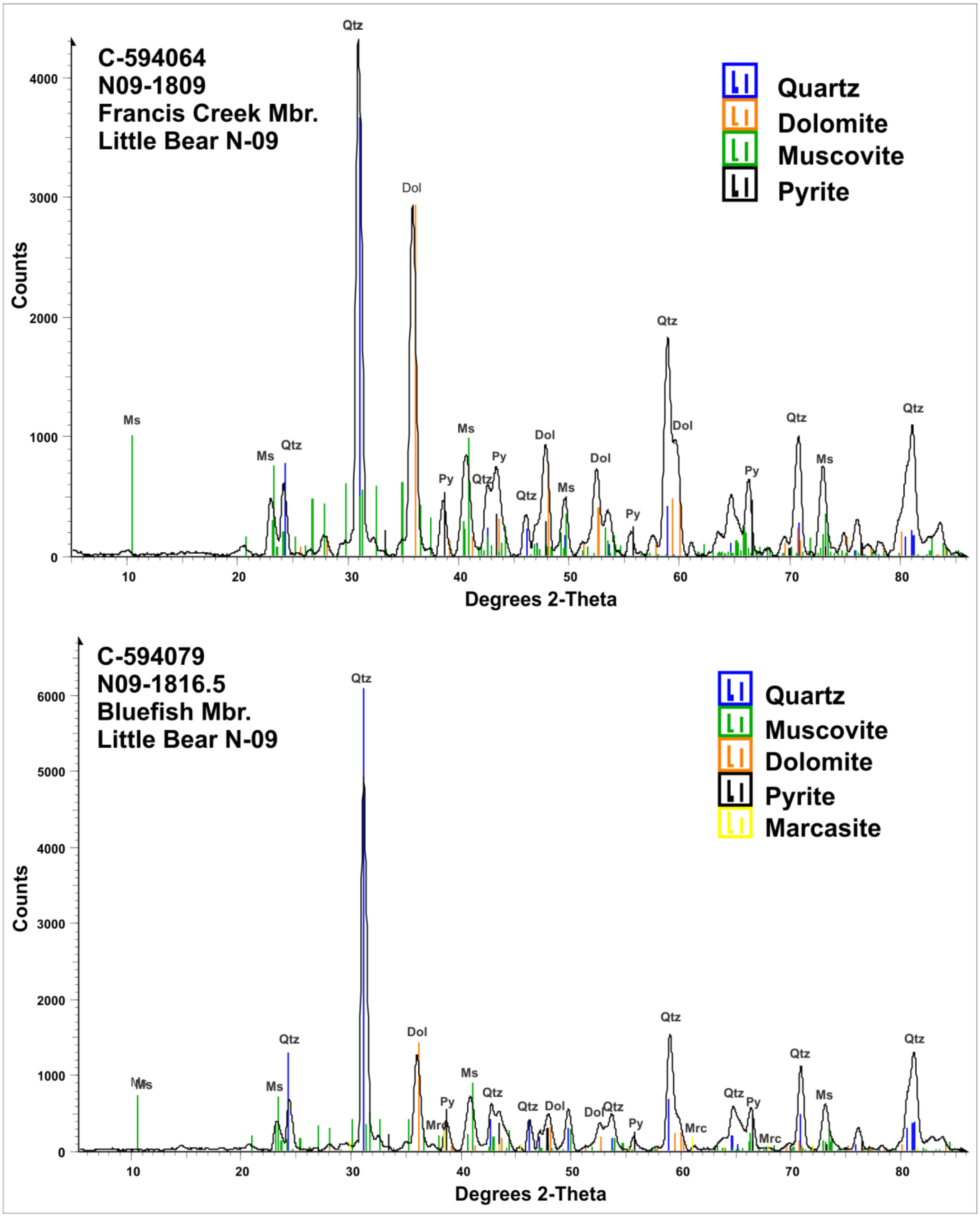

Figure 6 (continued): X-ray diffractograms of selected samples

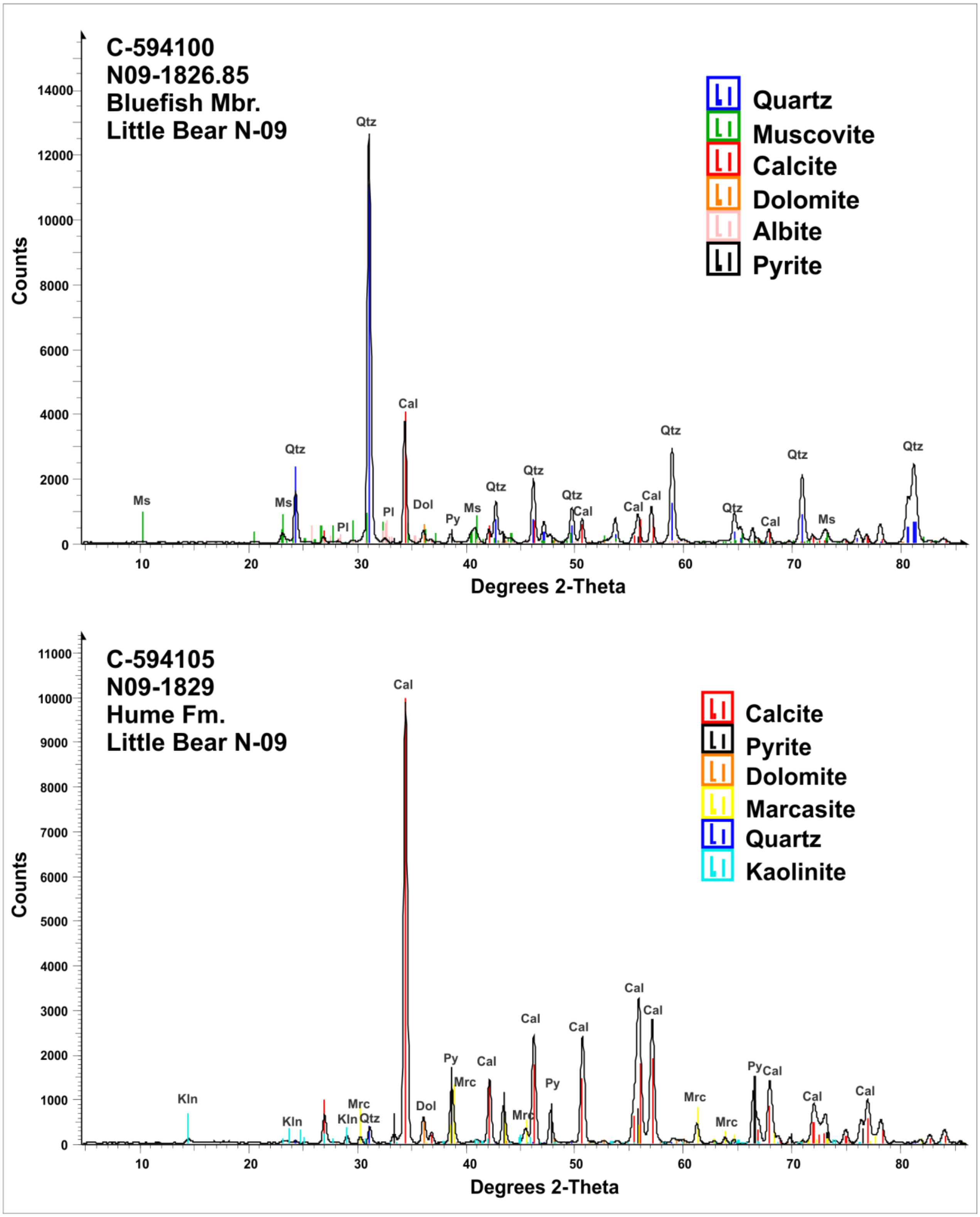

Figure 6 (finished): X-ray diffractograms of selected samples

Hume-Bluefish contact in Kugaluk N-02 well

In the central and northern Mackenzie Corridor, the base of the Horn River Group is a planar surface at the outcrop and regional scale and a prominent seismic horizon. This surface was repeatedly described as either abrupt but conformable (Bassett 1961; Pugh 1983, 1993) or disconformable with negligible amount of truncation prior to deposition of the black shale (Norris 1968, 1997; Gal et al. 2009; Pyle et al., 2014). Most recently it was referred to as the drowning unconformity (Kabanov and Gouwy, in press). The continuously cored Kugaluk N-02 well was drilled in the area of sparse geological data making it a regional reference section for lithostratigraphic units (Fig. 7A; Kabanov, 2015).

Nineteen new XRD samples across the Hume-Bluefish contact augment lithofacies observations described earlier (Figs. 7-9; Kabanov, 2013). Several lines of evidence suggest that the contact in Kugaluk N-02 well is conformable at the microscale, although usually appears sharp in outcrops. The upper 2.5-2.7 m of the Hume limestone is argillaceous and pyritic, in common with the overlying black shale. Mica is dominated by muscovite/illite. Increase of siliciclastic input prior to cessation of benthic carbonate factory is also evidenced by plagioclase grains in the upper two samples of the Hume Formation (Fig. 7B). Trace amounts of siderite in three samples are interpreted as forming during a reducing environment intermediate between oxic shallow subtidal conditions typical of the underlying Hume facies and anoxic sulphate reducing conditions for the overlying black shale.

Paleontological line of evidence includes, most obviously, the complete decline of bioturbation across the contact. Changes in the fossil assemblage include disappearance of colonial favositid and rugose corals and entry of tentaculitids in the upper 2.5 m of Hume limestone (Fig. 7B). As best seen in the thin section, transition to the Bluefish Member is marked by decline of calcareous matrix and rapid increase in tentaculitids to a rock-forming abundance. The basal few millimeters of the Bluefish Member black shale are rich in benthic fossils including brachiopod, bryozoan, and crinoidal debris (Fig. 9B). This basal coquina of the Bluefish Member is strongly compacted, which masks the possible development of burrowing patterns. However, the tentaculitid-rich shale 1.5 cm above the contact is already laminated with no clear sign of burrowing. An example of typical siliceous and calcareous shale of the lower Bluefish Member is shown on Figure 8.

CONCLUDING REMARKS

Combination of new XRD data from cores and the extensive XRD database collected by NTGS from cutting and outcrop samples (Pyle et al., 2014; Pyle and Gal, 2016) allows for robust mineralogical characterization of mudrock units (Fig. 5 and Table 1). These data show that the Canol Formation stands out as having the highest quartz (82% median) and the lowest clay mineral content (7% median). Clays in the HRG are dominated by illite. The Hare Indian and lower Canol formations are variably calcareous and dolomitic; the upper Canol is dolomitic and non-calcareous; basal Imperial shales are siderite-dolomite-bearing and non-calcareous.

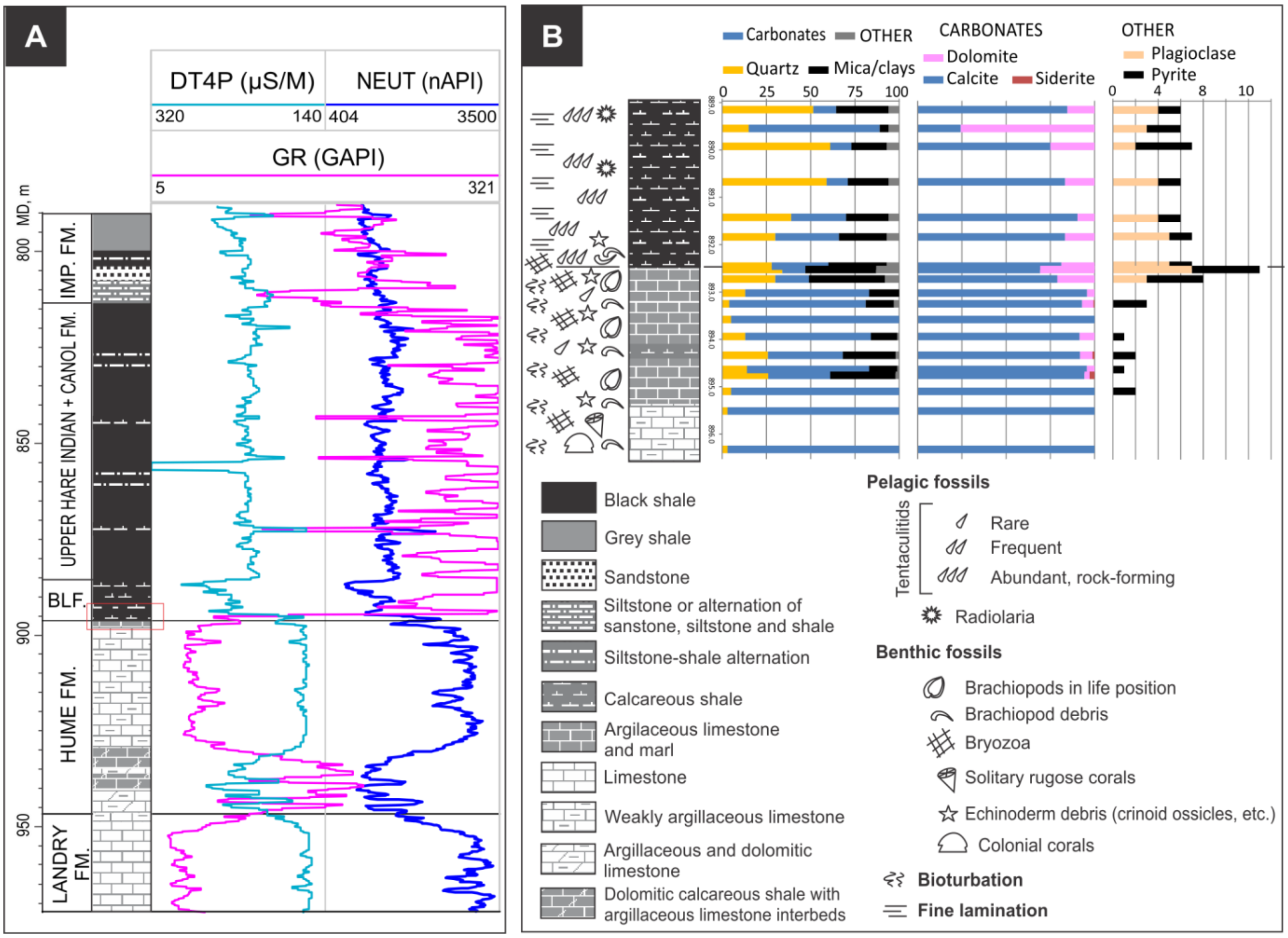

Figure 7. Hume-Canol interval in Kugaluk N-02 well: (A) Continuously cored section with well logs; red box indicates detailed section from (B); BLF. is Bluefish member; IMP. FM. is Imperial Formation. (B) Hume-Bluefish contact with XRD data, lithological and paleoecological information; calcite, dolomite and siderite are normalized to summed carbonates.

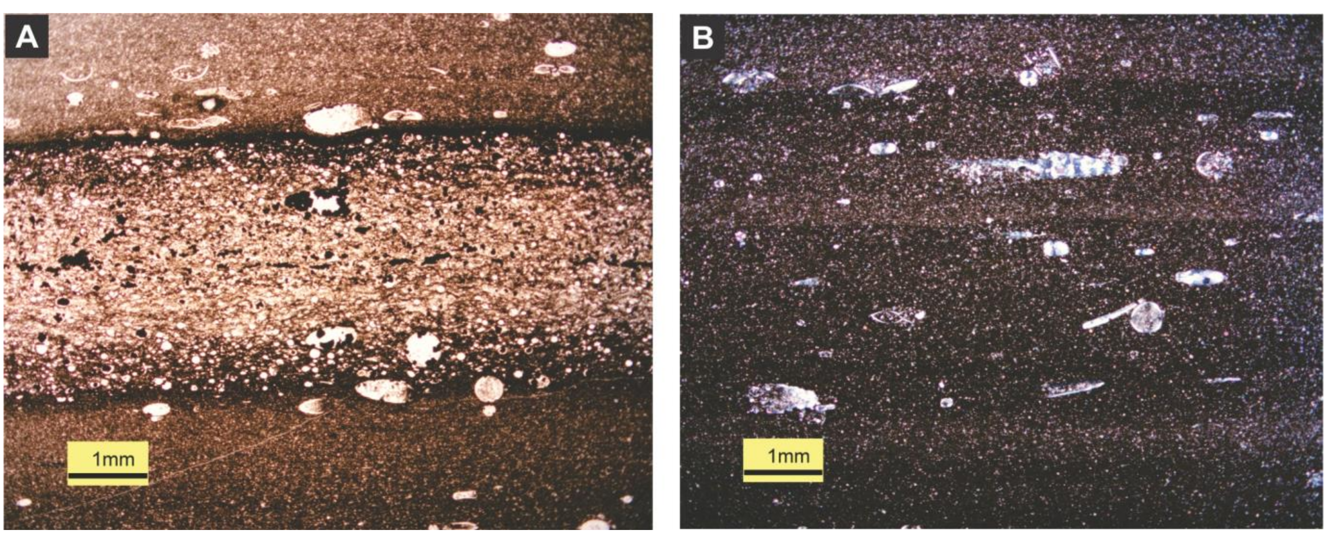

Figure 8. Black shale of the lower part of Bluefish Member in thin section, 891.2 m MD, GSC # C-605120. (A) A lamina of radiolarian chert and small tentaculitid cones, plane polarized light. (B) Laminated shale with variously collapsed tentaculitids; dust-like inclusions with high birefringence color are microcrystals of calcite and dolomite; crossed polarized light.

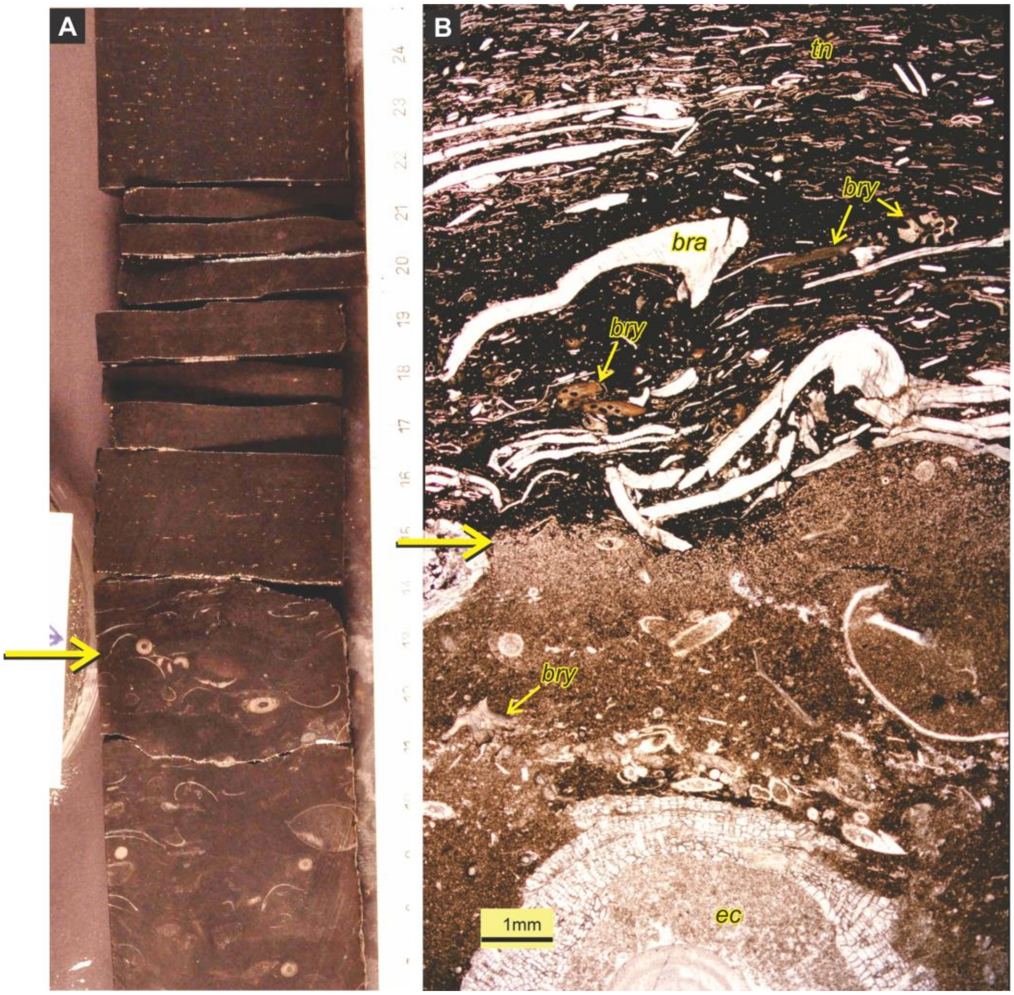

Figure 9. Hume-Bluefish contact (arrows): (A) on core face; (B) in thin section under plane polarized light, GSC # C-605121; note bryozoan-brachiopod coquina on top of bioturbated limestone; note mass collapsed dacryoconarid tentaculitids (*tn*) in the upper part of thin section; (*ec*) crinoid ossicle encrusted by a bryozoan; (*bra*) brachiopods; (*bry*) bryozoans (mostly fenestellids).

However, a caution on direct utilization of statistics (Table 1) is warranted, since this XRD dataset from the Horn River Group blends results of different labs produced with different approaches. These data, compiled in Pyle et al (2014), were produced at GSC Calgary (Pyle et al., 2011), Mineralogy Lab at GSC Ottawa (Pyle et al., 2011; Gal and Pyle, 2012; Pyle and Gal, 2013; Bilot and Percival, 2013), and Acme Labs in Vancouver, BC (Pyle and Gal, 2012). This issue will hopefully be addressed in near future with partial or complete reassessment of previously made results.

Twenty-nine XRD samples from Kugaluk N-02 well include 10 samples from the Landry Formation and 19 samples across the contact of Hume Formation and the Bluefish member of HRG. The latter subset, in combination with earlier core observations, demonstrates the conformable nature of this important stratigraphic surface and existence of a transitional limestone in the uppermost 3 m of the Hume Formation. This limestone is still rich in benthic fossils and bioturbated, but is enriched in quartz, muscovite mica (probably illite clay), and its uppermost part contains plagioclase grains. This observation corroborates the regionwide “drowning unconformity” nature of the Hume/HRG contact and lack of large-scale erosional truncation at that sharp surface.

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