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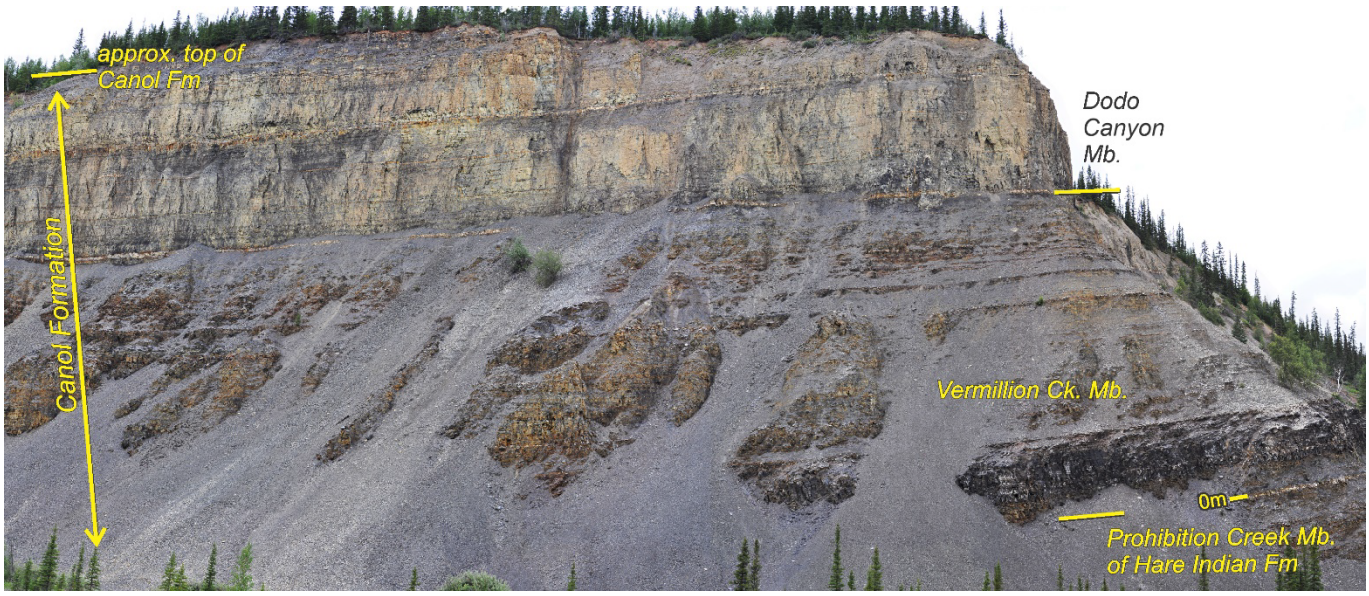

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Geological and geochemical data from Mackenzie corridor. Part XI: new geochemical, magnetic susceptibility, and X-ray diffraction data from the Horn River Group (Devonian) in cores and outcrops south of Norman Wells, Northwest Territories

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Cover photo: Canyon wall at Prohibition Creek, Norman Range, Northwest Territories. Taken in July 2015. NRCan Photo 2022-444

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

Appendices:

A. Nitrogen isotope ($\delta^{15}N_{tot}$) and %N data from Mirror Lake N-20 and Little Bear N-09 wells
B. Carbon and nitrogen isotope ($\delta^{13}C_{org}$ and $\delta^{15}N_{tot}$) and %CN data from Loon Creek O-06 well
C. Carbon isotope data ($\delta^{13}C_{org}$), %C data, and decarbonation report from Prohibition Creek composite section
D. X-ray diffraction data from Prohibition Creek composite section
E. Elemental data from Prohibition Creek composite section
F. HAWK pyrolysis-combustion data from Prohibition Creek composite section
G. HAWK dynamic pyrolysis reports and pyrograms from Prohibition Creek composite section
H. Bulk magnetic susceptibility data from Prohibition Creek composite section
I. Prohibition Creek composite section and cross-plots of select geochemical proxies

INTRODUCTION

The Horn River Group of the study area was in the focus of research attention over the last decade, fuelled by shale oil and gas prospectivity and shale hydrocarbon exploration in these strata during 2011-2014 (Enachescu et al., 2013; AANDC, 2014; NEB-NTGS, 2015). An inquiry into "what controlled deposition and preservation of hydrocarbon source rocks" was included in the list of scientific questions of GeoMapping for Energy and Minerals (GEM-II) Program of NRCan (2014-2020), and relevant research was funded within the GEM Mackenzie Project, focusing on the Devonian System of the Mackenzie River corridor that hosts a major total petroleum system of the inland Northwest Territories (excluding the Mackenzie Delta). Analytical work and data reported here are the follow-up of this research funded under "GEM Legacy" in 2020-2021 and partnering studies of petroleum systems within the ongoing Geoscience for New Energy Supplies (GNES) Program of NRCan.

Here we report data from Prohibition Creek outcrops and three continuously cored exploration wells (Fig. 1). The core samples from Little Bear N-09 (1735.3-1829.05 m) and Mirror Lake N-20 (1891.55-2052.53 m) were collected in 2018 at ~0.6-0.7 m stratigraphic intervals. Carbon isotope data from these two overlapping sections were reported previously (supplementary data table to Kabanov and Jiang, 2020), and nitrogen isotope data are reported here for the first time. Samples from Loon Creek O-06 (1788-1806.13 m) were collected in December 2020 with the goal to acquire $\delta^{13}C_{org}$ and $\delta^{15}N_{tot}$ logs across the contact of Hume and Hare Indian formation and the basal black shale of the Hare Indian Formation with highest possible sampling density (~0.3 m), as this stratigraphic interval is one of world's records of the Middle Devonian Kačák event (House, 1996; Kabanov and Gouwy, 2017: Uyeno et al., 2017; Gouwy, 2020).

The 2019 field work on Prohibition Creek was a joint effort of GSC-Calgary and the DEEP research group of the University of Liverpool (UK), aimed to remeasure and sample the Eifelian-Frasnian succession of the Hume Formation and the Horn River Group. The latter is well exposed along Prohibition Creek and recommended as a reference section for the Horn River Group in the region (Kabanov et al., 2019). This outcrop occurs in the zone of relatively low thermal maturity indicated in this particular section by average Tmax 430°C within 413-446°C range except one outlier (Appendix G) and conodont colour alteration index of 1.5-2.0 (GSC internal paleoreport SAG-2016-3). This relatively low thermal maturation implies that primary paleomagnetic, organic-matter, and geochemical signals are likely well preserved. Baseline descriptions, gamma spectrometry data, and correlation of measured sections (=field stations) were published earlier (Kabanov et al., 2019), and the composite section is provided here in Appendix I. In each field station, samples were collected mostly at ~0.5 m stratigraphic interval. We doubled the sampling density across the contact of the Vermillion Creek and Dodo Canyon members of the Canol Formation, which resulted in the highest resolution for this interval correlated globally as the basal *punctata*

isotopic event (Kabanov et al., in press a, b). Here we report the following data from Prohibition Creek samples: HAWK pyrolysis-combustion, whole-rock elemental geochemistry (including mercury concentration), stable carbon isotope analyses on organic matter ($\delta^{13}C_{org}$), mineralogy by x-ray diffraction analysis (XRD), and bulk magnetic susceptibility (see Figure 2 and Appendix I for cross-plots of select proxies).

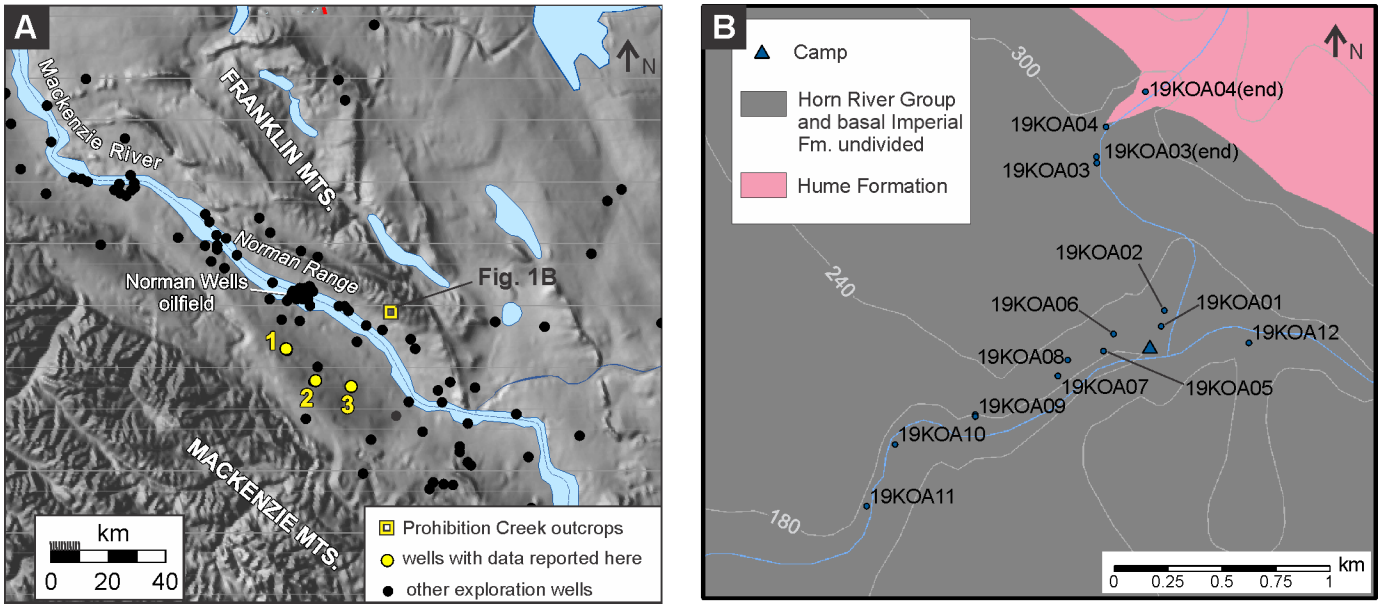

Figure 1. (A) Location of Prohibition Creek outcrops and cored well sections on the digital elevation model; wells: (1) Loon Creek O-06, (2) Mirror Lake N-20, (3) Little Bear N-09. **(B)** Field stations along Prohibition Creek from field work 2019 (Kabanov et al., 2019).

CARBON AND NITROGEN ISOTOPE ANALYSES

Sample preparation and analyses

Samples from 2018 (Mirror Lake N-20 and Little Bear N-09) were crushed in a clean lab at GSC Calgary, then split for stable nitrogen and carbon isotope analyses. Aliquots for carbon isotope study underwent acid treatment (with hydrochloric acid) to remove carbonate minerals. In 2020 the same preparation protocol was executed on Loon Creek O-06 Prohibition Creek samples at AGAT Laboratories Ltd. in Calgary.

Analyses were run at the Ján Veizer Stable Isotope Laboratory of the University of Ottawa. The lab job orders were B2021-246 and B2021-247 for 2020/21 batch; B1819-171, 172, 173, 174, 352, 353, 354, 355, 414, 415 for samples processed in 2018. Part of samples from Mirror Lake N-20 were reprocessed in 2022 under job order B2022-044 after identification of artificial positive offset in $\delta^{15}N$ values in 2018 results. Only new B2022-044 results that show consistency are reported herein. Samples and standards were weighed into tin capsules and loaded into an elemental analyser interfaced to an isotope ratio mass spectrometer. Sample (or standard) was flash combusted at about 1800°C (Dumas combustion), and the resultant gas products carried by helium through columns of oxidizing/reducing chemicals were optimised for CO_2 and N_2. The gases are separated by a "purge and trap" adsorption column and sent to the Isotope Ratio Mass Spectrometer (IRMS) interface, then to IRMS.

Accuracy and precision

The internal standards of Ján Veizer Laboratory for $\delta^{15}N$ and $\delta^{13}C$ are: C-51 Nicotiamide (0.07‰ and -22.95‰ respectively), C-52 mix of ammonium sulphate + sucrose (16.58‰ and -11.94‰), C-54 caffeine (-16.61‰ and -34.46‰), blind standard C-55 or glutamic acid (-3.98‰ and -28.53‰). The data are reported in Delta notation δ, the units are per mil (‰) and defined as $d = ((R_x-R_{std}))/R_{std})*1000$ where R is the ratio of the abundance of the heavy to the light isotope, x denotes sample and std is an abbreviation for standard. All $\delta^{15}N$ is reported as ‰ vs. AIR and normalized to internal standards calibrated to International standards IAEA-N1 (+0.4‰), IAEA-N2 (+20.3‰), USGS-40 (-4.52‰) and USGS-41(47.57‰). All $\delta^{13}C$ is reported as ‰ in reference to Vienna Pee-

Dee Belemnite (V-PDB) and normalized to internal standards calibrated to International standards IAEA-CH-6 (-10.4‰), NBS-22 (-29.91‰), USGS-40 (-26.24‰) and USGS-41 (37.76‰). Note that the PDB and V-PDB scales are identical and interchangeable. The lab analytical precision is based on the internal standard (C-55) which is not used for calibration and is usually better than 0.2 ‰ and is found at the end of the data columns in the 'final report' tab.

Instrumentation

The Ján Veizer Laboratory uses the Vario EL Cube elemental analyser manufactured by Elementar, Germany. The interface is Conflo IV manufactured by Thermo, Germany. The IRMS system is Delta Advantage manufactured by Thermo, Germany. After %NC determination (total N and C wt. %), organic material samples are prepared on glass fibre filters (or quartz fibre) in one of the following ways:

1) If the whole filter is needed, it is carefully cut up into small pieces in order to be ground up in a mortar and pestle. The ground material is pressed into a tin capsule along with a small scoop of tungsten oxide catalyst (WO_3). Both materials are mixed in the capsule before sealing.

2) If only a portion of the filter is needed, 1 or more punches are pressed into a tin capsule with a small scoop of WO_3. The diameter of a single punch is 6.2mm.

MAGNETIC SUSCEPTIBILITY

Mass-normalised bulk susceptibility on rock chips was measured at room temperature using an MFK-1 Kappabridge at the Geomagnetic Laboratory of the University of Liverpool, UK. The reported bulk susceptibilities are an average of 3 measurements.

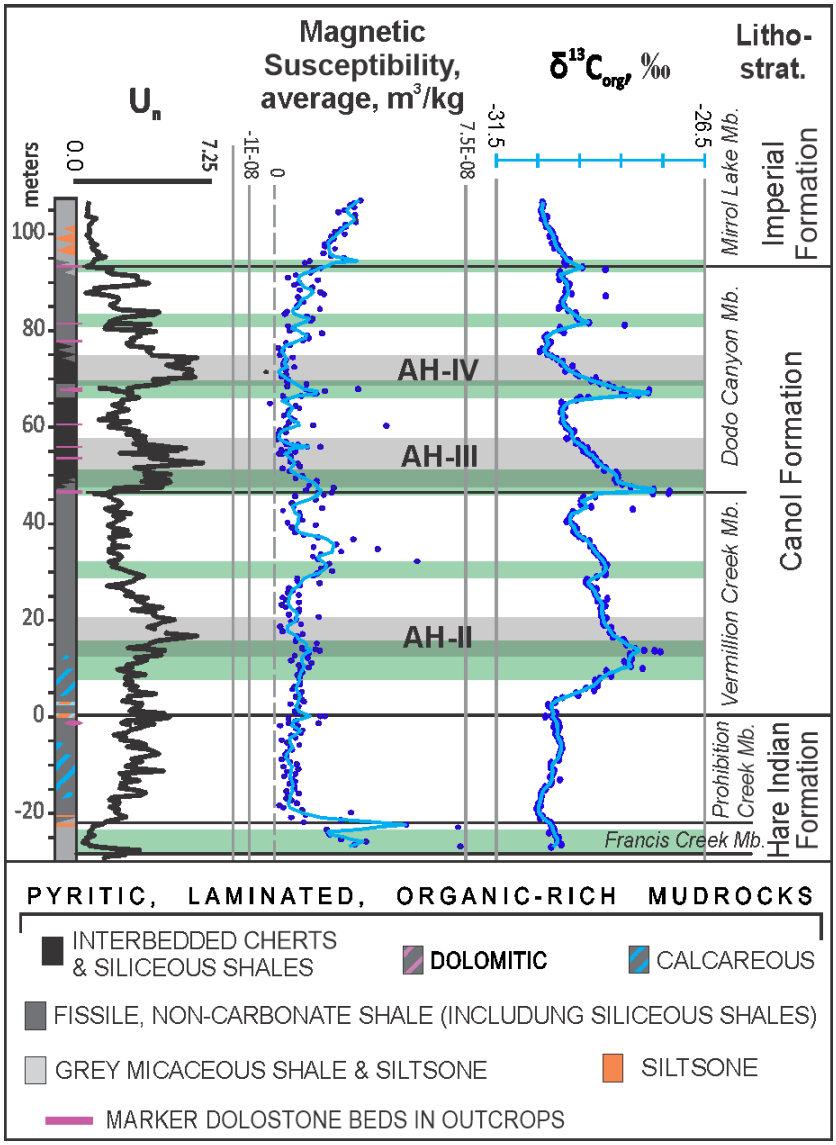

Figure 2. Magnetic susceptibility of the Prohibition Creek composite section plotted on the event stratigraphy framework. Carbon isotopic excursions are traced with green bars, anoxic horisons with grey bars. U_n is gamma spectrometry proxy to authigenic uranium enrichment (U_n = 0.29*U[ppm]*K[%]$^{-1}$). Data smoothers (light blue curves) are LOWESS regression with sliding window 2.5%. See Kabanov et al. (in press, a) for details.

ELEMENTAL GEOCHEMISTRY

Multi-acid digestion ICP-ES/MS

The Prohibition Creek samples were analyzed for elemental concentration at Bureau Veritas Analytical Laboratories in Vancouver, BC. The multi-acid digestion protocol MA250 is an ultra-trace ICP-ES/MS technique reporting 59 elements with detection limit mostly ≤ 0.1 ppm. A 0.25 g split is heated in HNO_3, $HClO_4$ and HF to

fuming and taken to dryness. The residue is made to volume with diluted HCl and analyzed by the ICP-ES/MS. This four-acid digestion allows for more complete release of chalcophyle elements than in the previously used aqua regia solution (AQ200). A minor limitation in accuracy is due to the fact that multi-acid digestion under this protocol is partial for some Cr- and Ba- containing minerals and oxides of Al, Fe, Hf, Mn, Sn, Ta, Zr and REEs. Volatilization during fuming may result in loss of As, S, Se and Sb.

Elemental mercury

Elemental Hg data were acquired with the cold vapor method (Hall and Pelchat, 1997) at Bureau Veritas using LECO AMA254 Mercury Analyzer (CV402 lab code). The AMA254 is an atomic absorption spectrometer designed to determine total mercury content in samples. The instrument's operation may be separated into three phases during analysis: decomposition, collection, and detection. Decomposition phase involves heating a nominal amount of analyte in a combustion tube to ~750°C, leading to thermal decomposition into gaseous form. The evolved gas is then transported (via an oxygen carrier gas) to the other side of the combustion tube. This portion of the tube, pre-packed with specific catalytic compounds, represents the area in the instrument where all interfering impurities (*i.e.* ash, moisture, halogens, and minerals) are removed from the evolved gases. Following decomposition, the evolved gas is transported to the amalgamator for the Collection phase. The amalgamator, a small glass tube containing gold-plated ceramics, collects all of the mercury in the vapor. With a strong affinity for mercury and a significantly lower temperature than the decomposition phase, the amalgamator is capable of trapping all mercury for subsequent detection. When all mercury has been collected from the evolved gases, the amalgamator is heated to ~900 °C, thus releasing all mercury vapor to the detection system. The released mercury vapor is transported to the final, detection phase. During the detection, vapor passes through two sections a cuvette. The cuvette is positioned in the path length of a standard Atomic Absorption Spectrometer. This spectrometer uses an element-specific lamp that emits light at a wavelength of 253.7 nm, and a silicon UV diode detector for mercury quantitation.

X-RAY DIFFRACTION AND MINERALOGY

X-ray Diffraction Analysis

The mineralogy of bulk powdered materials and clay-size separates were determined by XRD analysis. Bulk samples were pulverized using a McCrone mill in isopropyl alcohol until a grain size of about 5- 10 µm is obtained (~ 5 minutes). The samples were air-dried and then back pressed into an aluminum holder to produce a randomly-oriented specimen. For clay-size separates, 40 mg were suspended in distilled water and pipetted onto glass slides and air-dried overnight to produce oriented mounts. X-ray patterns of the pressed powders or air-dried samples are recorded on a Bruker D8 Advance Powder Diffractometer equipped with a Lynx-Eye Detector, Co Kα radiation set at 35 kV and 35 mA.

Mineral Identification and Quantitative Analysis

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) and other available databases. Quantitative analysis was completed using TOPAS (Bruker AXS, Inc.) software, a PC-based program that performs Rietveld refinement (RR) of XRD spectra, based on a whole pattern fitting algorithm. The program relies on having mineralogical structure files (.cif) such that the reference minerals are as close a match to the unknown as possible. The goodness of fit shows the how well the reference minerals match the unknowns. Results are reported in wt %.

Quantitative analyses appear reasonable when minerals in the samples can be matched to the standards. The lower the Goodness of Fit (GoF) value, the closer the standards match the unknowns and the better the results. Difficulty arises when clay minerals of varying composition (e.g., expandable layers, mixed-layers) are encountered, or when mineral species have overlapping X-ray peaks (e.g., kaolinite and chlorite; quartz and

graphite). Also, there are a limited number of reference minerals available as structure files; these may not be an exact match to the mineral being analyzed (e.g., using actinolite rather than ferro-hornblende). Differences in estimation will also arise if using a pressed powder (preferable for RR) vs. smear (oriented) samples. Smear samples can be used if insufficient material is available. Occasionally smear mounts are made in order to better identify clay minerals that are found in minor to trace amounts, as the orientation enhances the 00l peaks relative to the hkl peaks.

Mineral composition of Horn River Group at Prohibition Creek

As exemplified by diffractograms on Figures 3-13, the samples from the Prohibition Creek section are generally dominated by quartz with minor muscovite mica. Rare samples are carbonate-rich and mostly dominated by dolomite rather than calcite. Minor to trace amounts of plagioclase feldspar, K-feldspar, kaolinite, mixed-layer clay minerals, barite, gypsum, jarosite, hematite, marcasite and pyrite can be found in some to many of the samples.

Figure 14 shows a ternary plot of the total carbonates, silicates and clay minerals. The samples are mostly siliceous with a few trending towards the calcareous field. None are very argillaceous, a maximum of about 35 wt. %. Consistently with field observations (Kabanov et al., 2019), Figure 14 highlights robust lithologic differences between the three stratigraphic subsets: the Vermillion Creek Member is relatively rich in carbonates, the Dodo Canyon Member is heavily dominated by quartz-rich facies (cherts), whereas clay content is the highest in the basal shales of the Imperial Formation.

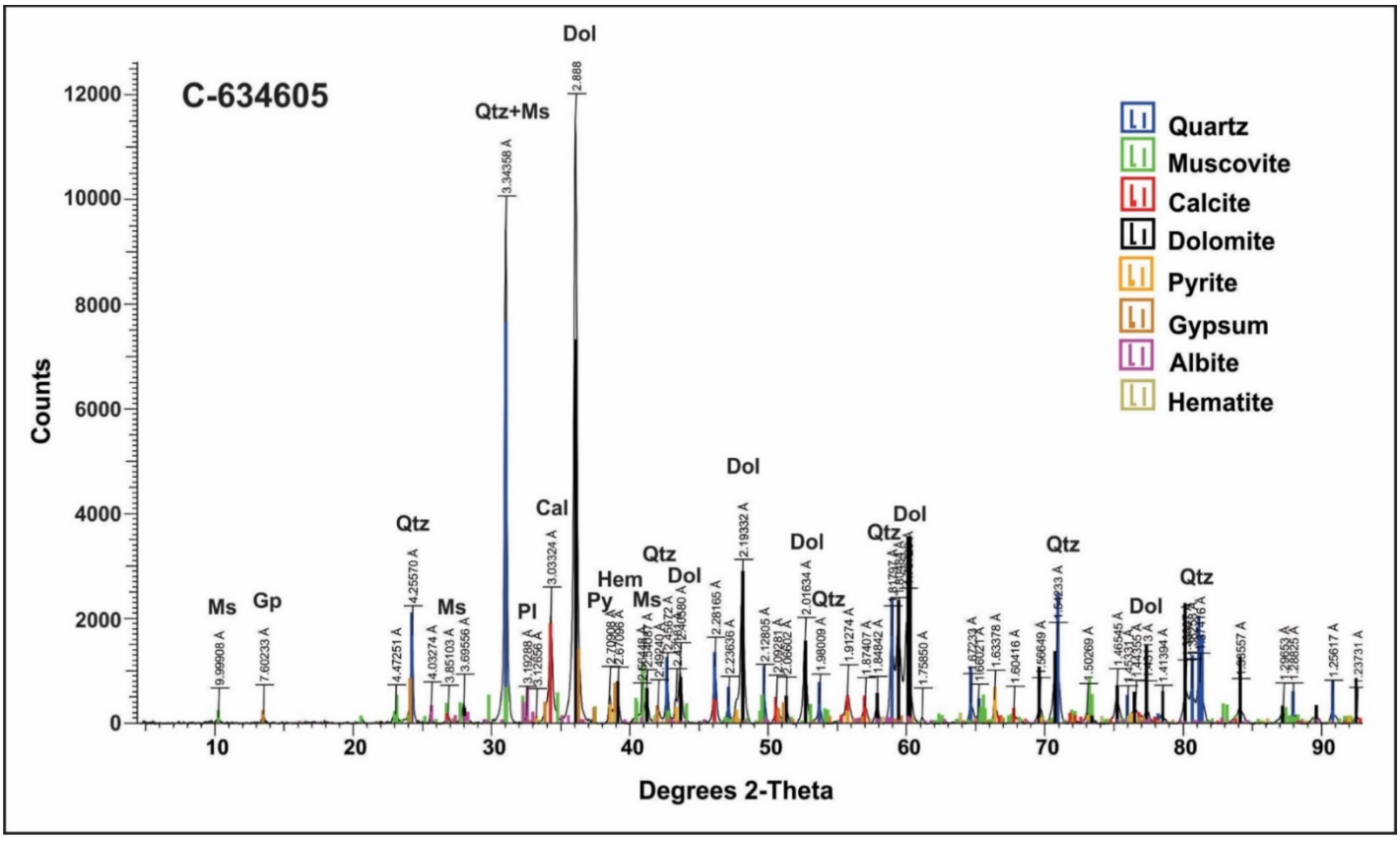

Figure 3. Results of Search/Match by EVA (Bruker AXS, Inc.) software for sample C-634605 from Field Station 19KOA-002. Major X-ray peaks are labelled.

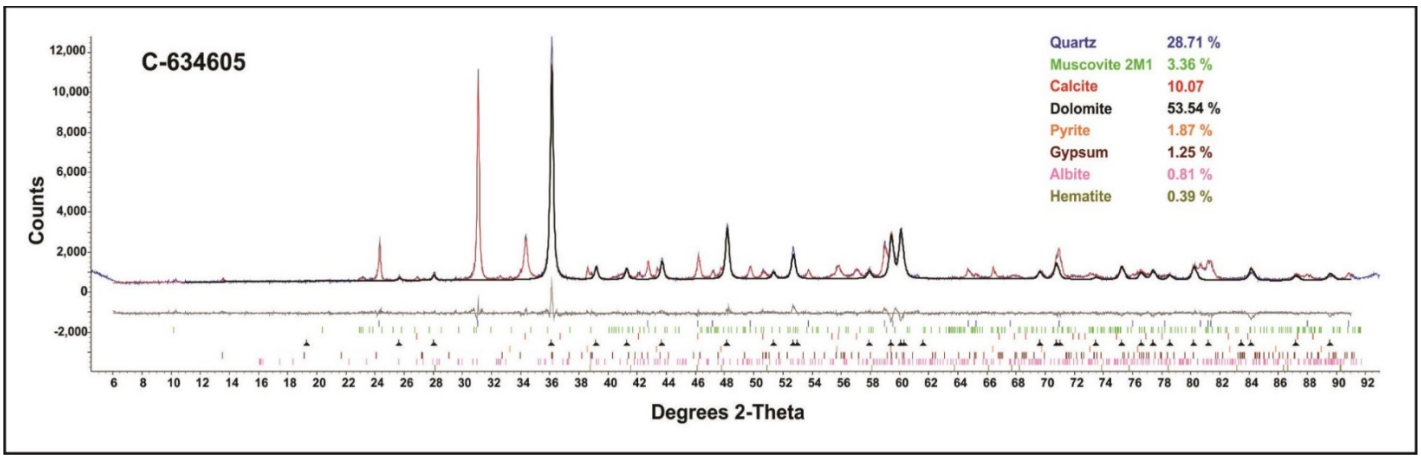

Figure 4. Result of RR analysis (TOPAS software; Bruker AXS, Inc.) for sample C-634605 from Field Station 19KOA-002. Note the black diffractogram overlay is for dolomite. The residual (grey line below) shows the goodness of fit match between reference minerals and the sample.

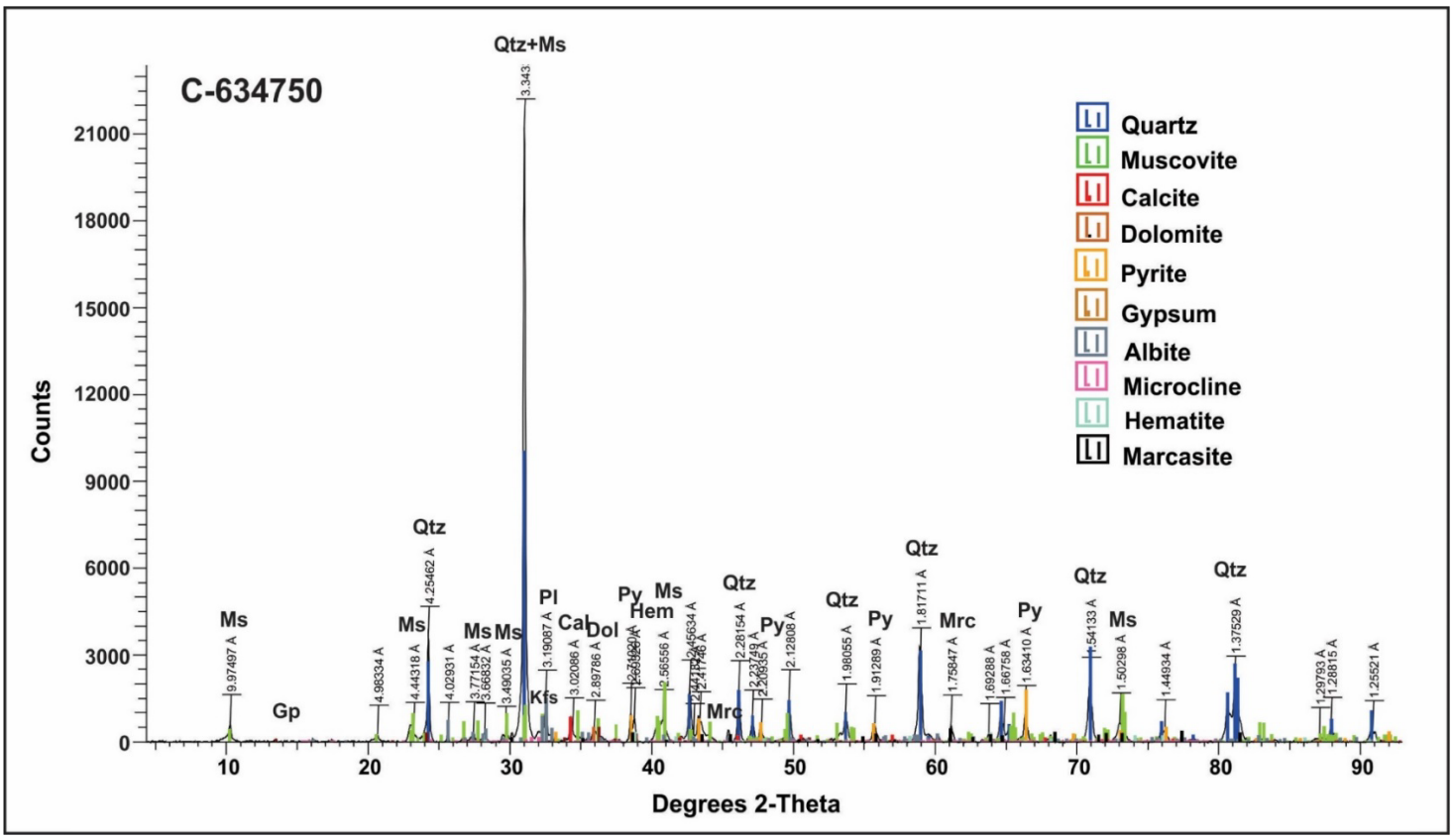

Figure 5. Results of Search/Match by EVA (Bruker AXS, Inc.) software for sample C-634750 from Field Station 19KOA-007. Major X-ray peaks are labelled.

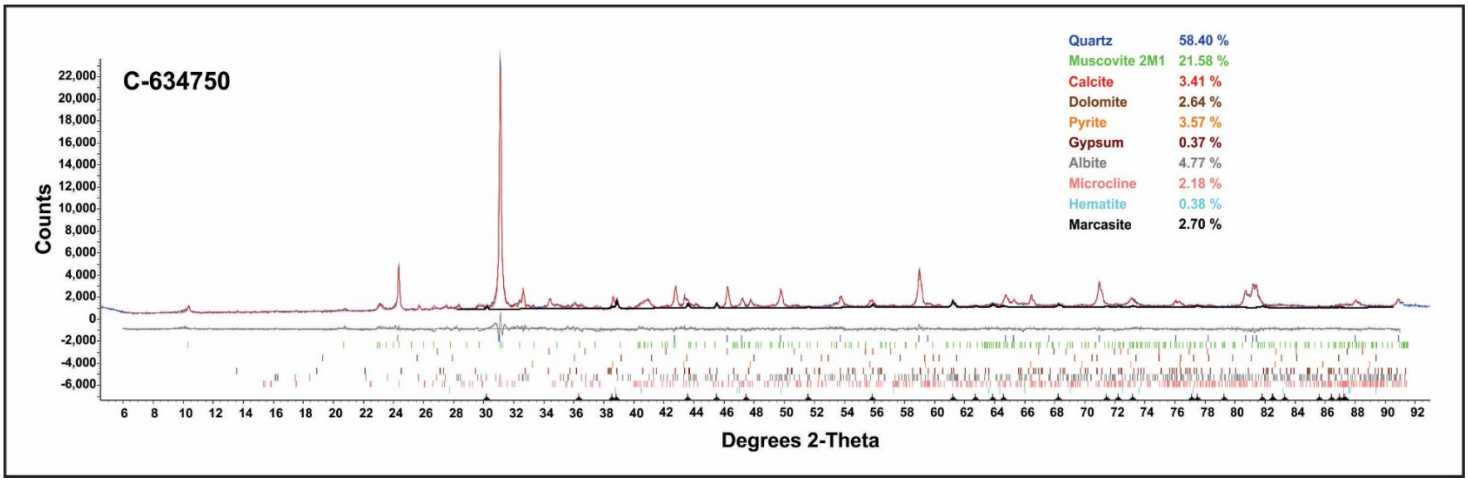

Figure 6. Result of RR analysis (TOPAS software; Bruker AXS, Inc.) for sample C-634750 from Field Station 19KOA-007. Note the black diffractogram overlay is for marcasite. The residual (grey line below) shows the goodness of fit match between reference minerals and the sample.

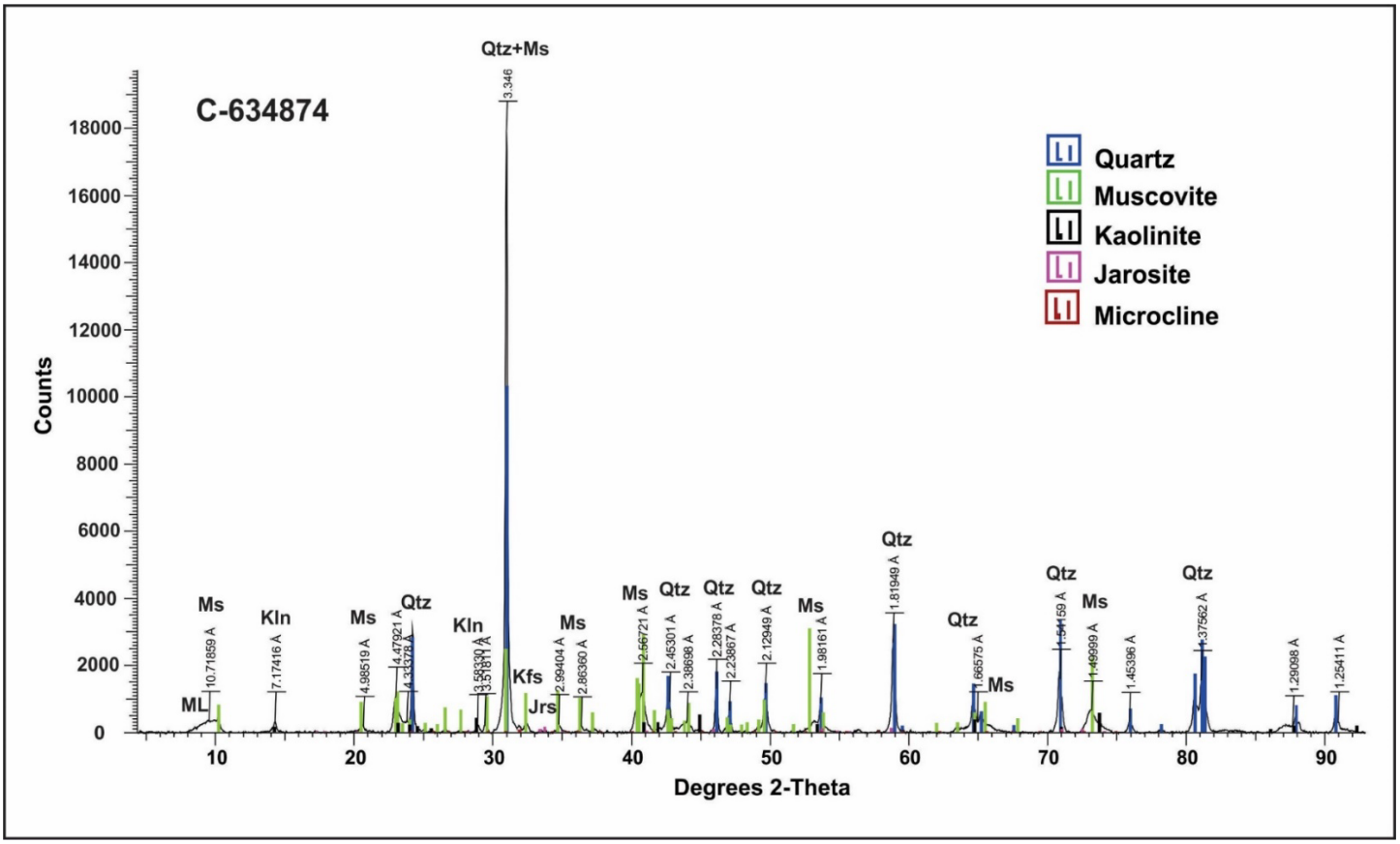

Figure 7. Results of Search/Match by EVA (Bruker AXS, Inc.) software for sample C-634874 from Field Station 19KOA-010. Major X-ray peaks are labelled.

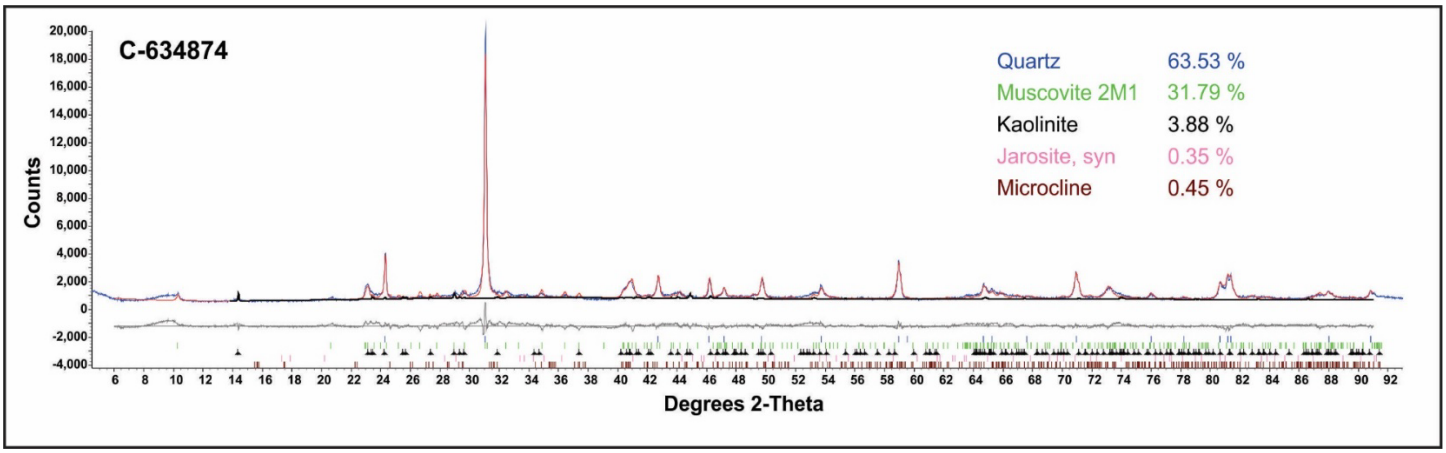

Figure 8. Result of RR analysis (TOPAS software; Bruker AXS, Inc.) for sample C-634874 from Field Station 19KOA-010. Note the black diffractogram overlay is for kaolinite. The residual (grey line below) shows the goodness of fit match between reference minerals and the sample.

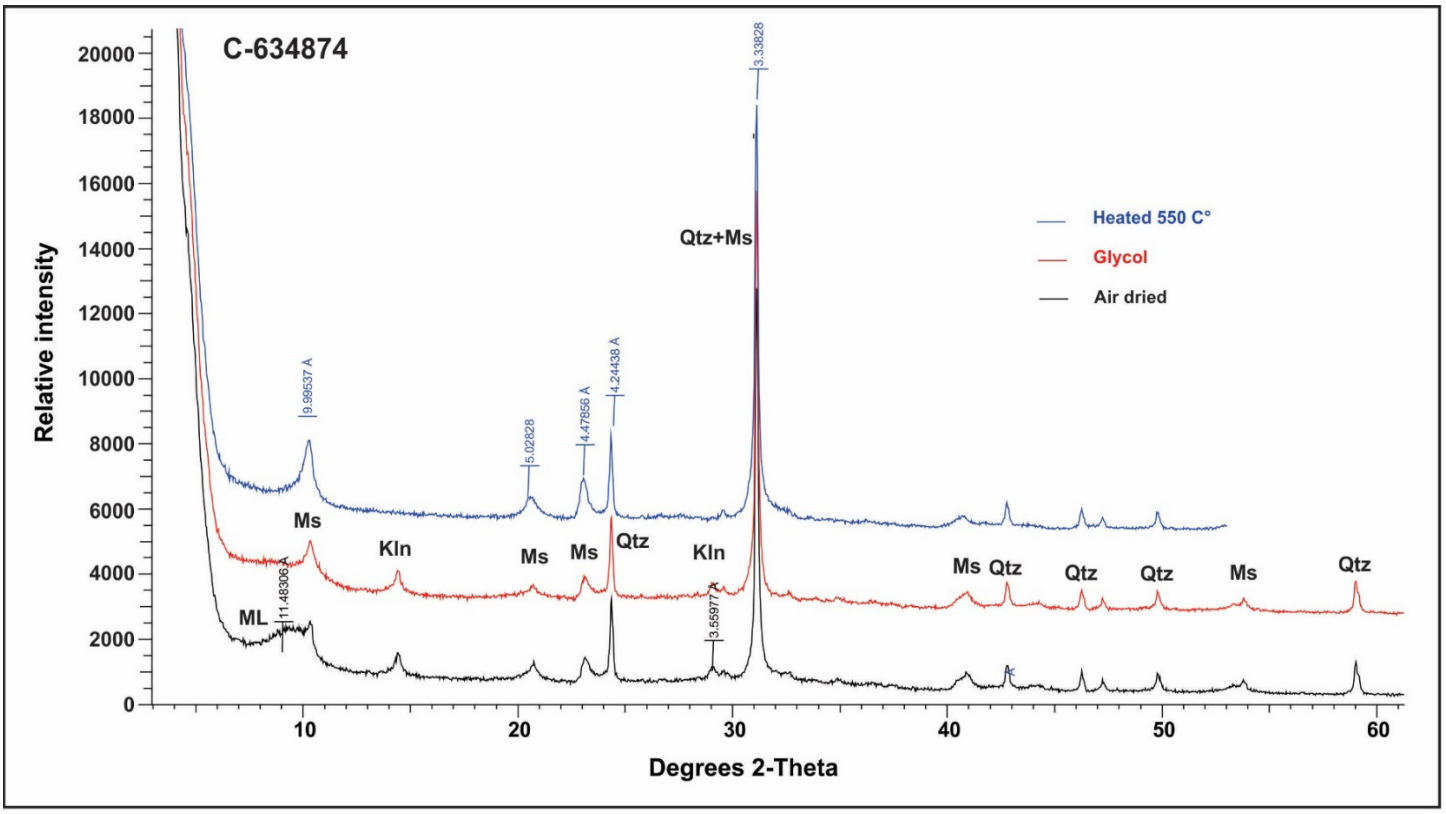

Figure 9. Stacked diffractograms for air-dried, glycol-saturated and heat treated mounts of sample C-634874 from Field Station 19KOA-010. Major-X-ray peaks are labelled. Note the shoulder on the low angle sides of the muscovite-mica (MS) peaks indicates the presence of a mixed-layer clay mineral (ML).

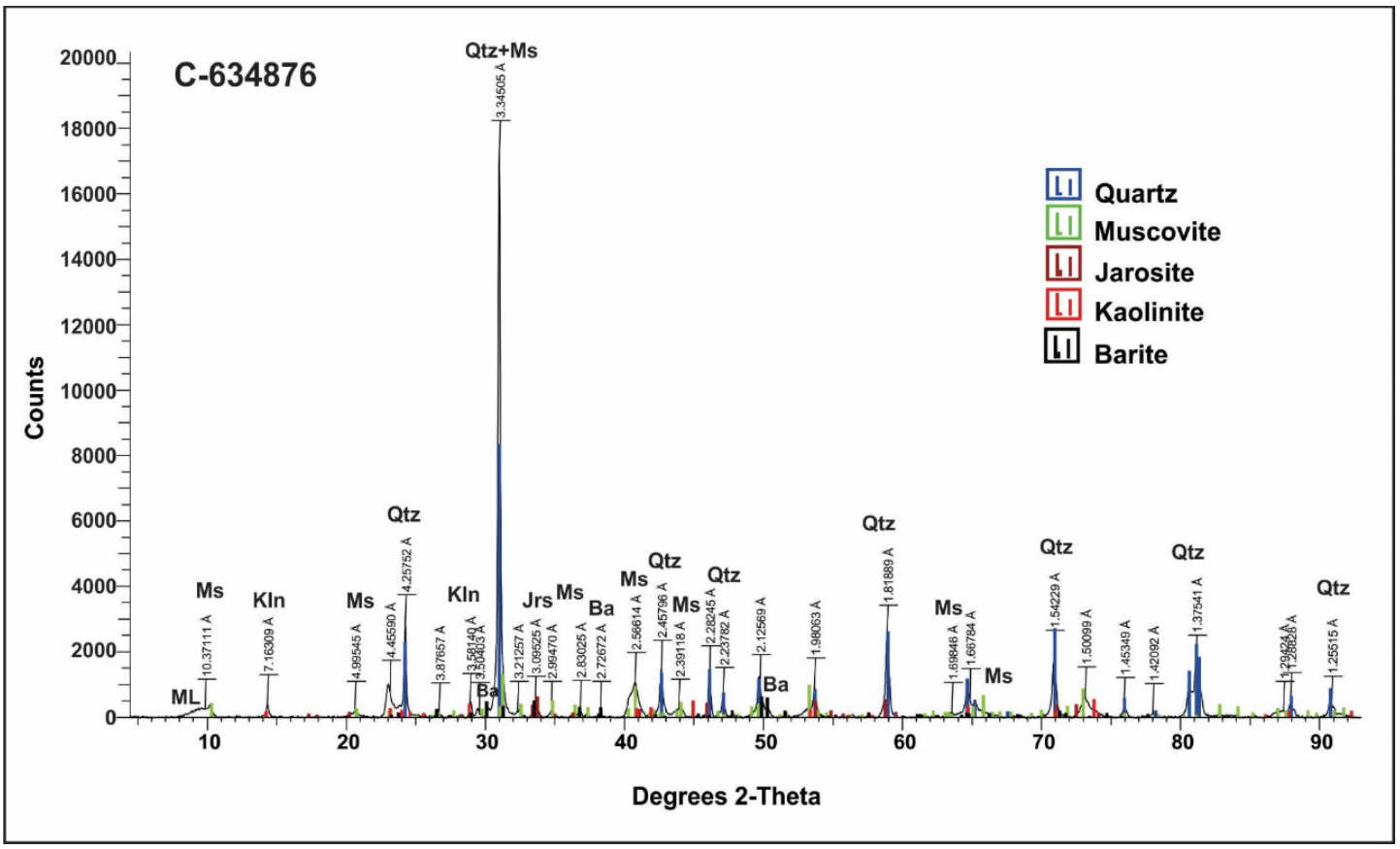

Figure 10. Results of Search/Match by EVA (Bruker AXS, Inc.) software for sample C-634876 from Field Station 19KOA-010. Major X-ray peaks are labelled.

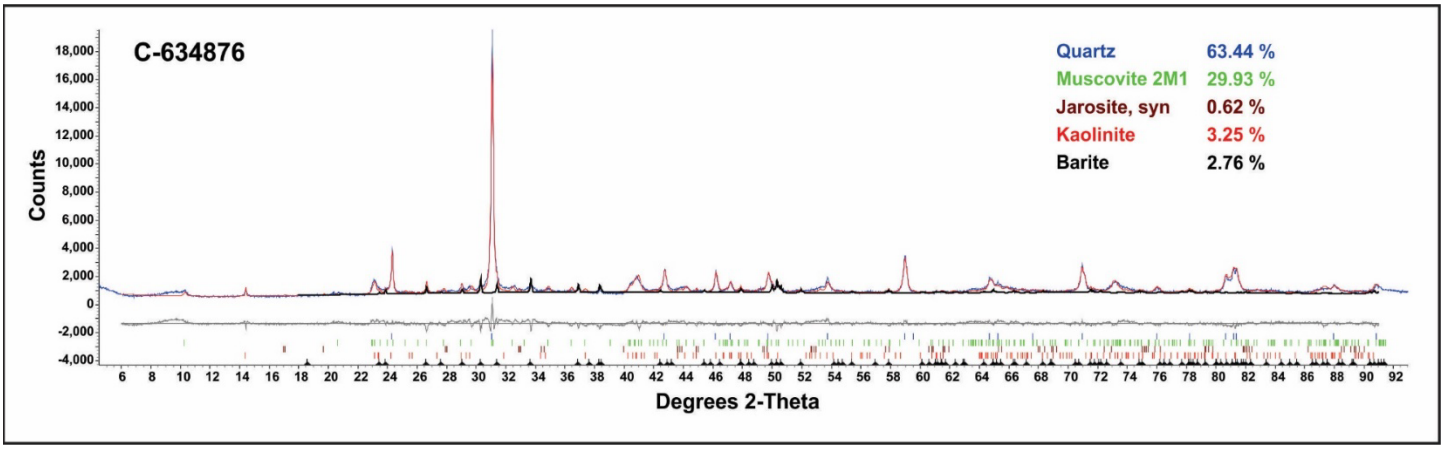

Figure 11. Result of RR analysis (TOPAS software; Bruker AXS, Inc.) for sample C-634876 from Field Station 19KOA-010. Note the black diffractogram overlay is for barite. The residual (grey line below) shows the goodness of fit match between reference minerals and the sample.

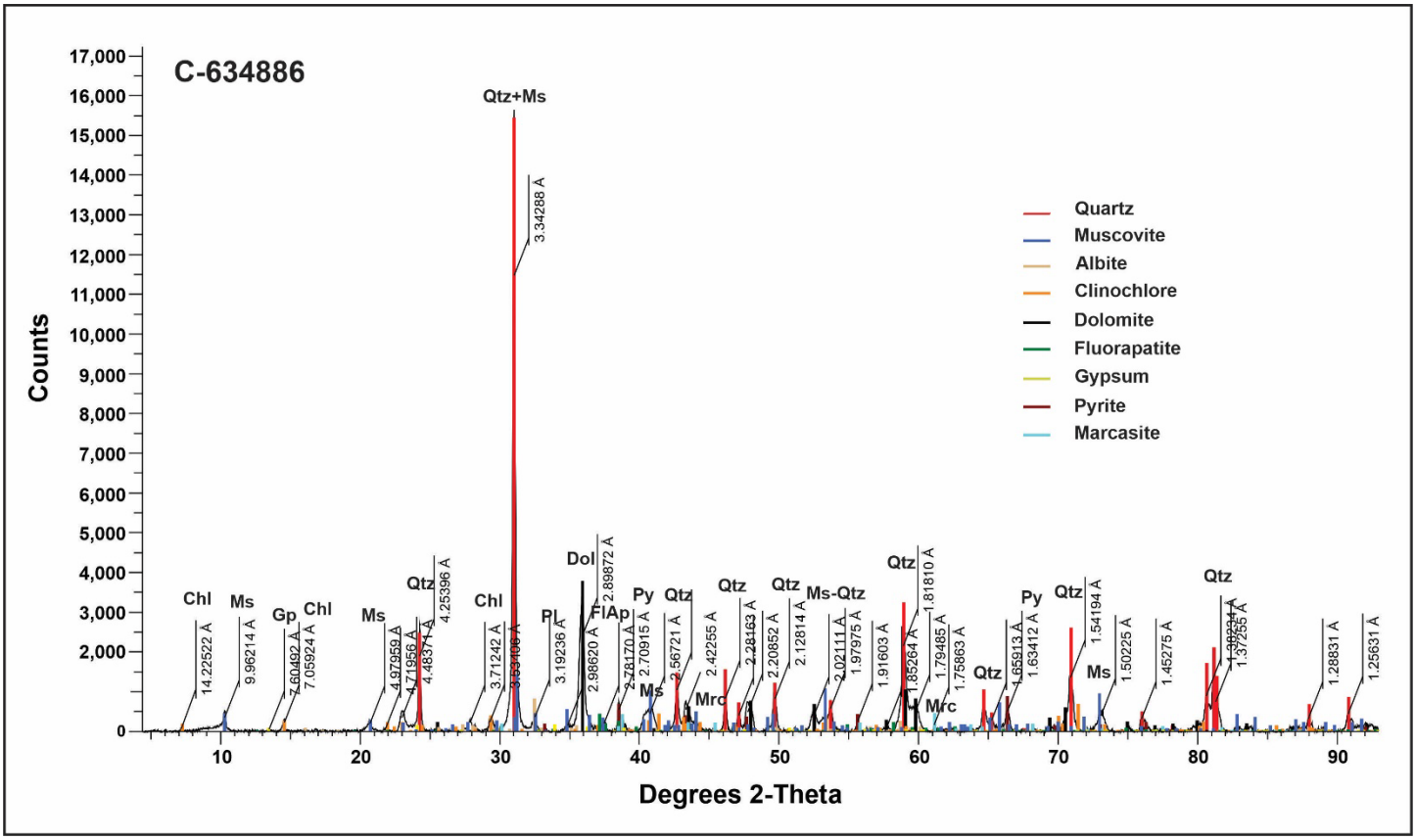

Figure 12. Results for search/Match based on EVA (Bruker AXS, Inc.) software for sample C-634886, Field Station 19KOA-011. Major peaks are labelled. Note shoulder on the ~ 10 Å mica peak indicates slight trace of a mixed-layer clay mineral.

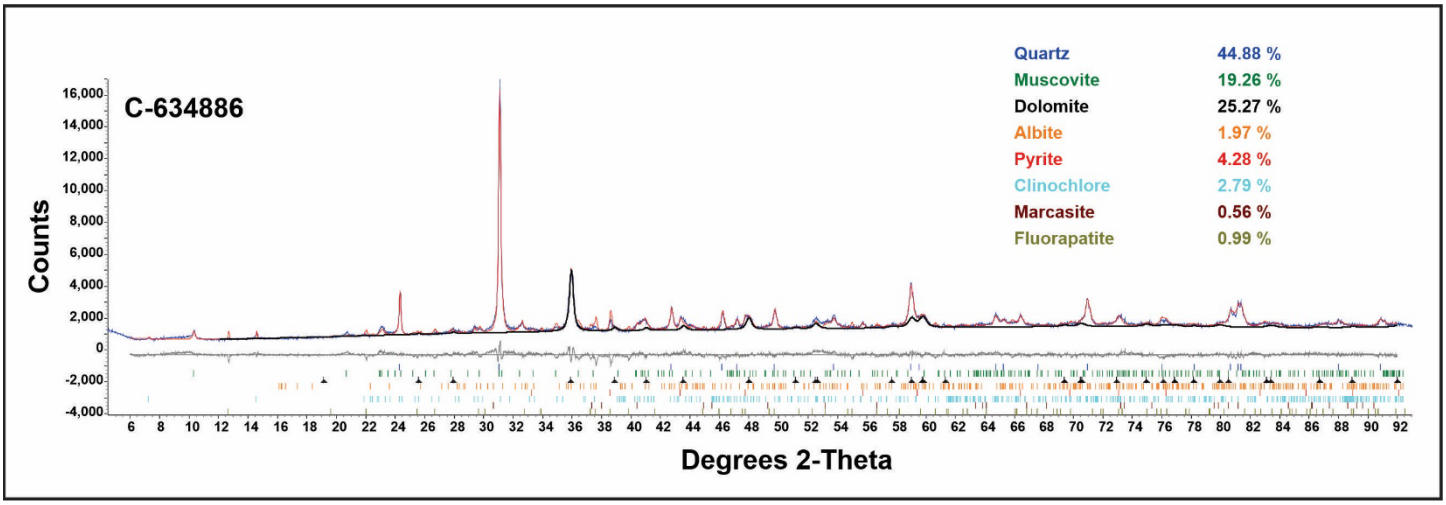

Figure 13. Quantitative results of sample C-634886 (, Field Station 19KOA-011) based on Rietveld refinement analysis using TOPAS (Bruker AXS, Inc.) software. Trace for dolomite overlays the diffractogram and the grey line below is the residual, an indication of the goodness of fit.

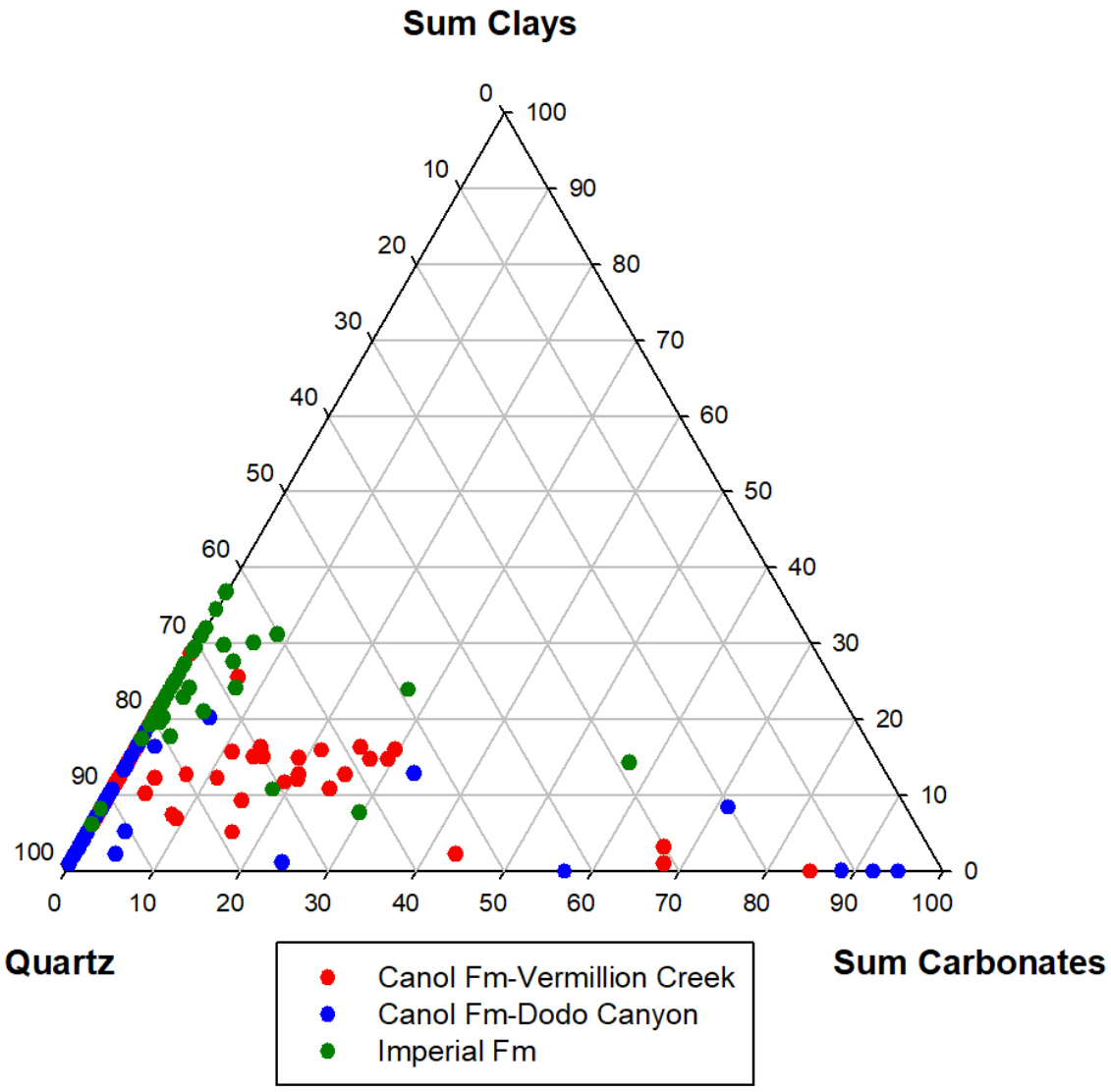

Figure 14. Ternary plot showing the sum of silicates (Qtz+Pl+Kfs), carbonates (Cal+Dol) and clay minerals (Ms+Chl+Kln+ML) for samples from the Canol Formation (Vermillion Creek Member and Dodo Canyon Member subsets) and the basal Imperial Formation (field stations 19KOA-10 and 19KOA-11 on Figure 1).

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