

**Mineralogical Characterization of Selected Samples from the Tanglefoot
Formation, Laberge Group, Whitehorse Trough**

Contribution to the Yukon Geological Survey

**QUANTITATIVE X-RAY
DIFFRACTION ANALYSES**

By

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For

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X-ray Diffraction Analysis

The mineralogy of bulk materials and clay-size separates is determined by X-ray powder diffraction analysis (XRD). Bulk samples are micronized using a McCrone mill in isopropyl alcohol or distilled water until a grain size of about 5- 10 μm is obtained. The samples are dried and then back pressed into an aluminum holder to produce a randomly oriented specimen. For clay-size separates, 40 mg are suspended in distilled water and are 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 K α radiation set at 40 kV and 40 mA. The samples are also X-rayed following saturation with ethylene glycol and heat treatment (550 EC).

Treatments:

air-dried:	2-86 E2 \oplus
glycolation:	2-86 E2 \oplus
heat (550 EC for 2 hours):	2-35 E2 \oplus

Mineral Identification and Quantitative Analysis

Initial identification of minerals is 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 is carried out using TOPAS (Bruker AXS Inc.), a PC-based program that performs Rietveld refinement (RR) of XRD spectra. This is based on a whole pattern fitting algorithm. It relies on having particular mineralogical structure files (.cif) such that the reference minerals are as close a match to the unknown as possible.

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.

Results

The objectives of this study are to characterize the lithology in Tanglefoot Formation and determine if it is a suitable source for conventional hydrocarbons. The Tanglewood Formation is located within the Whitehorse Trough in south-central Yukon, south of the Tintina fault. This is considered a frontier intermontane basin with strata unconformably overlying the Stikine terrane (Lewes River arc), and also the Quesnel terrane locally. The 2.44 million hectare basin straddles the Yukon-British Columbia border, with its northernmost margin in the Carmacks area. Its geology is characterized by an approximately 3000 m thick deformed Jurassic sedimentary succession (the Laberge Group), underlain by a depositional basement of Triassic sediments (the Lewes River Group), and capped by Cretaceous and Neogene volcanic rocks (Hutchison, Pers. Comm. 2016).

Samples were collected along the Klondike and Robert Campbell highways to the south and east of Carmacks, and Red Ridge or Teslin Creek near Division Mountain. Samples include immature sandstone. The goal of this study is to document the mineralogy of the sandstone samples to aid in interpreting their porosity and texture and their potential as a conventional reservoir system.

In all, 17 samples were submitted for XRD analyses. Their location, depth and lithology are given in Table 1. The quantitative mineralogy (wt.%) is given in Table 2 and Table 3 summarizes the basic statistics for each sample suite with regard to the mineral phases present in amounts greater than trace. Samples all contain abundant to minor quartz, plagioclase feldspar and calcite. Quartz ranges from 2 to 63 wt.% (mean=33 wt.%) and plagioclase feldspar from 5 to 57 wt.% (mean= 28 wt.%). For the samples containing lower quantities of quartz, calcite (and dolomite in one sample) becomes more dominant, ranging from 1 to 46 wt.% (mean=12 wt.%). Most samples contain minor muscovite, chlorite and K-feldspar. Many of the samples also contain minor dolomite. Minor to trace kaolinite, amphibole, clinopyroxene, heulandite and hematite occur in several samples. A few samples also contain minor to trace talc, barite, gypsum, analcime, pyrite and a mixed-layer clay mineral. More details regarding mineralogy is provided below subdivided into their respective suites.

Suite 14-MH-010 (North Klondike Highway)

These four samples collected from the North Klondike Highway represent a section from the surface to 11.5 m (Table 1) and all are described as sandstone. The quartz content decreases with depth from 63 to 29 wt.%, although the sample from 4.2 m does have less quartz (43 wt.%) than the sample at 8.7 m (59 wt.%) (Table 2). The two samples with the lower quartz content have increased dolomite content (29 wt.% at 4.2 m and 47 wt.% at 11.5 m). All four samples contain minor plagioclase feldspar, muscovite, chlorite, kaolinite and calcite. One sample contains minor K-feldspar. The goodness of fit for this suite is very good (Table 2). These are the only samples in the larger group that contain kaolinite. A typical diffractogram for these samples is shown in Figure 1 for the sample at 4.2 m. The diffractogram shows the colour coded match between the reference minerals and the unknowns. Note that the chemical composition of some minerals needs to be verified through microprobe analyses, as these matches are based on mineral structure not chemistry. Figure 2 shows

the results from the Rietveld refinement analyses using the Bruker software TOPAS. The black spectrum shows the match for dolomite and the grey is the residual, which provides a goodness of fit between the selected reference minerals and the sample. Figure 3 shows the variation in mineralogy with depth plotted as stacked bars. Note the changes in quartz and dolomite content are more readily visible in this format.

Suite 14-MH-013 (Eagle's Nest Bluff)

Six samples were collected from the Eagle's Nest Bluff locale between 19.5 and 89.9 m. These are all described as sandstone (Table 1) but the presence of plagioclase feldspar, amphibole, clinopyroxene and possible olivine point to an alkali basalt type lithology (Table 2). This suite of samples has a complex mineralogy. They contain abundant to minor plagioclase feldspar (17-57 wt.%, mean=38 wt.%) and minor quartz (2-17 wt.%, mean=10 wt.%). Calcite is abundant in one sample (46 wt.%) and minor in four others and absent in the sample from 30.5 m. The samples contain minor amphibole (mean=4 wt.%), clinopyroxene (mean=10 wt.%), chlorite (mean=5 wt.%), hematite (mean= 6.3 wt.%), heulandite (7 wt.%), analcime (2.8 wt.%) and K-feldspar (5 wt.%). A few samples contain minor to trace talc, dolomite, barite and possibly olivine (although many X-ray peaks are overlapping, this should be verified in thin section). There is a trace amount of mixed-layer clay minerals associated with the chlorite. Muscovite appears to be absent. The goodness of fit is good.

Sample 14-MH-013 (30.5 m) is illustrated in Figure 4. The diffractogram and the colour coded mineral comparison shows the complexity of this sample. This sample was also analysed as a smear mount to examine the clay mineral(s) in more detail. From Figure 5, the shift of the low angle chlorite X-ray peak at 14.5 Å after glycol saturation indicates the presence of smectite or a chlorite-smectite mixed-layer clay mineral. The shift to about 12 Å following heat treatment suggests this is a mixed-layer mineral rather than discrete smectite or chlorite. For this suite of samples, more analyses are required to tease out the nature of the clay minerals. For Rietveld refinement purposes, chlorite was selected as the reference mineral but may not be correct. Results from the Rietveld refinement of this sample are shown in Figure 6. For comparison purposes, the mineralogy is shown as stacked bars in Figure 7, illustrating the variation with depth. In this figure, the dominance of plagioclase feldspar is readily visible.

Suite 14-MH-016 (Robert Campbell Highway)

The seven samples collected along the Robert Campbell Highway are described as sandstone (Table 1). Quantitative mineralogy is provided in Table 2 and statistics in Table 3. These samples have a simpler mineralogy and are more comparable to the samples from the North Klondike Highway. The samples are dominated by quartz (28-52 wt.%, mean=43 wt.%) and plagioclase feldspar (19-38 wt.%, mean=29 wt.%). A few samples also contain abundant to minor calcite (1-24 wt.%, mean=12 wt.%). Minor K-feldspar occurs but appears to decrease with depth from 19 wt.% in the uppermost sample at 3m, to 11 wt.% at 9 m followed by 5-3 wt.% below. Muscovite, chlorite and dolomite occur in minor to trace amounts and two samples contain minor gypsum and two minor to trace

pyrite. Trace amounts of mixed-layer clay minerals are also observed in two samples. The goodness of fit is moderate to good. The variation in mineralogy with depth is well illustrated in Figure 8. In this plot, it is obvious that both the quartz and plagioclase feldspar dominate the mineralogy.

Overall Comparison

To compare the major mineral components in these samples a ternary plot was made (Figure 9). In this plot, quartz and feldspar minerals are summed and then plotted against the sum of the clay minerals and the sum of the carbonate minerals. The samples are subdivided into the suites noted above. Most of the samples tend to be siliceous with a few tending toward carbonaceous. None are plotting in the “argillaceous” field. There is overlap between suites MH-013 and MH-016 and MH-010 tends to be slightly more clay-rich.

Next Steps

The samples from the Eagle’s Nest Bluff will require further treatments to verify the presence of discrete smectite and chlorite vs. a chlorite-smectite mixed-layer clay mineral. Although in Table 2 a trace amount is reported, it may be slightly more abundant. However, mineral structure files for these types of minerals are not usually available. Hence, the results would be reported as qualitative or at best semi-quantitative.

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Figures and Tables

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Table 1. Locations and lithology for samples from the Yukon.

Sample ID	Depth (m)	Location Name	Lithology
14-MH-010	0.0	North Klondike Highway	sandstone
14-MH-010	4.2	North Klondike Highway	sandstone
14-MH-010	8.7	North Klondike Highway	sandstone
14-MH-010	11.5	North Klondike Highway	sandstone
14-MH-013	19.5	Eagle's Nest Bluff	sandstone
14-MH-013	30.5	Eagle's Nest Bluff	sandstone
14-MH-013	41.5	Eagle's Nest Bluff	sandstone
14-MH-013	57.5	Eagle's Nest Bluff	sandstone
14-MH-013	78.0	Eagle's Nest Bluff	sandstone
14-MH-013	89.9	Eagle's Nest Bluff	sandstone
14-MH-016	3.0	Robert Campbell Highway	sandstone
14-MH-016	9.0	Robert Campbell Highway	sandstone
14-MH-016	19.8	Robert Campbell Highway	sandstone
14-MH-016	36.1	Robert Campbell Highway	sandstone
14-MH-016	51.0	Robert Campbell Highway	sandstone
14-MH-016	58.0	Robert Campbell Highway	sandstone
14-MH-016	64.9	Robert Campbell Highway	sandstone

Table 2. Quantitative mineralogy (wt.%) for samples from the Yukon.

Sample ID	Depth (m)	Qtz	Pl	Kfs	Amp	Cpx	Ol?	Ms	Chl	Kln	Tlc	ML	Anl	Hul	Cal	Dol	Brt	Gp	Hem	Py	GoF
14-MH-010	0.0	63	11					7	11	4					2	2					3.51
14-MH-010	4.2	43	7					7	8	4					6	25					3.04
14-MH-010	8.7	59	16	3				2	8	4					7	1					3.75
14-MH-010	11.5	27	5					10	4	2					5	47					2.83
14-MH-013	19.5	7	22	4	1	17	<i>l</i>		5			tr	3	15	14	2	1		8		3.17
14-MH-013	30.5	13	57	5	4	8	<i>tr</i>		4			tr	1	2		1	tr		5		3.62
14-MH-013	41.5	17	48	8	4	5			4		3	tr		1	4				6		3.84
14-MH-013	57.5	10	49	3	10	10			6		1	tr		5	1				5		3.52
14-MH-013	78.0	2	17			11			4			tr	4	9	46		tr		7		3.24
14-MH-013	89.9	12	36		1	10			7			tr	3	10	14				7		2.96
14-MH-016	3.0	44	24	19				3							10						3.77
14-MH-016	9.0	28	33	11				4	2			tr			22						3.84
14-MH-016	19.8	50	31	5				7	3			tr			1	1		2			3.78
14-MH-016	36.1	44	38	3				5	3						1	1		4		1	3.85
14-MH-016	51.0	47	19	4				3	2						24	1					3.87
14-MH-016	58.0	35	28	4				4	3						23	2				1	3.73
14-MH-016	64.9	51	32	4				5	2						5	1					3.97

Qtz: quartz, Pl: plagioclase, Kfs: K-feldspar, Amp: amphibole, Cpx: clinopyroxene, Ol: olivine, Ms: muscovite, Chl: chlorite, Kln: kaolinite, Tlc: talc, ML: mixed layer clay minerals, Anl: analcime, Hul: heulandite, Cal: calcite, Dol: dolomite, Brt: barite, Gp: gypsum, Hem: hematite, Py: pyrite, GoF: goodness of fit.

Table 3. Basic statistics for each sample collection location.

Sample ID	Qtz	Pl	Kfs	Amp	Cpx	Ol?	Ms	Chl	Kln	Tlc	Anl	Hul	Cal	Dol	Brt	Gp	Hem	Py	GoF
<i>14-MH-010 (n=4)</i>																			
Min	29.0	5.0	3.0				2.0	4.0	2.0				2.0	1.0					2.83
Max	63.0	16.0	3.0				10.0	11.0	4.0				7.0	47.0					3.75
Mean	48.5	9.8	3.0				6.5	7.8	3.5				5.0	19.8					3.283
Std. Dev.	15.61	4.86					3.32	2.87	1.00				2.16	22.32					0.422
<i>14-MH-013 (n=6)</i>																			
Min	2.0	17.0	3.0	1.0	5.0	0.1		4.0		1.0	1.0	1.0	1.0	1.0	0.1		5.0		2.96
Max	17.0	57.0	8.0	10.0	17.0	1.0		7.0		3.0	4.0	15.0	46.0	2.0	1.0		8.0		3.84
Mean	10.2	38.2	5.0	4.0	10.2	0.6		5.0		2.0	2.8	7.0	15.8	1.5	0.4		6.3		3.39
Std. Dev.	5.19	16.02	2.16	3.67	3.97	0.64		1.26		1.41	1.26	5.33	17.87	0.71	0.52		1.21		0.32
<i>14-MH-016 (n=7)</i>																			
Min	28.0	19.0	3.0				3.0	2.0					1.0	1.0		2.0		1.0	3.73
Max	52.0	38.0	19.0				7.0	3.0					24.0	2.0		4.0		1.0	3.97
Mean	42.9	29.3	7.1				4.4	2.5					12.3	1.2		3.0		1.0	3.83
Std. Dev.	8.53	6.26	5.87				1.40	0.55					10.48	0.45		1.41		0.00	0.079

Qtz: quartz, Pl: plagioclase, Kfs: K-feldspar, Amp: amphibole, Cpx: clinopyroxene, Ol: olivine, Ms: muscovite, Chl: chlorite, Kln: kaolinite, Tlc: talc, Anl: analcime, Hul: heulandite, Cal: calcite, Dol: dolomite, Brt: barite, Gp: gypsum, Hem: hematite, Py: pyrite, GoF: goodness of fit.

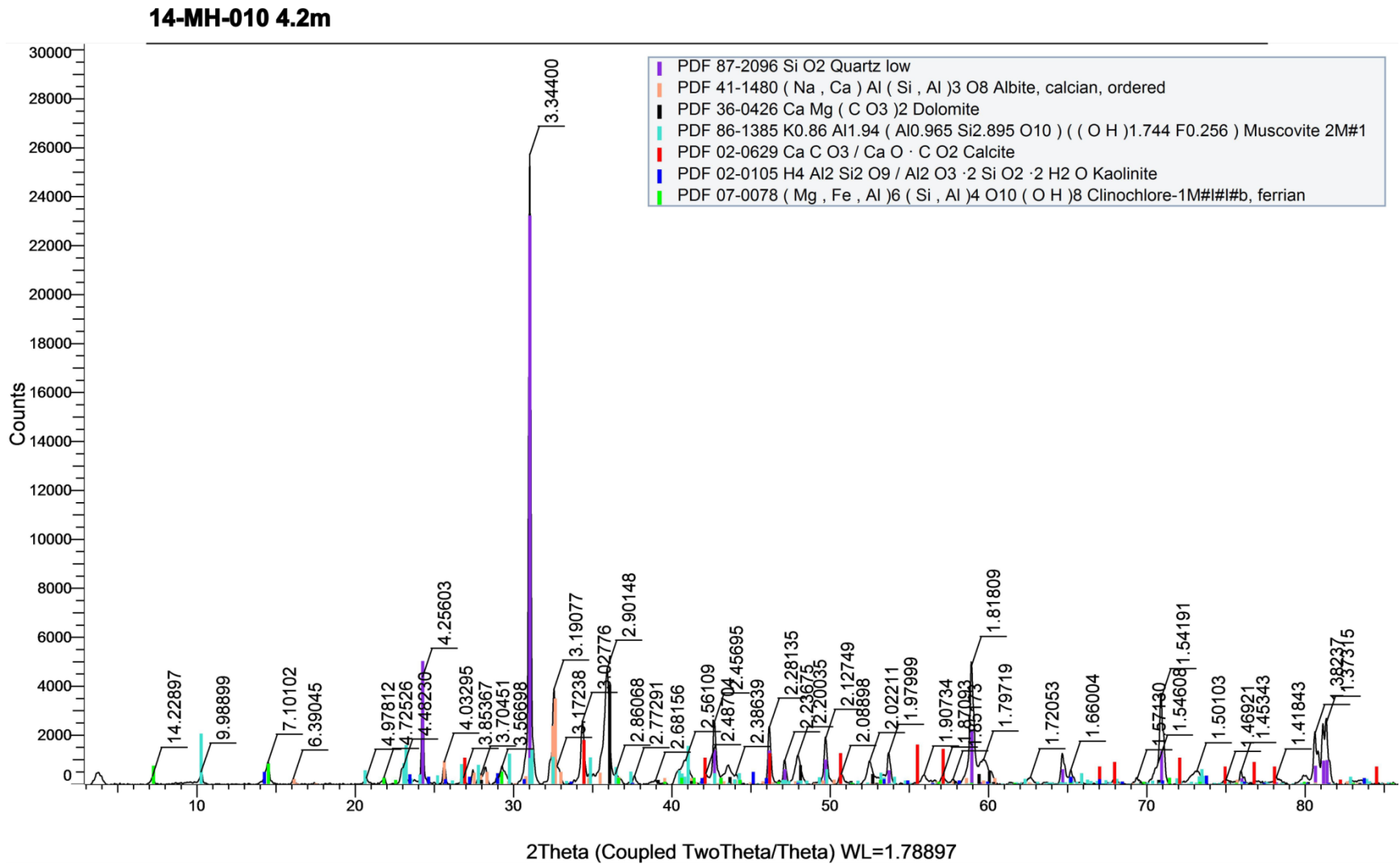


Figure 1. Diffractogram of sample 14-MH-010 4.2m showing search match results using EVA (Bruker AXS Inc.) software.

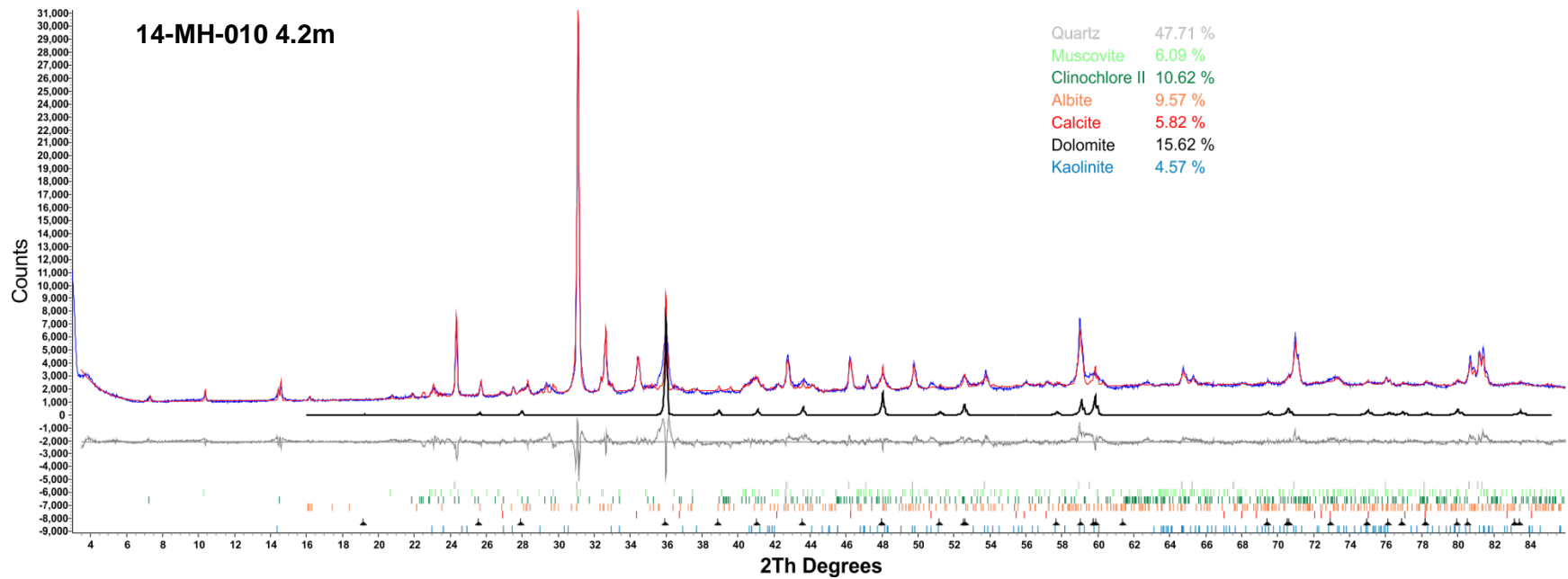


Figure 2. Rietveld refinement of sample 14-MH-010 4.2m using TOPAS (Bruker AXS Inc.) software. Black spectrum shows the match between the dolomite reference mineral (structure file) and unknown; grey is the residual showing how good the fit is between selected reference minerals and the sample.

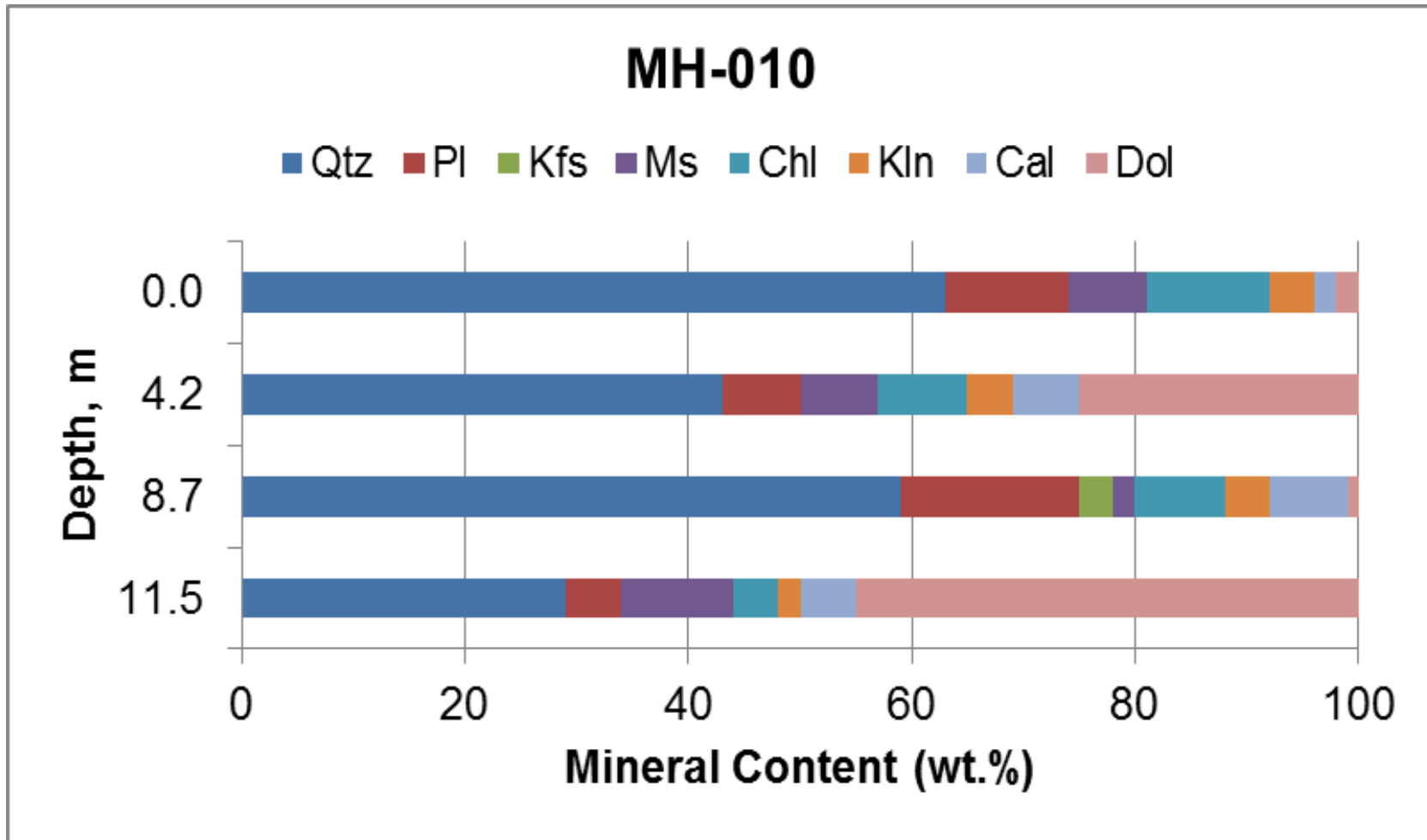


Figure 3. Mineral content in wt.% with depth for each sample from North Klondike Highway.

14-MH-013 30.5m (Coupled TwoTheta/Theta)

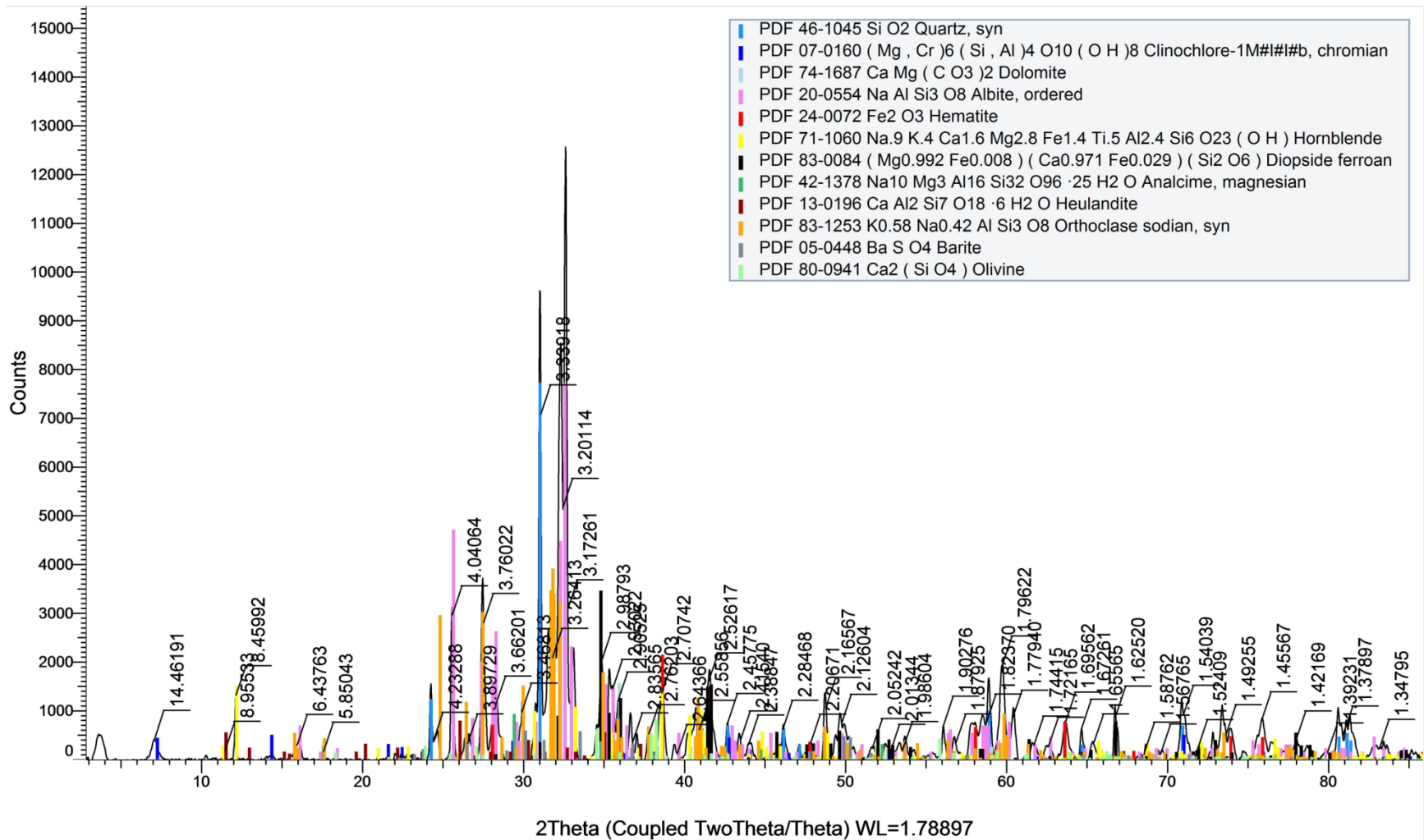


Figure 4. Diffractogram of sample 14-MH-013 (30.5m) showing search match results using EVA (Bruker AXS Inc.) software.

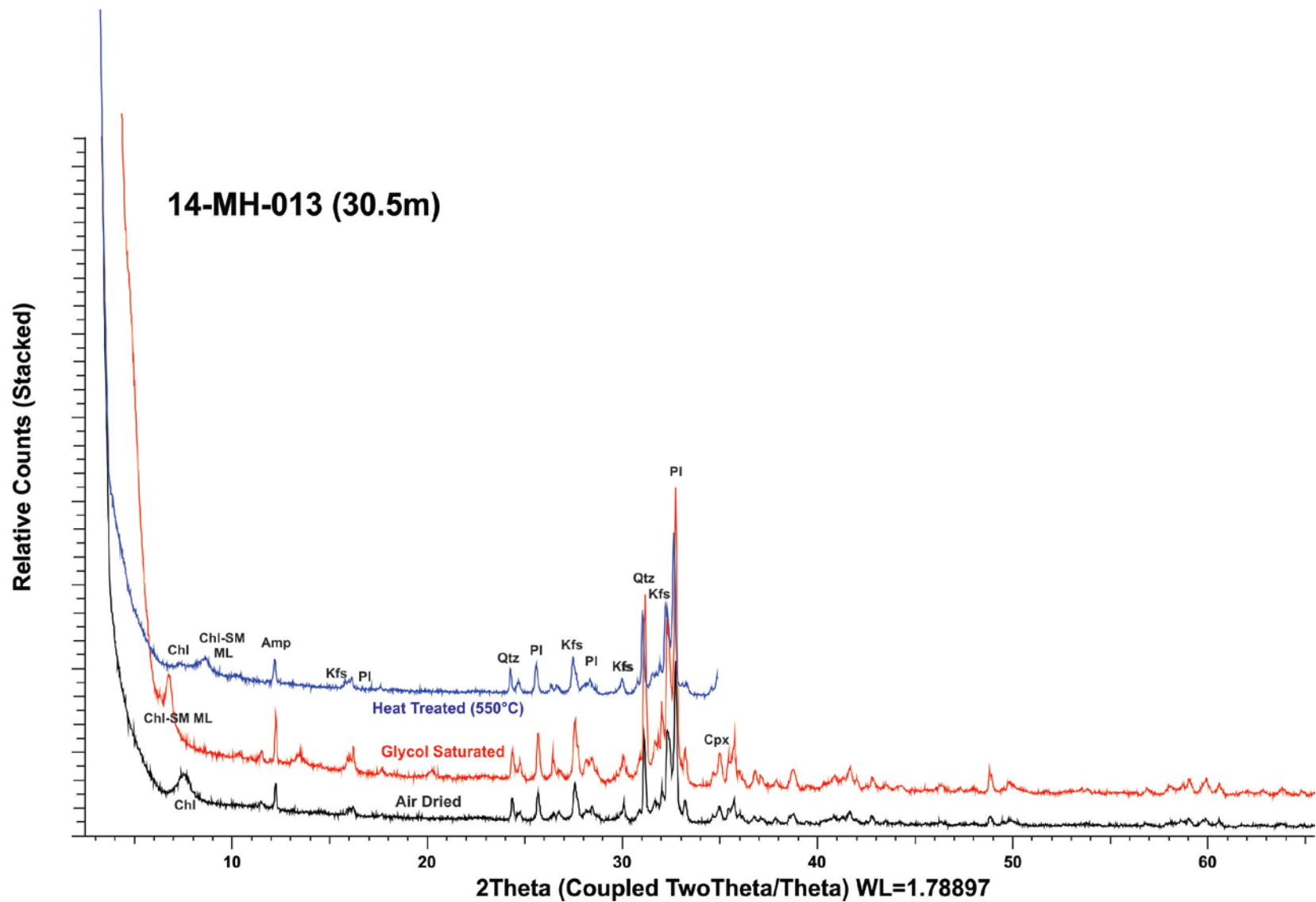


Figure 5. Stacked diffractograms for sample 14-MH-013 (30.5m) showing changes based on air drying, glycol saturation and heat treatment. The shift of the 001 chlorite peak (arrowed) suggests a chlorite-smectite mixed-layer clay mineral.

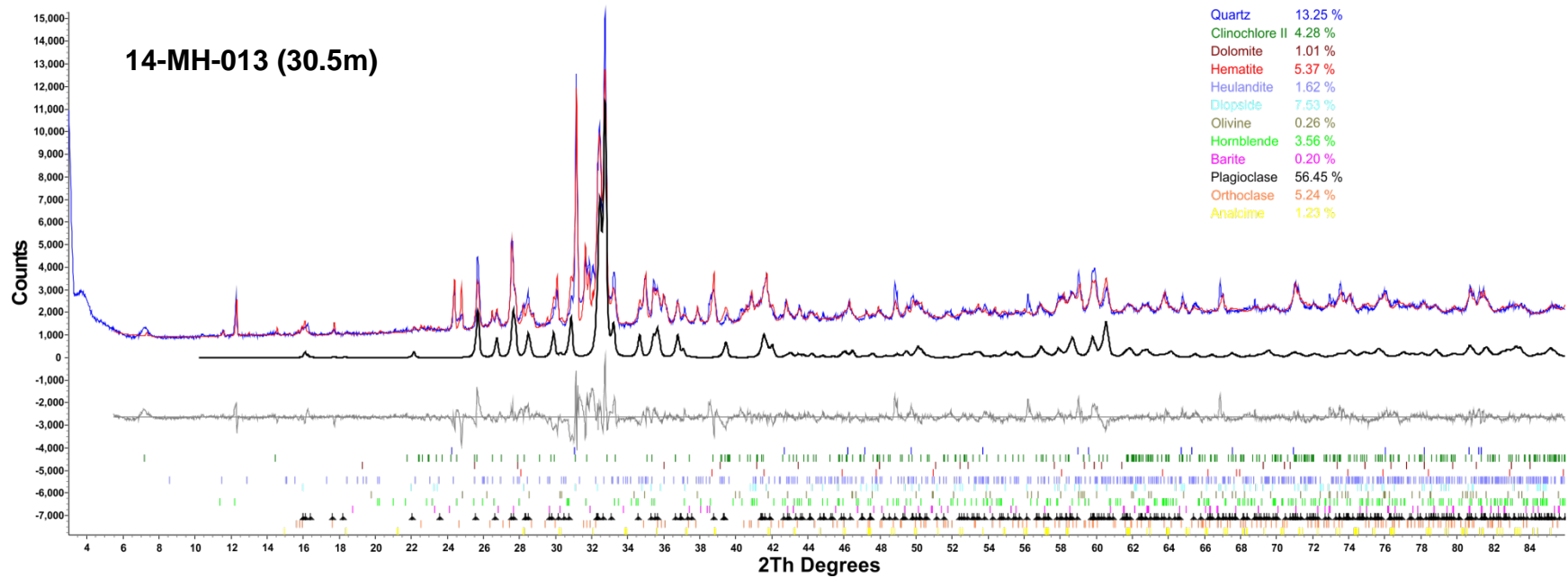


Figure 6. Rietveld refinement of sample 14-MH-013 30.5m using TOPAS (Bruker AXS Inc.) software. Black spectrum shows the match between the plagioclase feldspar reference mineral (structure file) and unknown; grey is the residual showing how good the fit is between selected reference minerals and the sample.

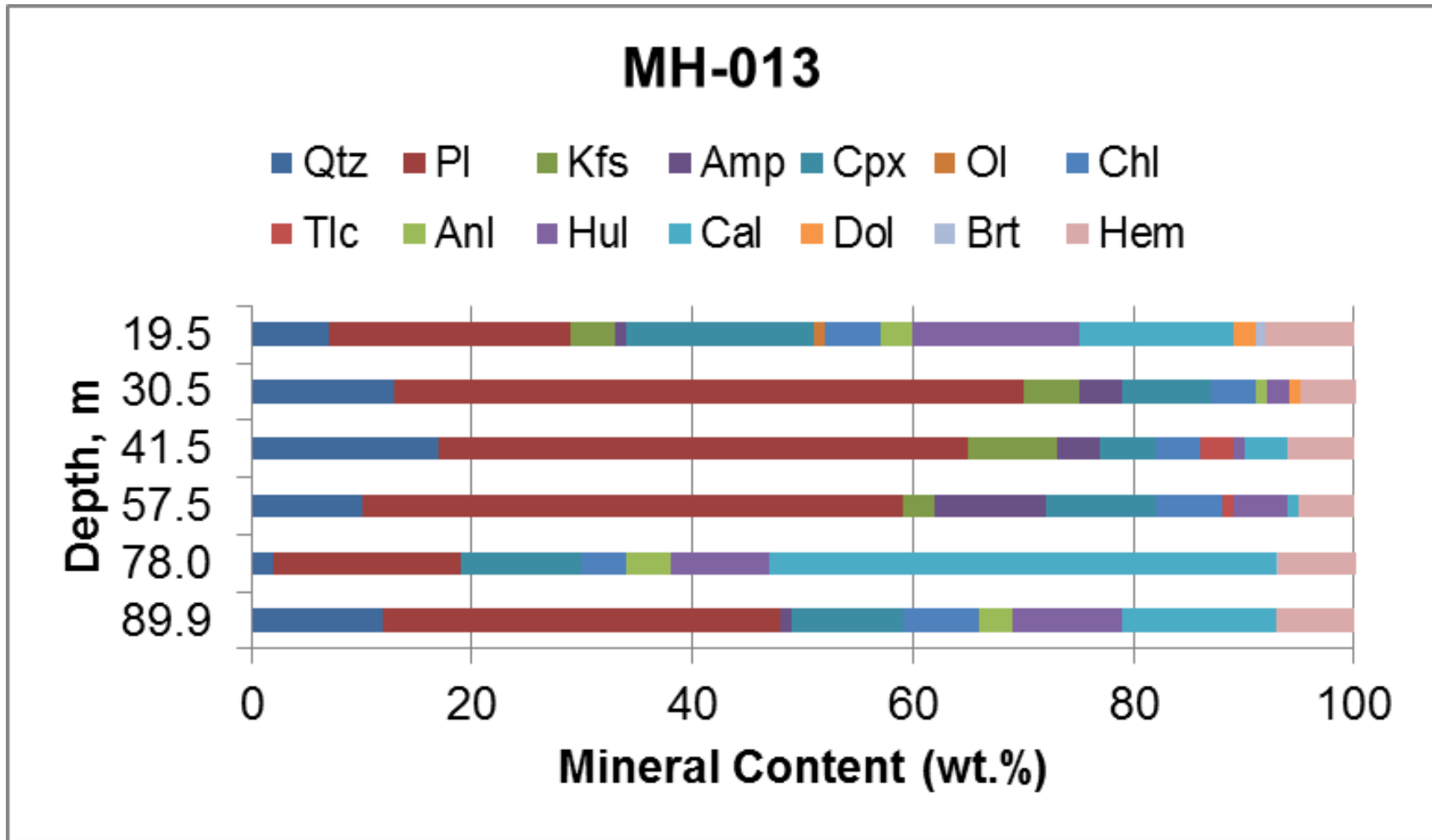


Figure 7. Mineral content in wt.% with depth for each sample from Eagle's Nest Bluff.

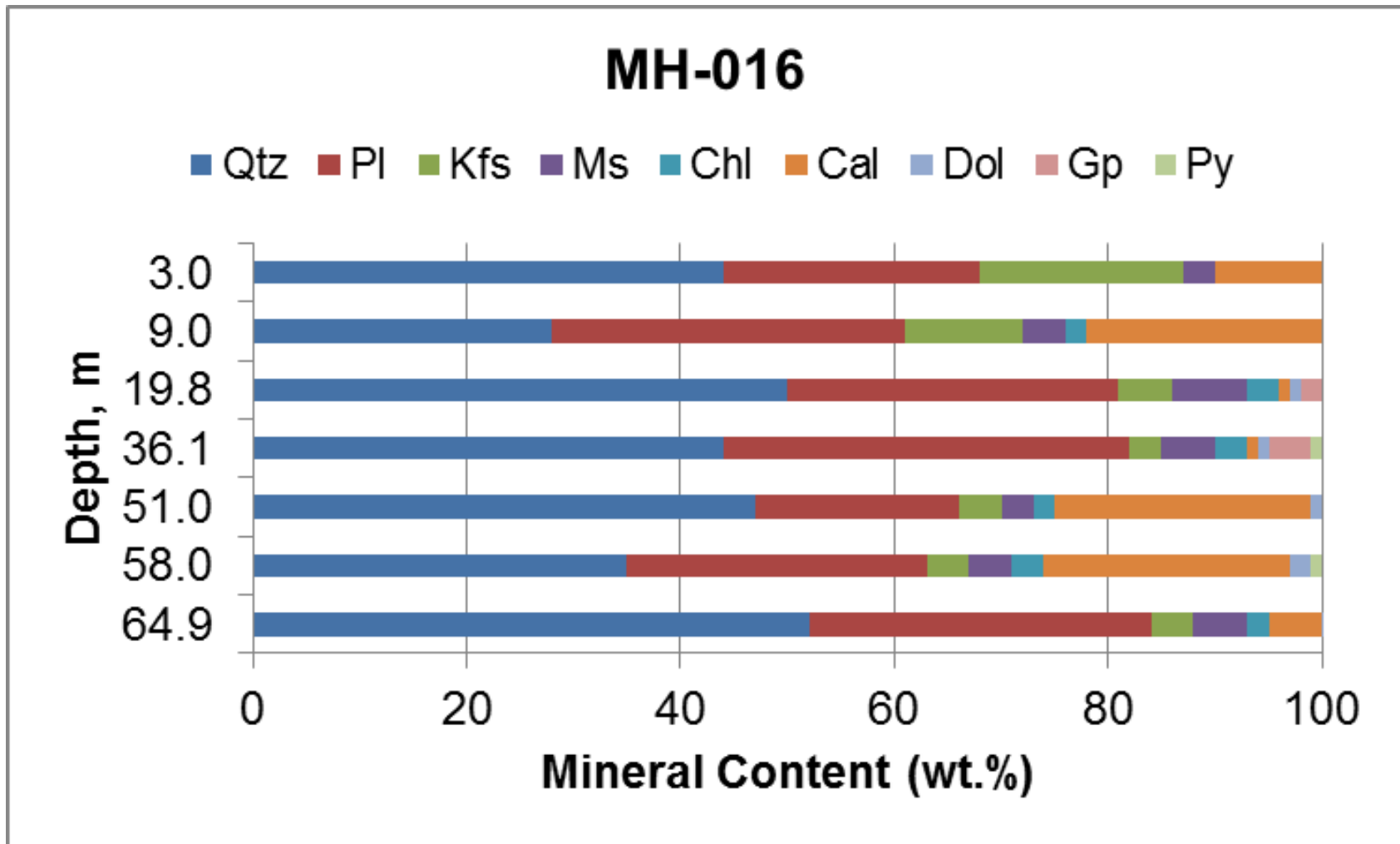


Figure 8. Mineral content in wt.% with depth for each sample from Robert Campbell Highway.

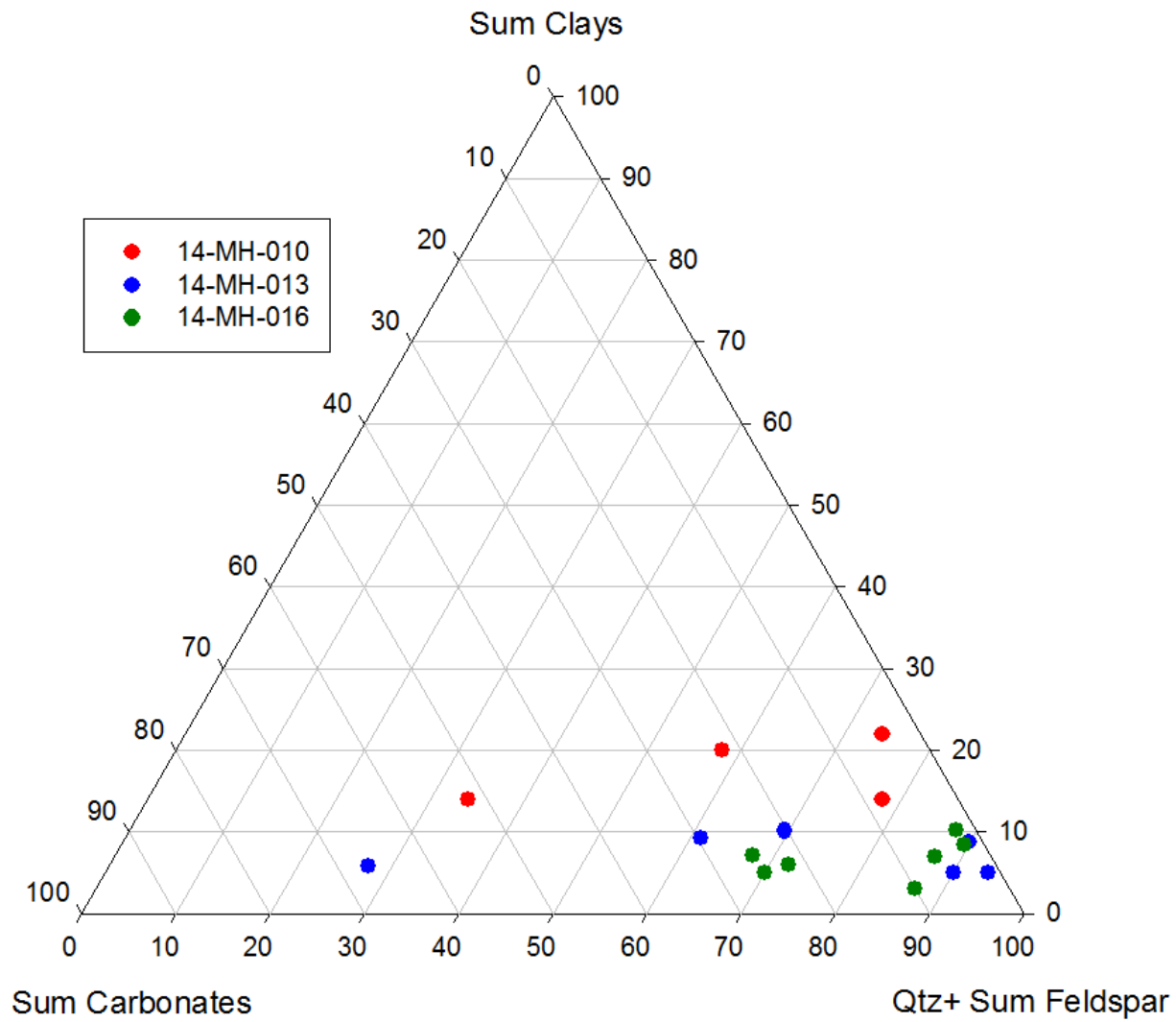


Figure 9. Ternary plot of sum quartz and feldspar, sum clays and sum carbonates grouped by each sampling location.