Evaluation of Tertiary (Paleocene) Bedrock Sandstone of the Sentinel Butte and Bullion Creek Formations for Potential Use as Proppant

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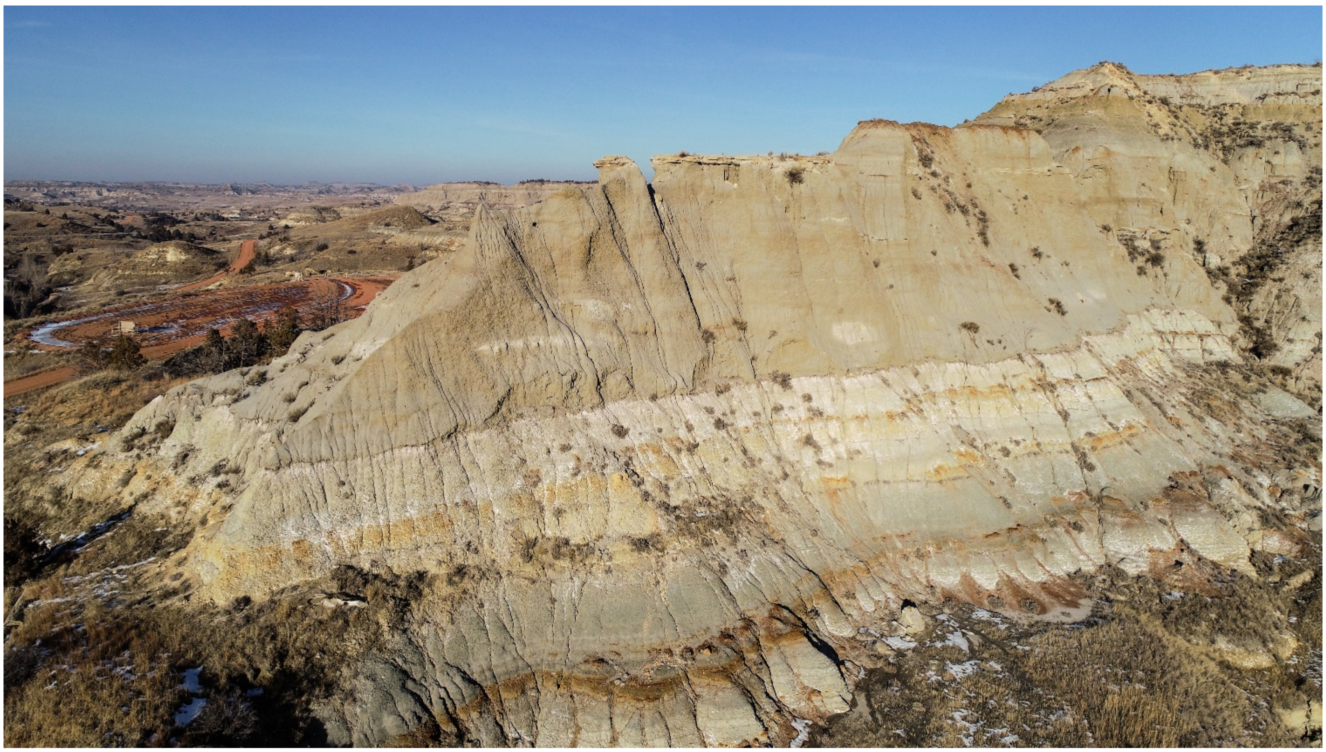

REPORT OF INVESTIGATION NO. 121
NORTH DAKOTA GEOLOGICAL SURVEY
Edward C. Murphy, State Geologist
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On the cover: Oblique drone image of a weakly-bedded sandstone outcrop of the Sentinel Butte Formation, just above the contact with the Bullion Creek Formation, in T. 144 N., R. 101 W., Sec.16, in northwestern Billings County, North Dakota. Sample Nos. 1 through 6 (this report) were collected along a vertical stratigraphic profile at this location.

Abstract

Sandstones in the Fort Union Group (Paleocene) were sampled in western and central North Dakota, along the Little Missouri River Valley in northwestern Billings, southern McKenzie, and northwestern Slope counties, and from surface coal mines near Underwood and Beulah, North Dakota, during the fall of 2018. During the following winter, some of these sandstones were evaluated for use as proppant sand in the hydraulic fracturing of oil and gas wells in the Williston Basin. Sandstone characterization testing was performed on 11 samples from six locations revealing that these sandstones are dominantly well-sorted, fine-grained (70/140), sands containing quartz contents ranging from 41 to 63%, with crush resistance K-values of 3,000 psi or less. Acid solubilities in these sandstones ranged from 6.5 to 22.2% with turbidity values ranging from 29 to 220 FTU. Loss on ignition testing, as a proxy for detrital lignite content, ranged from 2.44 to 9.62%. Sand grain roundness and sphericity averaged 0.5 and 0.6, respectively. Average bulk density of these sandstones is 77.2 pcf. Albite contents averaged 26% within a single 56-foot thick outcrop of channel sandstone of the Sentinel Butte Formation in northwestern Billings county.

Acknowledgements

The authors would like to thank the North Dakota Department of Trust Lands for access to the outcrops sampled in this report. Cartographic support was provided by Mr. Navin Thapa, GIS Specialist with the North Dakota Geological Survey. Proppant sand testing services were provided by Assured Testing Services, LLC in Ridgway, Pennsylvania. Mineralogical analyses (XRD) were completed by Mineral Labs, Inc. of Salyersville, Kentucky.

Author's Note

Recently the oil and gas industry has relaxed proppant testing specifications in part of the U.S. in favor of more regional or local proppant sand source utilization. This evaluation was conducted in response to requests by companies in the proppant sand industry and the North Dakota Oil and Gas Industry to further investigate the state's sand resources for potential use as proppant in the hydraulic fracturing of oil and gas wells in the Williston Basin. The evaluation and testing performed as a part of this investigation, which consist of the traditional proppant sand tests as described in current American Petroleum Institute (API), and the International Organization for Standards (ISO), recommended practices and standards, is described herein in order to provide the comprehensive characteristics of these sandstones.

Technical Note

Imagery for the cover as well as Figures 2, 3, and 5 were captured with the Survey's DJL Phantom 4 Pro Quadcopter Drone. The drone was operated by FAA-Part 107 Certified Pilot, Chris Maike.

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INTRODUCTION

Sandstone samples were collected from the Sentinel Butte and Bullion Creek Formations (Paleocene) in western North Dakota. These samples were primarily obtained along the Little Missouri River Valley in northwestern Billings, southern McKenzie, and northwestern Slope counties as well as surface coal mines near Underwood and Beulah, North Dakota. Samples were collected at outcrop using the methods outlined in Anderson, and others (2019). Eleven samples were collected from six locations (Figure 1). Nine of those were from the Sentinel Butte Formation (six along a vertical profile of a single outcrop) and two were from the Bullion Creek Formation (Table 1).

Sandstone samples were evaluated for proppant suitability in accordance with testing standards and specifications published by the American Petroleum Institute (API Standard-19C) and the International Organization for Standardization (ISO 13503-2). Tests were performed on the most abundant size class, as determined by sieve analysis, and included: gross sample inspection, sample washing and comparison, stereo microscope photomicrography, particle shape factors analysis, qualitative and quantitative mineralogy via X-ray diffraction (XRD) on unwashed (i.e. bulk) samples, crush resistance, acid solubility, turbidity, loss on ignition, and sand density testing. Laboratory testing services were provided by Assured Testing Services in Ridgway, Pennsylvania, during the late winter of 2019. XRD analyses were completed by Mineral Labs, Inc., in Salyersville, Kentucky.

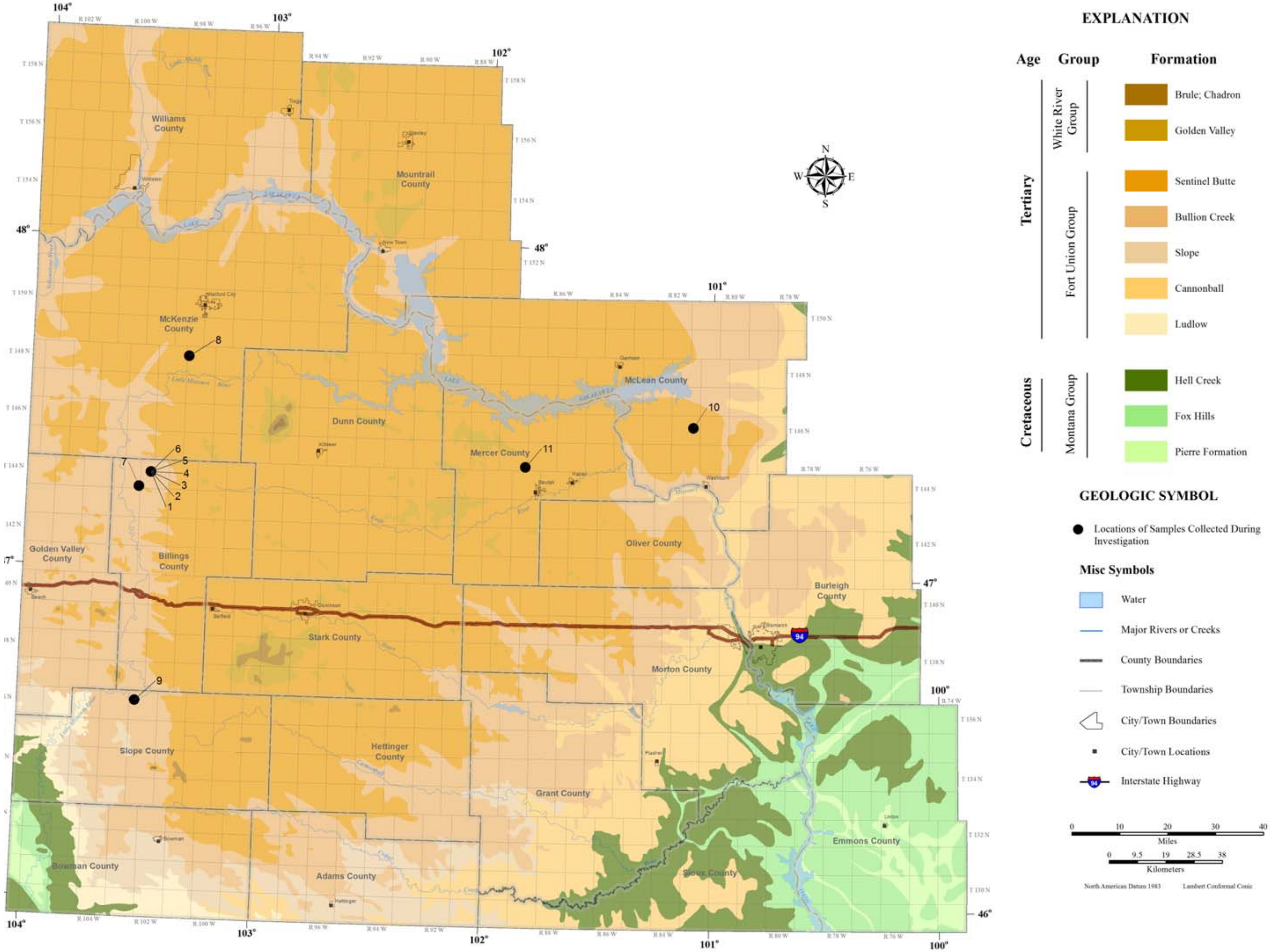

Figure 1. Location of samples collected from sandstones of the Sentinel Butte (Sample Nos. 1 - 6, 8, & 10 - 11) and Bullion Creek (Sample Nos. 7 & 9) Formations in North Dakota. Sample Nos. 1 - 6 were collected on a vertical stratigraphic profile along a single outcrop of basal sandstone.

Table 1. Sample Location Summary

Sample ID	County	Location (T-R-S-Q)	Sample Type/Source	Geologic Unit/Formation	**Geologic Map Unit Description** *(Clayton et al., 1980, Murphy et al., 2009)*
1	Billings	144-101-16-NW	Outcrop; Sandstone	Tsb	Sentinel Butte Formation (Paleocene) - Gray-brown silt, sand, clay, sandstone, and lignite; river, lake, and swamp sediment; as thick as 650 feet. Vertical set of samples (1-6) collected from base to top of outcrop on 10-ft interval.
2	Billings	144-101-16-NW	Outcrop; Sandstone	Tsb	
3	Billings	144-101-16-NW	Outcrop; Sandstone	Tsb	
4	Billings	144-101-16-NW	Outcrop; Sandstone	Tsb	
5	Billings	144-101-16-NW	Outcrop; Sandstone	Tsb	
6	Billings	144-101-16-NW	Outcrop; Sandstone	Tsb	
7	Billings	144-102-36-SE	Outcrop; Sandstone	Tbc	Bullion Creek Formation (Paleocene) - Yellow-brown silt, sand, clay, sandstone, and lignite; river, lake and swamp sediment as thick as 650 feet.
8	McKenzie	148-99-18-NW	Outcrop; Sandstone	Tsb	Sentinel Butte Formation (Paleocene) - Gray-brown silt, sand, clay, sandstone, and lignite; river, lake, and swamp sediment; as thick as 650 feet.
9	Slope	136-102-8-SW	Outcrop; Sandstone	Tbc	Bullion Creek Formation (Paleocene) - Yellow-brown silt, sand, clay, sandstone, and lignite; river, lake and swamp sediment as thick as 650 feet.
10	McLean	Falkirk Mine	Subcrop; Overburden Sandstone	Tsb	Sentinel Butte Formation (Paleocene) - Gray-brown silt, sand, clay, sandstone, and lignite; river, lake, and swamp sediment; as thick as 650 feet.
11	Mercer	Freedom Mine	Subcrop; Overburden Sandstone	Tsb	

BULLION CREEK AND SENTINEL BUTTE FORMATIONS

The Bullion Creek and Sentinel Butte Formations are the primary surficial bedrock units over much of central and southwestern North Dakota. Extensive outcrops of these rock units occur along the Missouri River, Little Missouri River, and their tributaries. Each formation contains up to 650 feet of alternating beds of sandstone, siltstone, mudstone, claystone and lignite deposited in Paleocene rivers, lakes, and swamps (Murphy et al, 2009). The two formations are very similar lithologically, but can usually be distinguished based on color. Bullion Creek strata are brightly-colored with yellow sands, while Sentinel Butte sediments typically appear more somber with gray sands.

Paleocene fluvial systems in North Dakota were low energy, depositing sandstones that are typically fine-grained, often silty, and up to 100 feet in thickness. Steiner (1978) reported that sandstones in the Bullion Creek Formation tend to be slightly finer-grained and marginally better sorted than those in the Sentinel Butte Formation. He found Bullion Creek sandstones to also be more mineralogically mature ranging from 32-68% quartz, 2-16% feldspar, and 26-53% lithic clasts. In comparison, Sentinel Butte sand grains ranged from 20-62% quartz, 8-43% feldspar, and 18-54% lithics.

Ledge-forming siliceous or calcareous concretions, limonitic ironstone nodules, and large spherical or log-shaped concretions are locally abundant in sandstones of both formations. Unweathered sandstones, as a whole, range from poorly to moderately cemented.

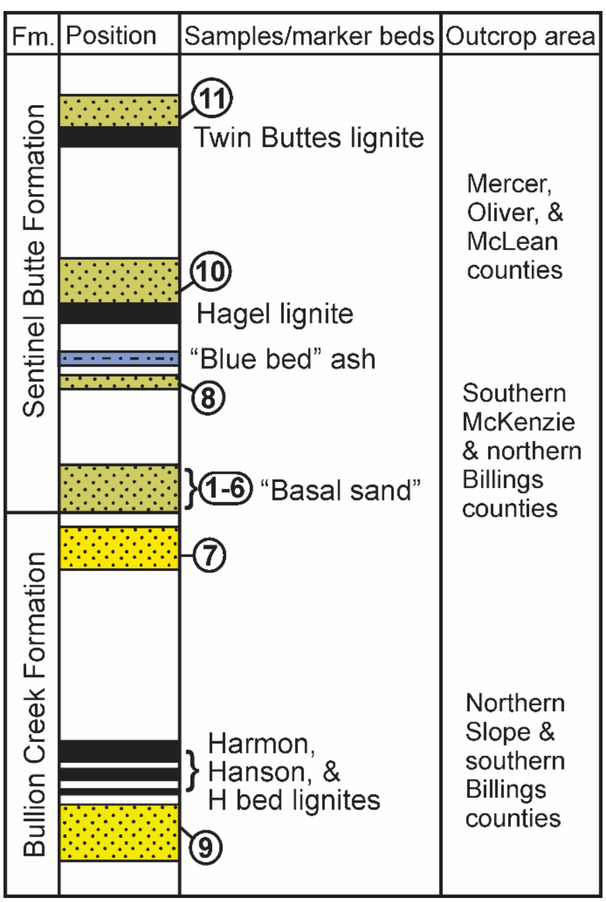

Figure 2. Generalized stratigraphic column indicating relevant marker beds and the relative positions of sandstones sampled (numbered circles) in this report.

DESCRIPTION OF BEDROCK SANDSTONES SAMPLED

Samples No. 1-6

The “basal” sandstone of the Sentinel Butte Formation was selected for a more detailed sampling profile because it is one of the most extensive individual sandstone units in western North Dakota. It is thick (30 to 100 feet), composed primarily of fine-grained sand, and outcrops extensively in oil and gas-producing areas of northern Billings and southern McKenzie counties. The unit varies from sandy siltstone (floodplain facies) to well-sorted fine sandstone (channel facies) (Royce, 1967). It is typically gray in color, exhibits large-scale trough cross-bedding, and contains locally abundant concretions.

Samples No. 1 through 6 were collected from this sandstone at a northern Billings County location where it is 56 feet thick. Sample No. 1 was collected at the base of the unit, and consecutive samples were taken in 10-foot vertical increments through the sandstone (Figure. 3). At this location the sandstone is moderately cemented, and all samples had a very slight reaction to dilute HCl.

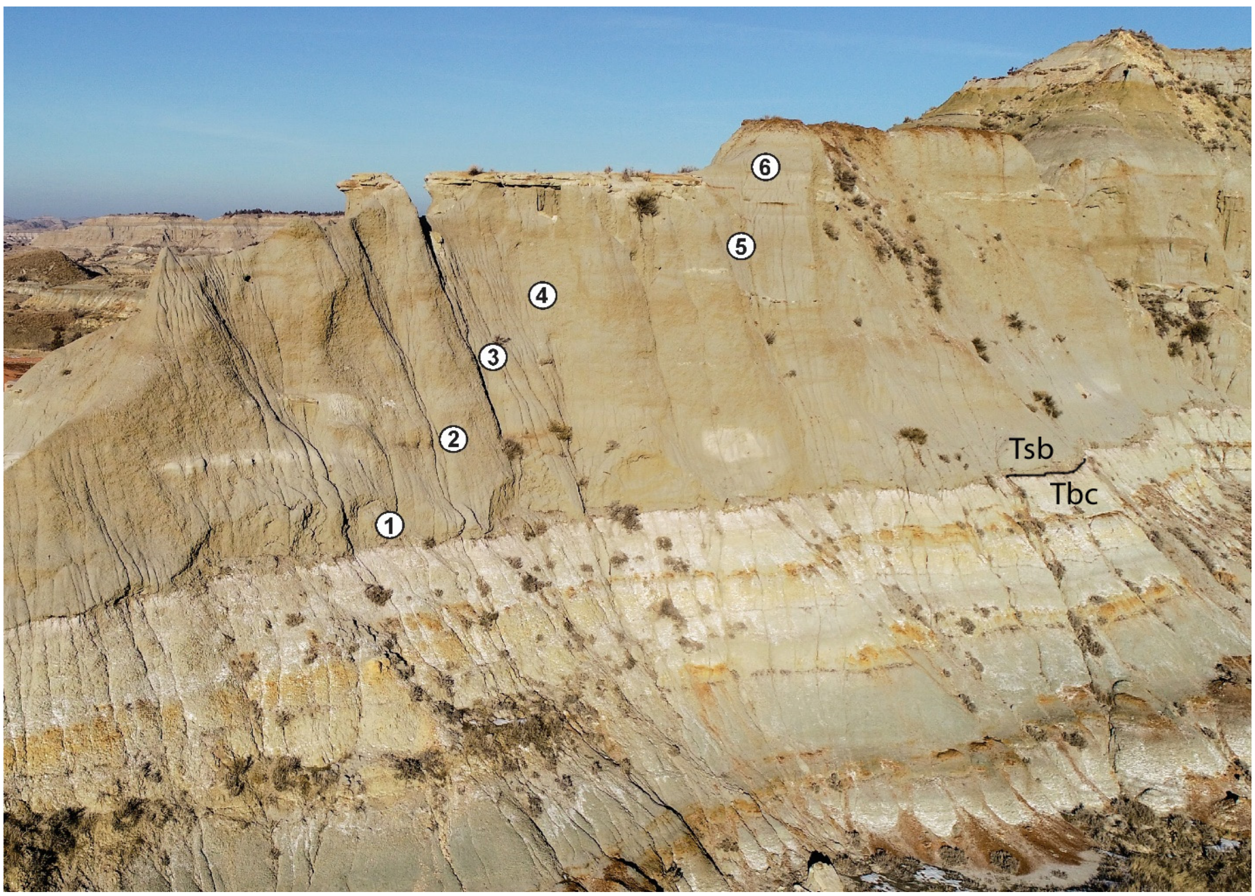

Figure 3. View to the northeast of an outcrop of the Bullion Creek and Sentinel Butte Formations in northern Billings County (T. 144 N., R. 101 W., Sec. 16., NW ¼). Samples No. 1 through 6 were collected along a vertical profile through the basal sandstone of the Sentinel Butte Formation. The contact of the Bullion Creek and Sentinel Butte Formations occurs at the base of this sandstone over much of its extent.

A previous study of the grain size distribution in this sandstone determined it ranged from fine- to medium-grained sand at its base to predominantly fine grained in its upper portions (Sample Nos. 9 - 11, Anderson and others, 2019). Quartz and feldspar appear as the dominant grains in hand sample, with abundant lithic clasts present throughout the profile (Plate I). Fines were present in all hand samples, but appeared to decrease upward through the profile.

Sample No. 7

A 45-foot-thick sandstone occurs in the upper part of the Bullion Creek Formation in northwestern Billings County. It is distinctly yellow in color and easily traceable between outcrops for several miles. Concretions are present at the top of this unit (Figure 4). Sample No. 7 was collected approximately eight feet above the base of the sandstone, where it is moderately cemented and exhibits a slight reaction to dilute HCl. It is weakly cross-bedded and predominantly fine grained, as reported in a previous examination of the unit at this locality (Samples No. 7 & 8, Anderson and others, 2019). In hand sample, the dominant grains are quartz, but also contain appreciable amounts of feldspar and lithic rock fragments, including detrital lignite (Plate I).

Figure 4. View to the northeast of an outcrop of upper Bullion Creek Formation sandstone in northwestern Billings County (T. 144 N., R. 102 W., Sec. 36., SE ¼). Sample No. 7 was collected near the base of this sandstone (circle).

Sample No. 8

Beds of silty sandstone are common throughout the Sentinel Butte Formation where they transition from channel sandstones and fine and thin laterally into channel marginal facies (Royse, 1967). In central McKenzie County, a 15-foot-thick sandstone in the lower portions of the Sentinel Butte Formation is traceable over several miles. It is typically weakly cross-bedded and light gray in color (Figure 5). Sample No. 8 was collected 10 feet above the base of this sandstone. The sandstone is moderately cemented and contains locally abundant nodular concretions. Where tested it exhibited a strong reaction to dilute HCl. Earlier work on this sandstone showed it to be fine grained with over 10% silt by weight (Sample No. 17, Anderson and others, 2019). Quartz was the dominant grain in hand sample, with abundant feldspar and lithic clasts apparent (Plate I).

Figure 5. View to the southeast of an outcrop of the Sentinel Butte Formation in central McKenzie County (T. 148 N., R. 99 W., Sec. 18., NW ¼). Sample No. 8 (circle) was collected from the light gray sandstone. Nodular concretions, common to the Sentinel Butte Formation, are abundant in the sandstone at this site. The stratigraphically significant "blue bed", a volcanic ash, is present at the top of this outcrop and serves as a convenient marker for relocating this unit in the field.

Sample No. 9

An 85-foot-thick sandstone is present in the Bullion Creek Formation in northern Slope County. It is laterally continuous over several miles, where remains at least 50 feet thick. The sand is white to light yellow in color and is typically strongly cross-bedded (Figure 6). Sample No. 9 was collected approximately 55 feet above the base of the sandstone, which contains relatively few concretions except for along its upper contact. This sandstone was moderately cemented where sampled, and it reacted strongly to dilute HCl. The dominant grain type as viewed in hand sample was quartz, with lesser amounts of feldspar and lithic clasts (Plate I). Primary carbonate is 24% in this sample representing a significant percentage of the grains. This sample contains the highest percentage of carbonates in this study, but falls within the 0 to 32% range reported by Royse (1967) for Bullion Creek sandstones.

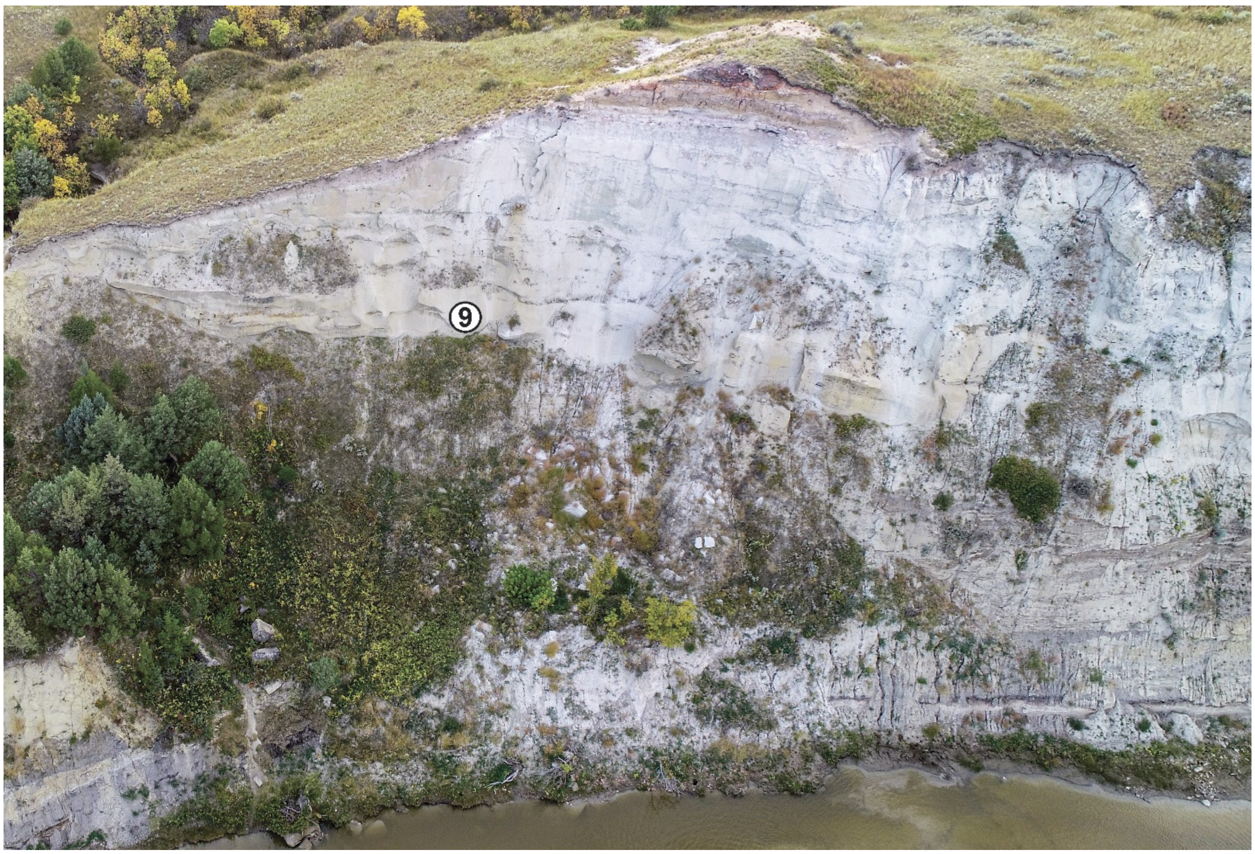

Figure 6. View to the west of an outcrop of Bullion Creek Formation above the Little Missouri River in northern Slope County (T. 136 N., R. 102 W.). Sample No. 9 (circle) was collected from the middle portions of an 85-foot-thick light-yellow sandstone. The clinker at the top of the outcrop was produced by the burning of the Hanson lignite bed.

Samples No. 10 & 11

Sentinel Butte Formation sandstones were also sampled in southern McLean (Sample No. 10) and central Mercer (Sample No. 11) counties where they were part of the overburden at existing surface coal mines. Although these sandstones are massive in subcrop and are favorably fine-grained, they contain considerable amounts of clays and carbonates which result in low crush resistance values <2,000 psi (2K). The sandstones from these two particular locations are not considered desirable at this time for use as proppant.

FIELD ACID REACTIVITY

Carbonate cement is highly variable within the sandstones of both formations. Royse (1967) examined the carbonate content across lithologies of both formations and found mean values for carbonate to be higher in Bullion Creek strata (12.1%) than that of the Sentinel Butte (6.1%). This is generally in agreement with the field reactivity assessment preformed in this study (Table X.) which served as a preliminary indication of the carbonate content of the sample. Each sample was tested for acid reactivity to dilute (10%) hydrochloric (HCl) using a five-class effervescence assessment (Table 2.)

Table 2. Field acid reactivity summary of sandstones sampled in this study.

Effervescence class	Observed reaction to dilute HCl	Samples
No reactivity	No reaction observed	11
Very slightly reactive	Few bubbles seen	1,2,3,4,5,6
Slightly reactive	Bubbles readily seen	7
Strongly reactive	Bubbles quickly form low foam	8,9,10
Violently reactive	Thick foam forms immediately	-

DESCRIPTON OF TESTING RESULTS

Sandstone samples were submitted for testing and characterization in accordance with recommendations and specifications for proppant sand including: particle size distribution (sieve analysis), determination of percent clusters, analysis of grain morphology (sphericity and roundness), acid solubility, turbidity (amount of silt and clay fines), crush resistance, mineralogic evaluation, loss on ignition, and traditional material density (Table 3). Long-term conductivity testing was not performed during this investigation due to lack of overall sand quality and budgetary considerations. Testing and analyses were completed by Assured Testing Services, Inc., in Ridgway, Pennsylvania and Mineral Labs, Inc. (XRD Mineralogy) in Salyersville, Kentucky from December, 2018 to May of 2019.

Sample Preparation

All samples submitted for testing were prepared for analysis by washing, drying, and disassociation. Gradational analysis was first performed on prepared samples to determine the dominant sand size fractions. Remaining tests were then performed on the dominant sand size fraction, in this case sands falling within the 70/140 size classification. Qualitative and quantitative XRD mineralogy was performed on bulk (unwashed) sand samples in order to provide a more accurate assessment of overall sandstone mineralogy.

Particle Size Distribution – Textural (Sieve) Analysis

Sieve analyses are conducted on sediment samples in order to quantify the different sized sand grains within an individual sample. A series of stacked, wire-mesh sieves of standard sizes, are used to sieve each sand sample. Amounts of sand either being retained by the screen on each successively smaller opening sized sieve (% retained) or passing through the screen (% passing) is recorded and reported commonly as tabular data (Table 4) or in graphical form on a grain-size distribution diagram (Figure 7).

The resulting graph and grain-size curves conveys information on the amounts of particle sizes present and the degree of sorting or the variability (or lack thereof) of grains sizes. A well-sorted sample (poorly graded in engineering terms), will have much of the sample volume within or near the same size classes (Table 5), resulting in a very steep curve on the grain size distribution diagram (Figure 7). All of the samples selected for testing were well sorted (poorly graded) sands.

There are several slightly different types of sediment classification schemes, most notably, Modified Wentworth, Unified Soil Classification System (USCS), and American Association of State Highway and Transportation Officials (AASHTO). Generally, these classifications vary in where they draw the boundaries between two different types of sediment (e.g. sand and gravel). The Modified Wentworth system was used in this study.

Table 3. Proppant Testing Analytical Summary of Selected Bedrock Sandstones in North Dakota

Sample No.	Quartz Content[1] (%)	Tested Size Class[2]	Acid Solubility (%)	Loss on Ignition (%)	Crush Resistance (K-Value)	Shape Factors		ISO Mean Particle Dia. (mm)	Median Particle Dia. (mm)	Turbidity (FTU)	% Clusters	Bulk Density (pcf)	Absolute Density (g/cm^3)
						Roundness	Sphericity						
1	50	70/140	7.0	2.62	3K	0.3	0.6	0.166	0.162	80	NCO	76.8	2.66
2	49	70/140	8.7	3.37	3K	0.4	0.6	0.185	0.185	69	NCO	78.7	2.67
3	42	70/140	7.8	3.01	3K	0.5	0.6	0.170	0.167	166	Red clusters on #50 sieve	79.3	2.68
4	45	70/140	6.5	2.44	3K	0.5	0.5	0.177	0.187	220	Red clusters on #30 & #40 sieve	77.4	2.71
5	54	70/140	16.2	3.30	3K	0.6	0.6	0.210	0.217	75	NCO	80.5	2.69
6	51	70/140	7.5	2.53	3K	0.5	0.6	0.197	0.192	81	Red clusters on #70 sieve	78.0	2.67
7	63	70/140	22.1	9.31	<1K	0.5	0.5	0.147	0.138	76	Red clusters on #70 sieve	79.3	2.71
8	55	70/140	16.0	6.18	<1K	0.5	0.5	0.1	0.096	115	Clusters on #70 sieve	71.8	2.68
9	55	70/140	22.2	9.62	<1K	0.5	0.5	0.133	0.124	83	Clusters on #70 sieve	75.5	2.71
10	41	70/140	14.2	3.19	<1K	0.5	0.6	0.114	0.115	51	Clusters on #70 sieve	76.2	2.67
11	57	70/140	11.3	2.82	2K	0.5	0.6	0.203	0.199	29	Clusters on #70 sieve	76.2	2.67

[1] *Quartz content analyzed by XRD methods is representative of entire sandstone sample not just the 70/140 material.*

[2] *Washed sample.*

K-Value is defined as the highest stress level which proppant generates no more than 10% crushed material, rounded down to the nearest 1,000 psi.

FTU = Formazin Turbidity Unit.

NCO = No clusters observed.

pcf = pounds per cubic foot.

Table 4. Bulk Sample Sieve Analysis Results (Weight % Retained)

	Sample No.	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**
U.S. Standard Sieve Size	30	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.4	0.0	0.2
	40	--	--	--	--	--	0.4	0.1	0.0	0.7	0.2	0.7
	50	1.1	1.8	0.5	6.8	2.6	0.1	0.1	0.6	1.3	0.3	4.4
	60	--	--	--	--	--	9.8	0.7	0.5	1.5	0.4	13.9
	70	12.7	29.8	13.7	26.0	50.7	24.9	3.1	0.5	1.9	0.2	24.1
	80	17.7	20.8	22.6	22.3	22.4	--	--	--	--	--	--
	100	31.0	20.5	30.4	24.9	13.6	51.3	32.9	2.5	12.1	3.2	37.7
	120	20.7	12.4	18.4	11.4	5.8	--	--	--	--	--	--
	140	9.5	6.5	3.4	5.0	2.8	11.6	50.7	32.8	58.3	58.8	13.4
	200	5.9	6.3	9.9	2.9	1.7	1.6	10.8	47.3	19.4	29.7	3.4
	Pan	1.4	1.9	1.1	0.7	0.4	0.3	1.6	15.8	4.4	7.2	2.2
Total		100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

-- No data collected on selected sieve.

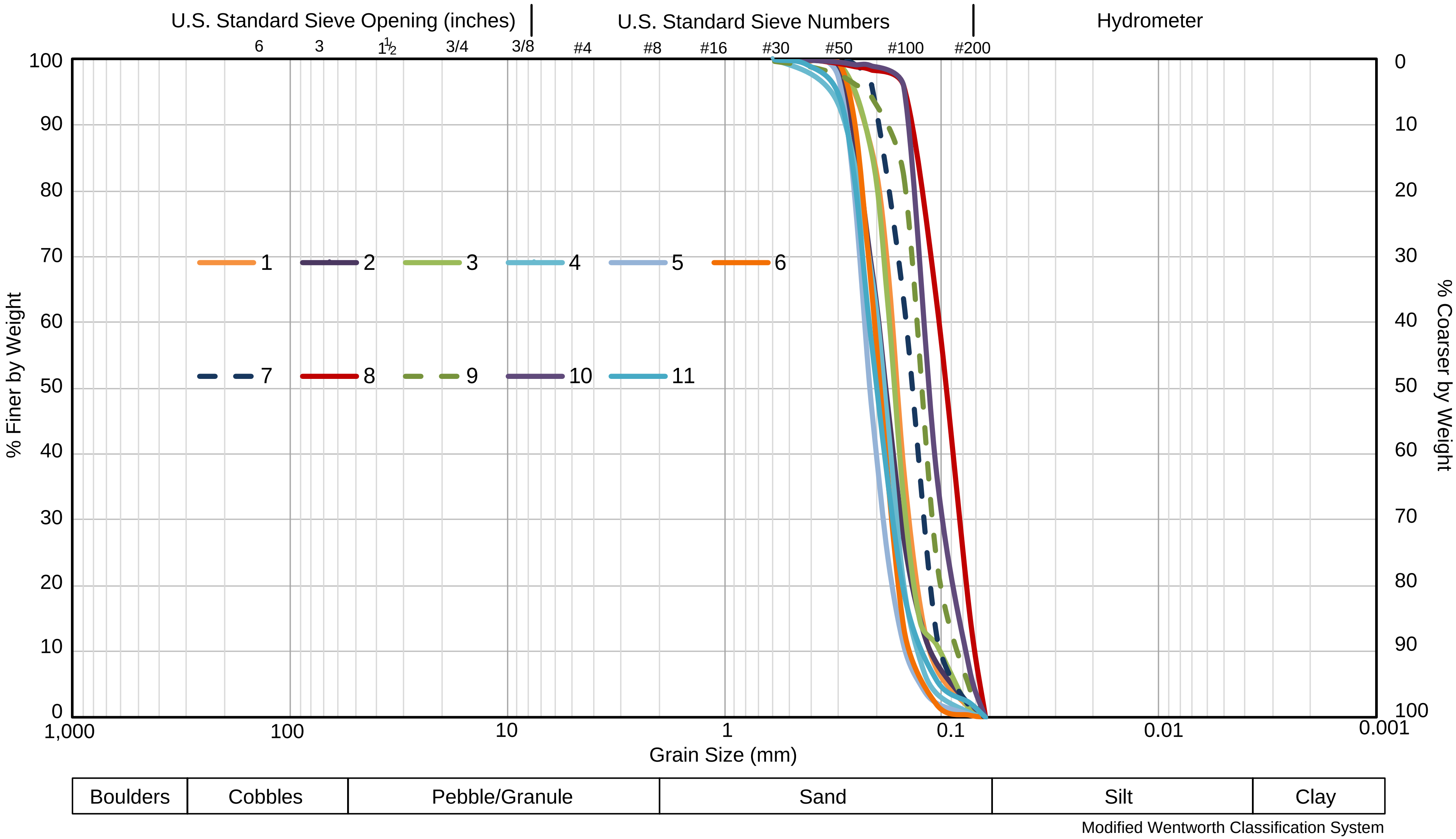

Figure 7. Grain-size distribution curves (sieve analyses) for sandstone samples from the Sentinel Butte (Tsb Sample Nos. 1 - 6, 8, & 10 - 11) and Bullion Creek Formation (Tbc Sample Nos. 7 & 9) in western North Dakota. These sandstones classify as being well sorted and fine-grained.

Table 5. % Sand in Size Class

	Sample No.	1	2	3	4	5	6	7	8	9	10	11
Size Class	40/70	13.8	31.6	14.2	32.8	53.3	34.8	3.9	1.6	4.7	0.9	42.4
	70/140	78.9	60.2	74.8	63.6	44.6	62.9	83.6	35.3	70.4	62.0	51.1
	100/200	36.1	25.2	31.7	19.3	10.3	13.2	61.5	80.1	77.7	88.5	16.8

All of the samples selected for further testing and characterization as potential proppants fall into the grain size ranges for classification as a “sand” according to the Modified Wentworth classification scheme (Figure 7) and can be further characterized as well sorted (poorly graded) to very well sorted, medium-to-fine-grained sands. All of the samples tested had the majority of grains fall in the 70/140 or “100 mesh” sand size range (Table 5).

Statistical analyses can be run on data generated in a grain-size distribution diagram and used to quantitatively compare individual samples for potential engineering applications. The mean grain-size diameter is commonly used to characterize proppant distribution in hydraulic fracturing applications while the median grain-size diameter is used to characterize gravel-packing distributions (Table 3).

% Clusters

The volume of grains that were aggregated in clusters was estimated by visual inspection of individual samples under a microscope at 10x to 20x magnification. Clusters may be problematic for proppant use depending on the size of the clusters and type of geochemical cement holding the individual grains together. Sand proppant is recommended to have less than 1% by count (<1/100) of clusters within a group of individual sand grains (API, 1995).

Only three of the samples (Nos. 1, 2, & 5) did not contain any observable clusters (Table 3). The remaining nine samples all reported clusters commonly found on the # 70 sieve with several red clusters reported from four of the samples (Sample Nos. 3,4,6, & 7). The red clusters may be attributed to the presence of iron, particularly along bedding planes.

Sand Grain Morphology (Sphericity and Roundness)

Individual sand grain sphericity and roundness are two particle shape factors that are evaluated when characterizing the proppant potential of a sand. These factors can be qualitatively observed through standard photomicrographs (Plate I). Sphericity refers to how closely a particular grain of sand resembles that of a sphere and roundness refers to the corners of an individual sand grain. For proppant use a sand with high sphericity and roundness is desirable. Recommended sphericity and roundness values of 0.6 or greater are desirable, with values of 0.7 or greater characteristic of high-strength proppants (API, 2018).

Most of the samples tested (10 of 12) had sphericity and roundness values of 0.5 or greater (Table 3). Samples No. 1 and No. 2, from different stratigraphic intervals in the same outcrop of Sentinel Butte sandstone had values less than 0.5. These two samples, spaced 10 feet vertically apart, came in just under the recommended values of 0.6 for sphericity and 0.3 to 0.4 for roundness. Most of the individual grains in these sandstones are quite angular (Figure 8).

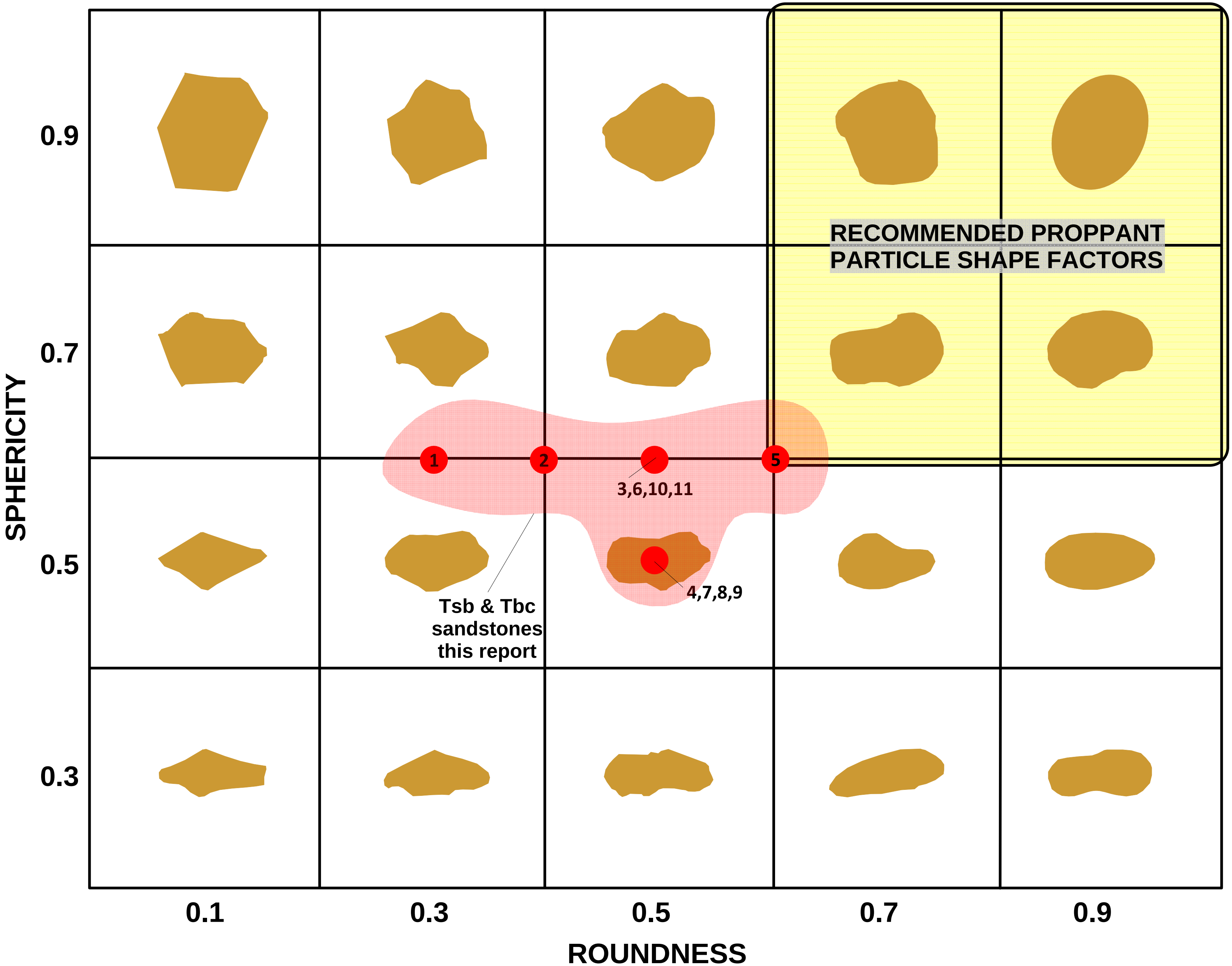

Figure 8. Comparison of Sphericity and Roundness values for sandstone samples from the Sentinel Butte (Tsb Sample Nos. 1 - 6, 8, & 10 - 11) and Bullion Creek Formations (Tbc Sample Nos. 7 & 9) in western North Dakota. These sandstones have particle shape factors that are generally below the desired specifications (shaded yellow) for frac sand as compared to "Ottawa White" silica sands.

Acid Solubility

The volume of a sand that is soluble in strong acid is an important test of an effective proppant as acid treatments of oil and gas wells are common during completions. API (2018) recommends for proppant sands that no greater than 3% (by weight) of <= 70/140 be soluble in a 12:3 hydrochloric (HCL) or hydrofluoric (HF) acid solution. None of the samples tested were below the recommended acid solubility threshold of 3% or less (Figure 9). Acid solubility on these sandstones ranged from 6.5% from a sample collected from the Sentinel Butte Formation in northwestern Billings County to a high value of 22.2 % in a sample collected from an outcrop of Bullion Creek Formation sandstone (Sample No. 9) in north-central Slope County.

Silt and Clay Fines Testing (Turbidity)

Turbidity tests measure the optical properties of water samples containing suspended sediment. It is commonly used to determine the percentage of fine materials (e.g. silts and clays) present within a particular water sample. With respect to proppant potential, the turbidity test measures the amount of associated fines within a particular sand sample. It can be used to identify sand sources that require additional washings, etc., during initial processing of raw product into frac-sand. Turbidity is measured and commonly reported in Formazin Turbidity Units (FTU). The recommended limit of frac sand is less than 250 FTU. All the Survey samples were below the recommended limit of 250 FTU. The samples ranged from 29 FTU in Sentinel Butte sandstone in Mercer County to 220 FTU from Sentinel Butte sandstone in northwestern Billings County (Figure 10).

Crush Resistance

Sand compositions can be quite variable as can the resultant strengths. A crush resistance test measures the amount of fine-grained material generated during the subjection of a given sand sample (within a specified size range) to a pre-determined amount of stress or load. Crush resistance testing was performed on all eleven sandstone samples (Figure 10). As a part of the testing procedure, samples were subjected to one set of three pre-determined stresses, depending on the size range of the samples, and a resultant K-value was determined. A K-value is determined from the amount of crushed fines generated at a given applied stress and is defined as the highest stress level that will generate no more than 10% crushed material (rounded down to the nearest 1,000 psi).

Sample Nos. 1 - 9 were subjected to a stress level set of 2,000, 3,000, and 4,000 psi and generated K-values of dominantly <3K. Sample Nos. 10 and 11 were subjected to a three-stress level set of 1,000, 2,000, and 3,000 psi and generated K-values of <2K (Table 5). Ottawa “white” sands typically generate K-values between 7 to 15K. It appears that the sandstones tested would not be suitable for hydraulic fracturing of Bakken oil and gas wells where the fracture closure stresses in the reservoir range typically around 9,500 psi.

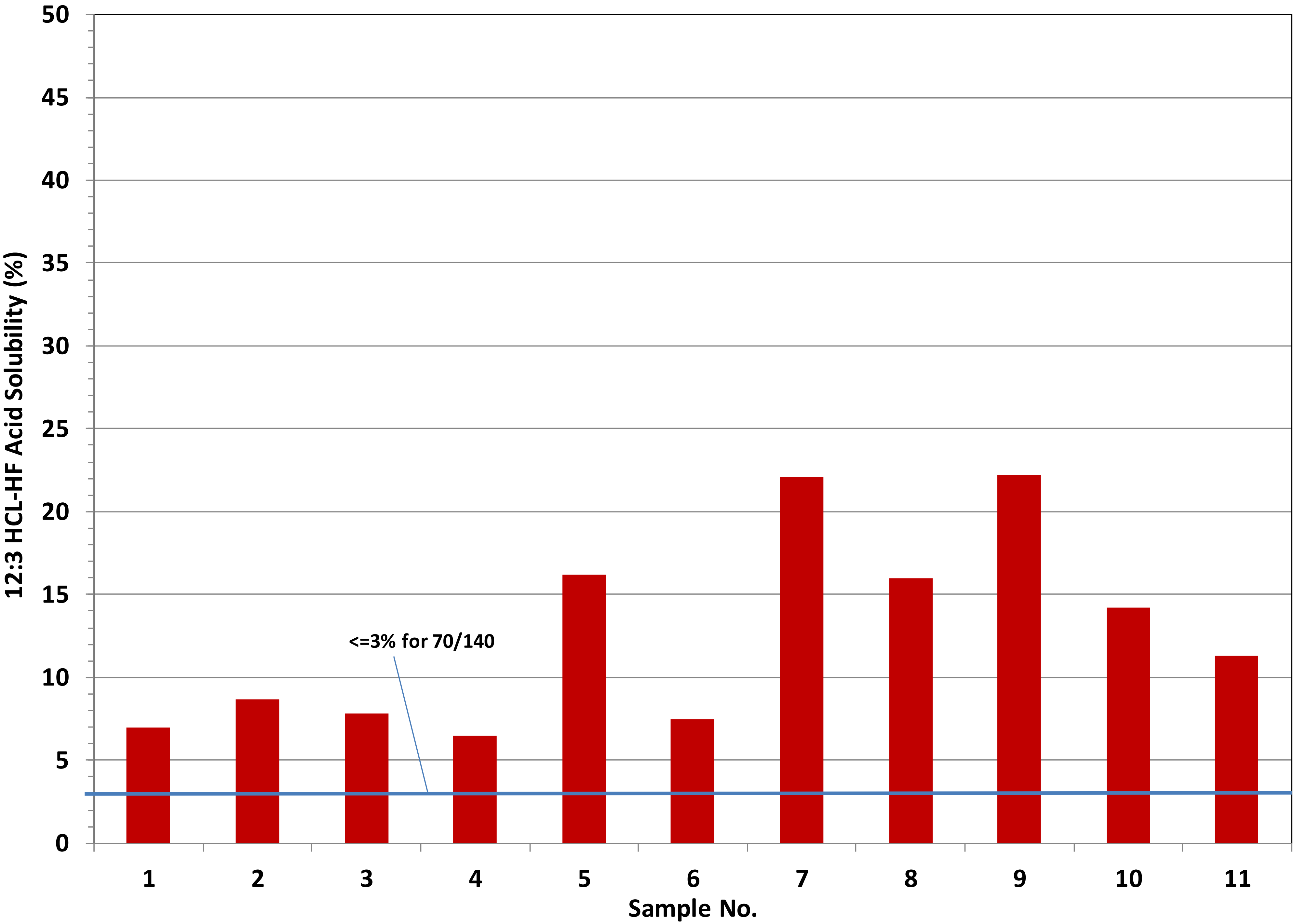

Figure 9. Comparison of HCl:HF acid solubility results for sandstone samples from the Sentinel Butte (Sample Nos. 1 - 6, 8, & 10 - 11) and Bullion Creek Formations (Tbc Sample Nos. 7 & 9) in western North Dakota. Recommended specifications for acid solubility on 70/140 sands are less than or equal to 3%.

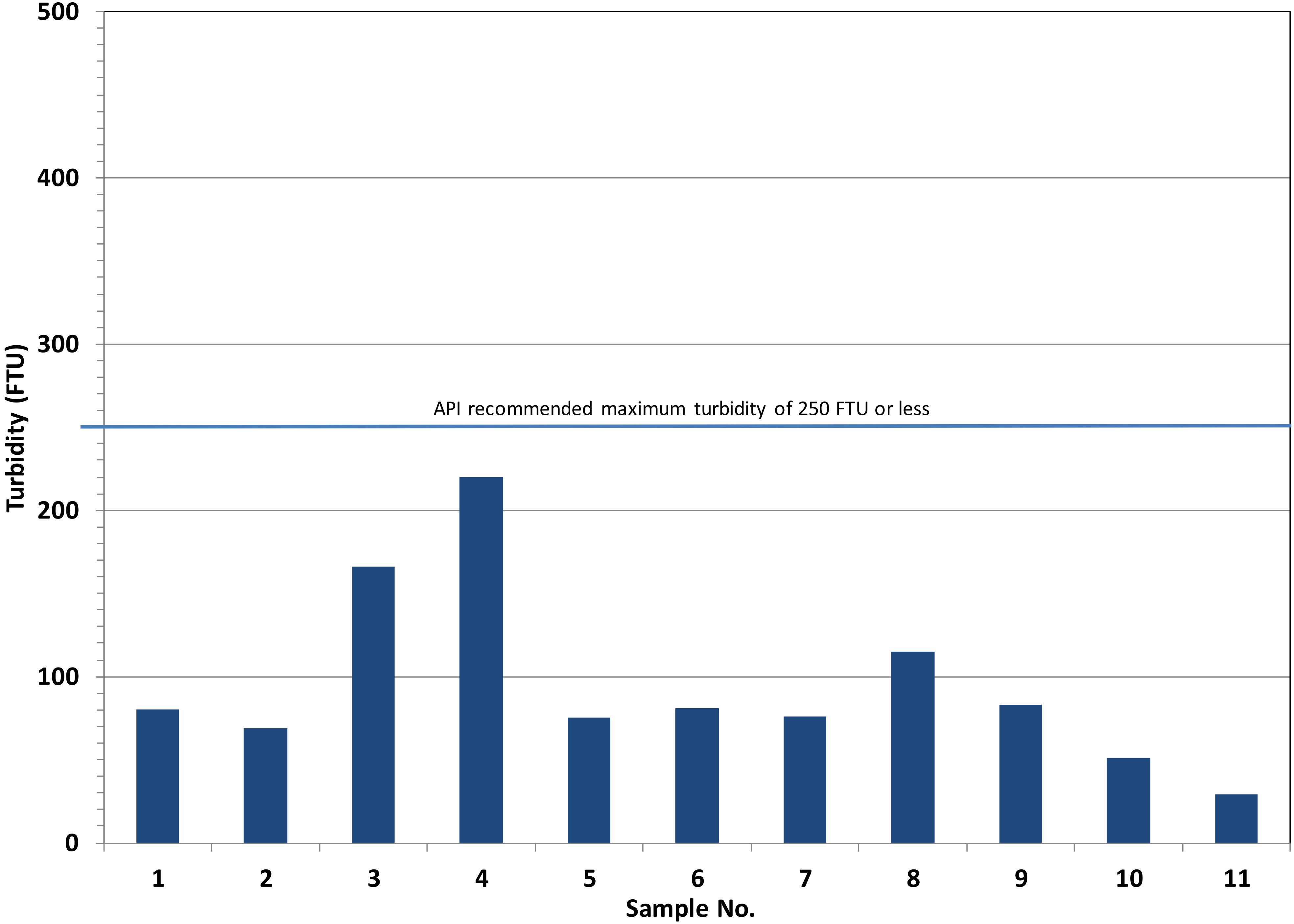

Figure 10. Comparison of turbidity results for sandstone samples from the Sentinel Butte (Tsb Sample Nos. 1 - 6, 8, & 10 - 11) and Bullion Creek Formations (Tbc Sample Nos. 7 & 9) in western North Dakota. The recommended specification for maximum turbidity is 250 FTU or less.

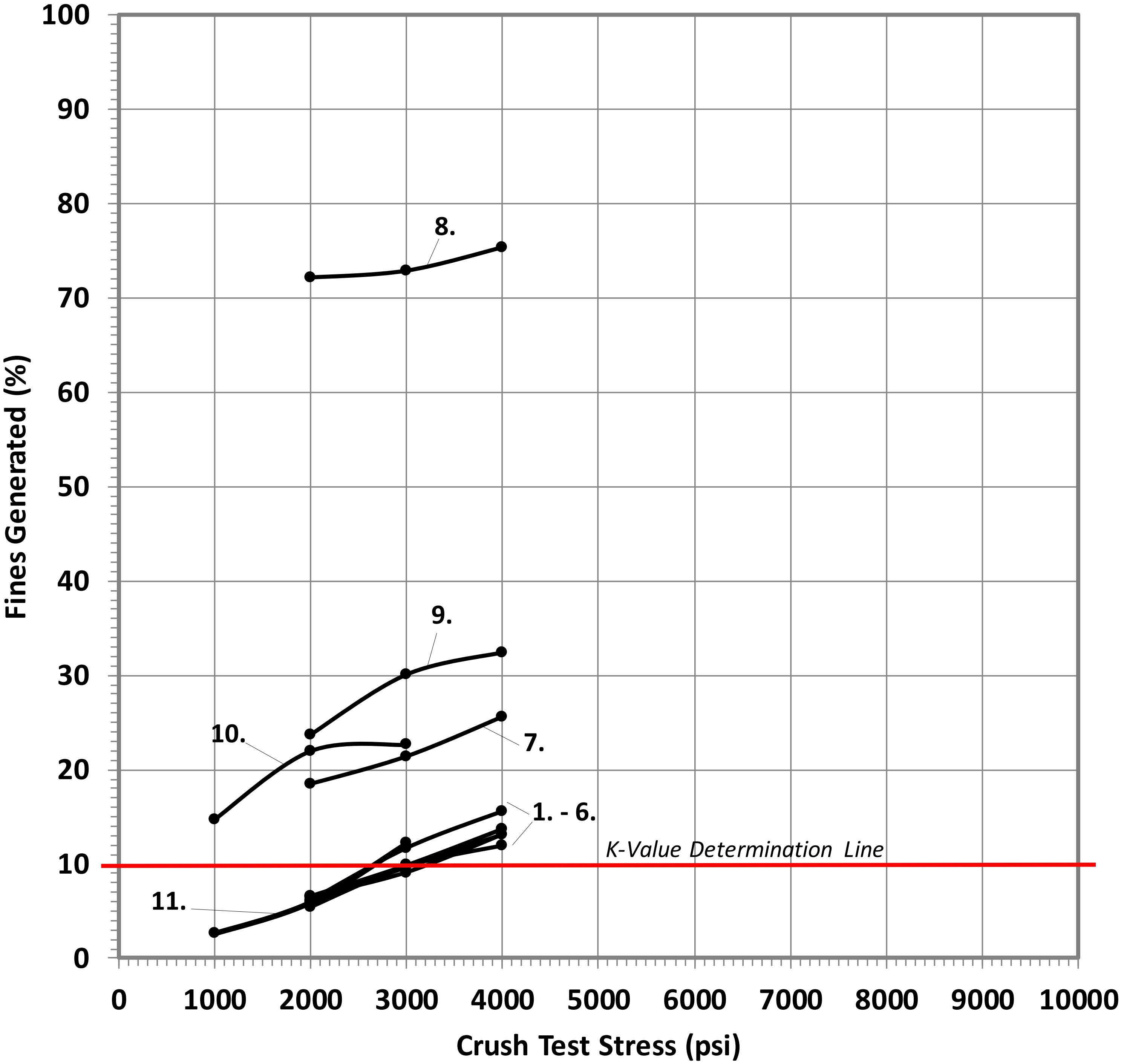

Figure 11. Comparison of crush resistance results for sandstone samples from the Sentinel Butte (Sample Nos. 1 - 6, 8, & 10 - 11) and Bullion Creek Formations (Sample Nos. 7 & 9) in western North Dakota. Reco - mmended specifications for crush resistance for the Bakken Petroleum System are in the range of 8K to 9K (8,000 to 9,000 psi).

Table 6. Crush Resistance Testing Data Summary

Sample No.		1	2	3	4	5	6	7	8*	9	10	11
		Fines Generated (%)										
Tested Stress (psi)	1000	NT	NT	NT	NT	NT	NT	NT	NT	NT	14.67	2.64
	2000	6.02	6.50	5.50	6.59	6.60	6.15	18.53	72.18	23.75	22.00	5.94
	3000	9.65	9.75	9.80	9.12	9.89	11.65	21.41	72.89	30.10	22.67	12.21
	4000	13.10	13.68	12.00	13.14	13.77	15.53	25.56	75.35	32.44	NT	NT
K-Value		**3**	**3**	**3**	**3**	**3**	**3**	**<1**	**<1**	**<1**	**<1**	**2**

** Sample No. 8 was ran twice with values of ~75% fines generated at 2,000 psi.*

Loss on Ignition Testing

Loss on Ignition (LOI) testing was performed on these samples in order to serve as a proxy for determination of the potential detrital lignite content of the sandstones. Values ranged from 2.62% to 9.31% and were found to be higher in the sandstones from the Bullion Creek Formation at 9.47% as compared to Sentinel Butte sandstones at 3.27% (Figure 12).

Mineralogy (X-Ray Diffraction)

Bulk sample geochemistry was determined using qualitative and quantitative X-ray diffraction (XRD) on all eleven sandstone samples (Appendix A). XRD analysis is commonly used to determine the mineralogy of fine-grained lithologies, particularly clays (Poppe et. al. 2001). In general, the samples had similar overall mineralogical compositions (Table 6) with some variability in the lower percentages of feldspars and carbonates (Figure 13). Quartz percentages ranged from 41 to 63% and feldspar contents (dominantly albite) ranged from 10 to 42%. Carbonate contents ranged from 2 to 24% with the clay content dominantly montmorillonite and potentially muscovite averaging around 12.5% (Figure 14). In comparison, Ottawa “white” silica sands are commonly 99% quartz sand.

Bulk Density

In regard to proppants, the bulk density describes the mass that fills a unit volume and includes both the proppant and the void space (i.e. porosity) in the sample. It is commonly used in determining the mass of proppants required to fill fractures, a storage vessel, or in completing general volume estimates. The bulk density of these sandstones (Table 3) ranged from 77.4 to 80.5 pounds per cubic foot (pcf).

Absolute (particle) density

The absolute density or particle density of a sandstone measures the density by way of pycnometric (gas displacement) methods. The absolute density of the sandstones ranged from 2.66 to 2.71 grams per cubic centimeter (g/cm^3) with an average of 2.68 g/cm^3 (Table 3). On average, the absolute density of sand is 2.65 g/ cm^3.

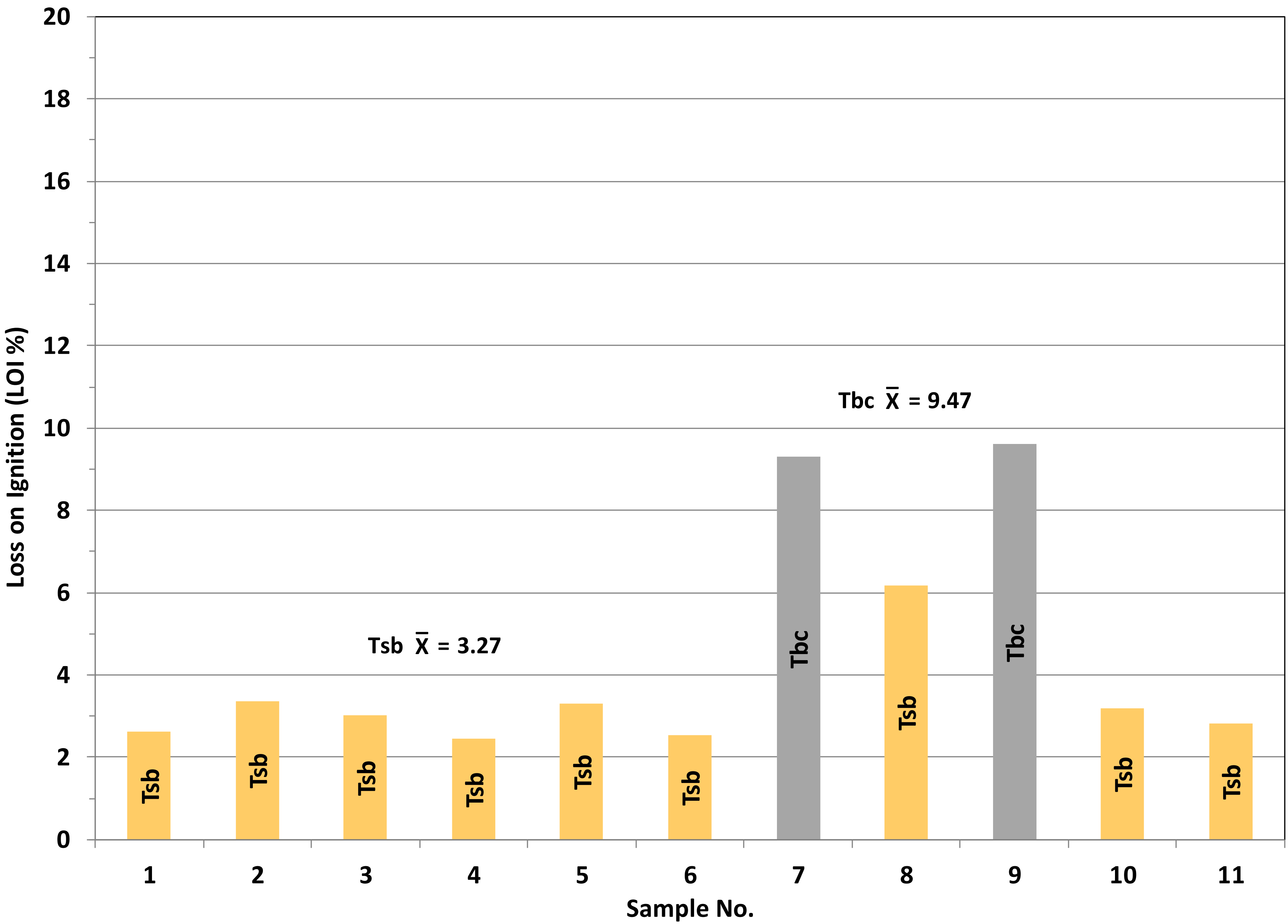

Figure 12. Comparison of loss on ignition (LOI) values as a proxy for detrital lignite content for sandstone samples from the Sentinel Butte (Tsb Sample Nos. 1 - 6, 8, & 10 - 11) and Bullion Creek Formations (Tbc Sample Nos. 7 & 9) in western North Dakota.

Table 7. Bulk Compositional Mineralogical Summary by XRD[1]

Sample No.	Quartz (%)	Feldspars (%)	Carbonates (%)	Clays (%)
1	50	25	6	19
2	49	35	3	13
3	42	35	13	10
4	45	42	2	11
5	54	36	4	6
6	51	40	2	7
7	63	10	17	10
8	55	19	15	11
9	55	11	24	10
10	41	19	14	26
11	57	27	2	14

[1] *Unwashed (Bulk) Sample Analysis on Crystalline Phase Constituents.*

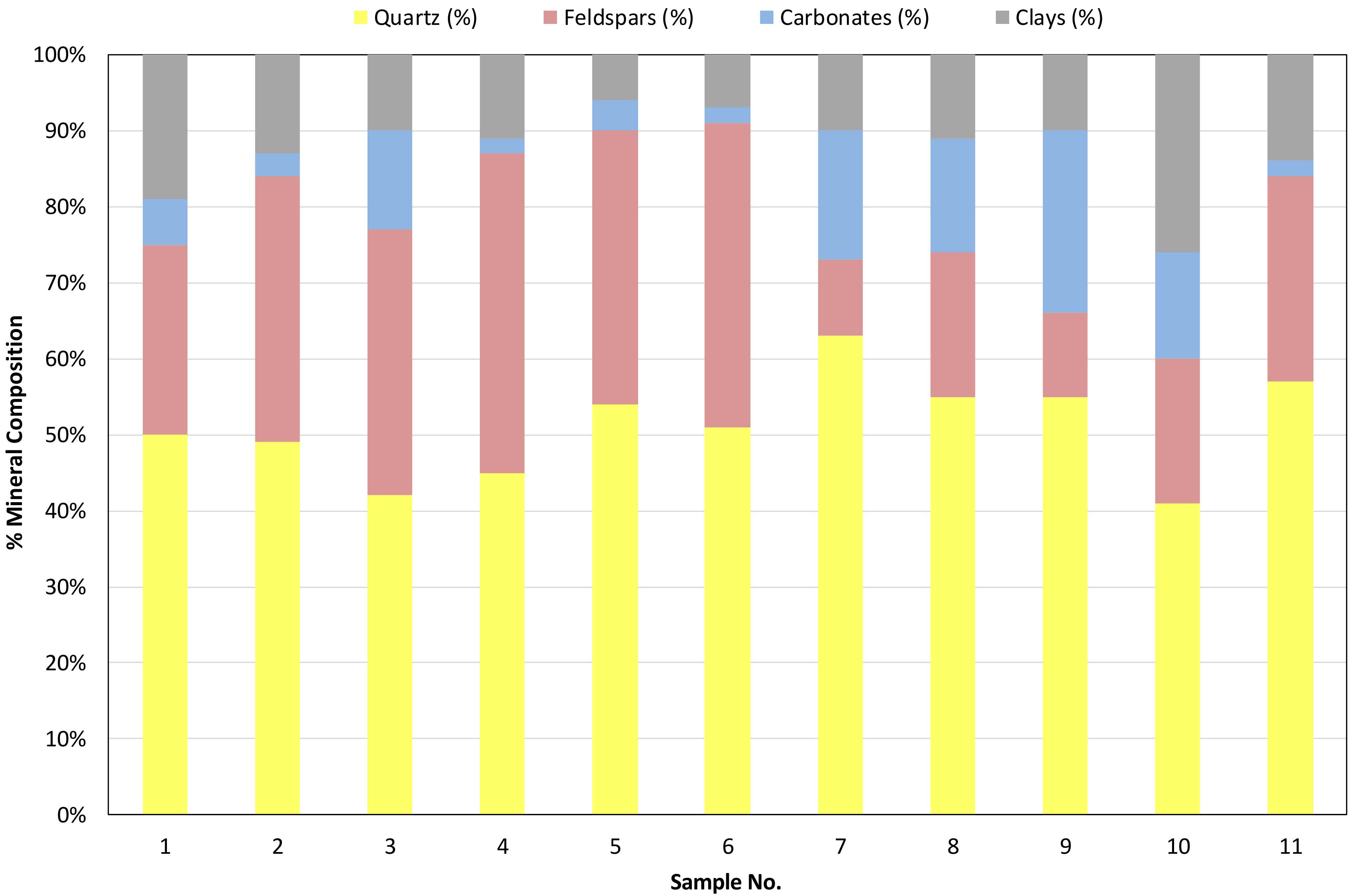

Figure 13. Summary X-ray diffraction (XRD) mineralogical analyses for sandstones of Sentinel Butte (Sample Nos. 1 - 6, 8, and 10 - 11) and Bullion Creek (Sample Nos. 7 & 9) Formations in North Dakota.

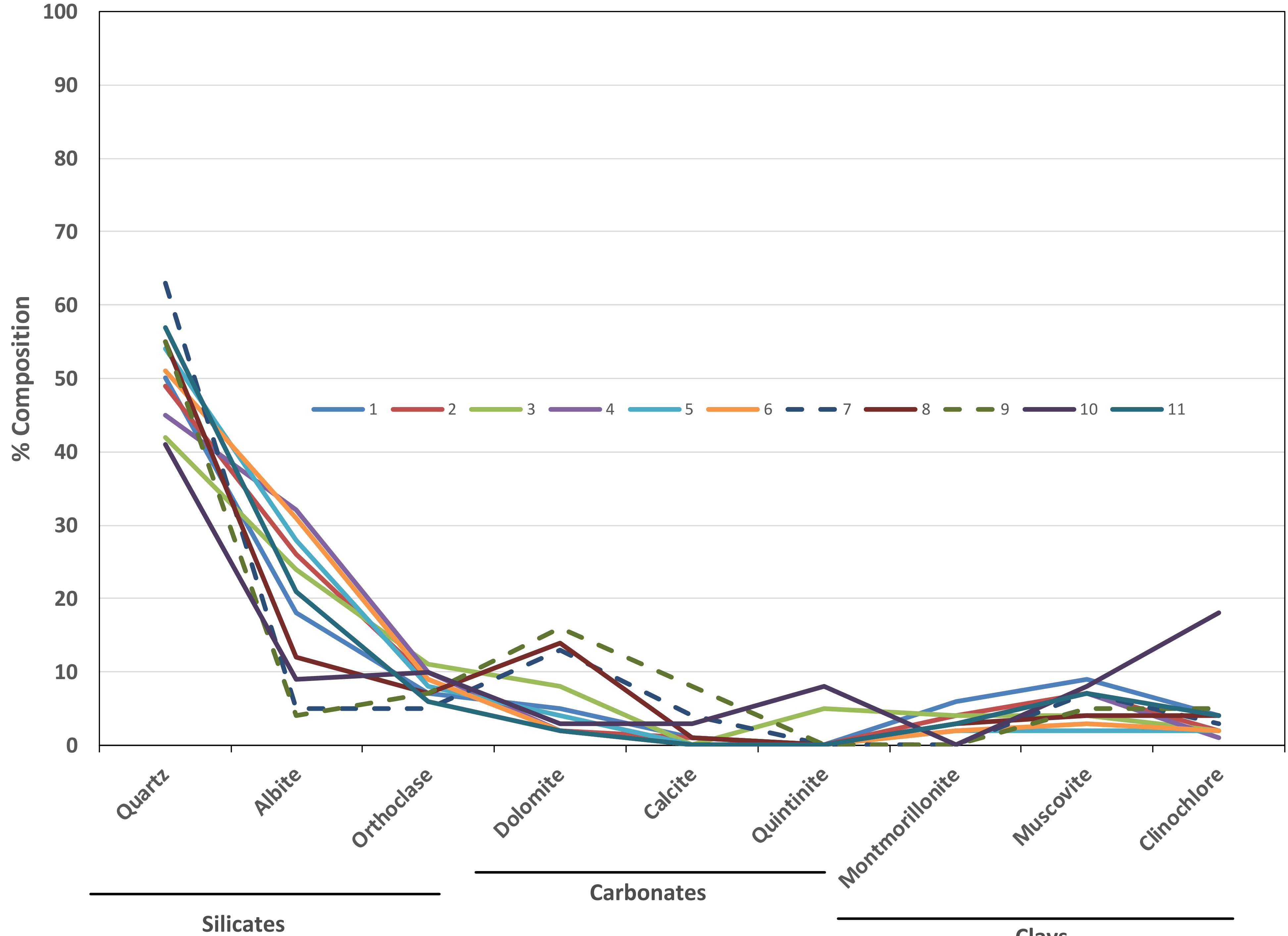

Figure 14. X-ray diffraction (XRD) mineralogical analyses for sandstones of the Sentinel Butte (Sample Nos. 1-6, 8, 10 & 11) and Bullion Creek (Sample Nos. 7 & 9) Formations in North Dakota.

CONCLUSIONS

Sentinel Butte and Bullion Creek Formations in North Dakota have a favorable grain size range that consists of 100 mesh sands and are generally uniform in textural composition. However, the results of this study suggest these sandstones are not likely to be suitable for use as natural sand proppant due to their variable mineralogy (containing relatively low amounts of quartz and high amounts of carbonates and clays), high degrees of sand grain angularity, and low resistance to crush (less than 3K). Vertical stratigraphic profiling on the channel sandstone of the Sentinel Butte Formation in northwestern Billings County resulted in relatively consistent results throughout the outcrop, suggesting little stratigraphic differentiation and overall consistency in depositional environment at this location. The relatively high concentrations of albite within the Sentinel Butte Formation sandstones are likely lowering overall crush resistance. The testing data in this report should prove valuable for other potential industrial sand uses and future sedimentological research.

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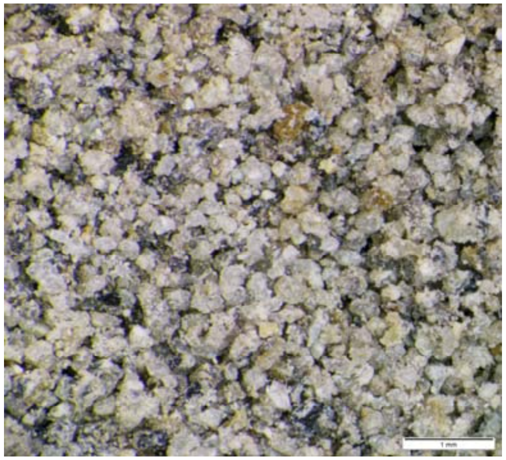

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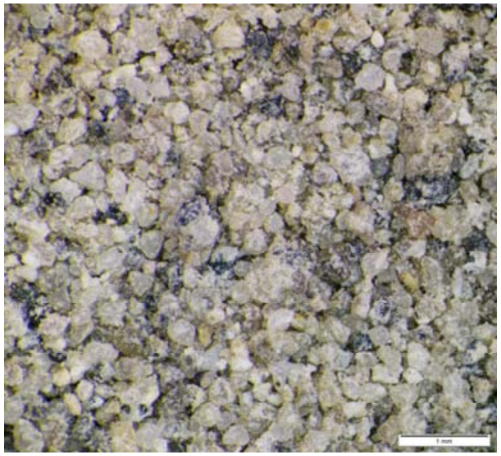

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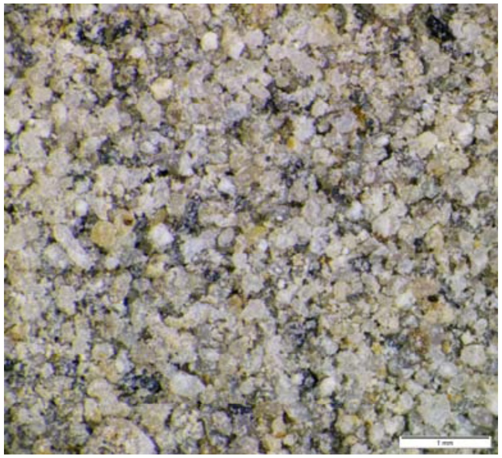

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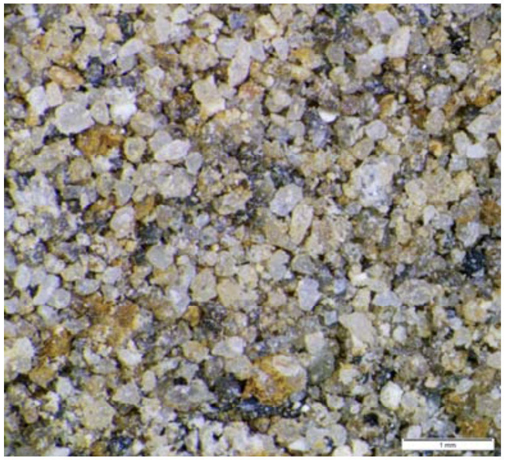

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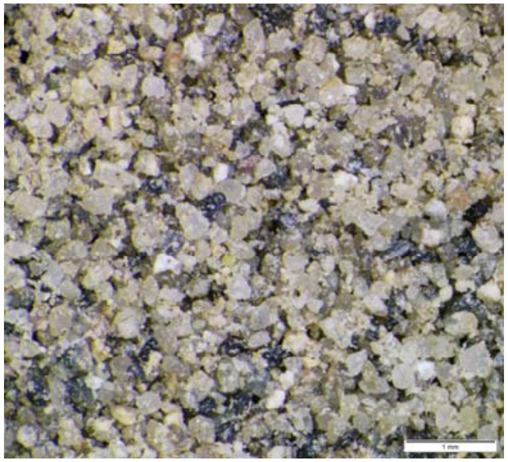

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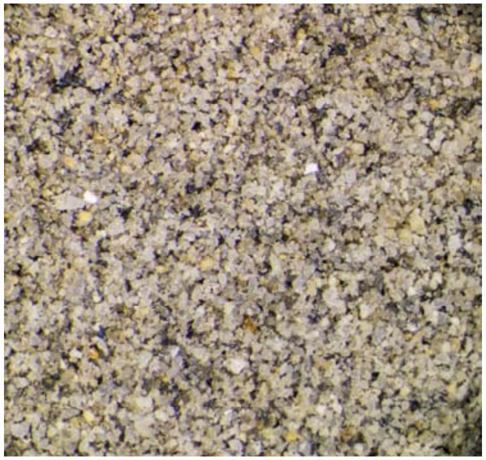

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Plate I. Bulk sand (25x) photo -micrographs of sandstone samples from the Sentinel Butte (Sample Nos. 1 - 6, 8, & 10 - 11) and Bullion Creek Formations (Sample Nos. 7 & 9) in western North Dakota. Quartz grains tend to be clear to opaque, dark-colored grains can be feldspars, detrital lignite, or sand grains from the weathering of igneous and metamorphic rocks.

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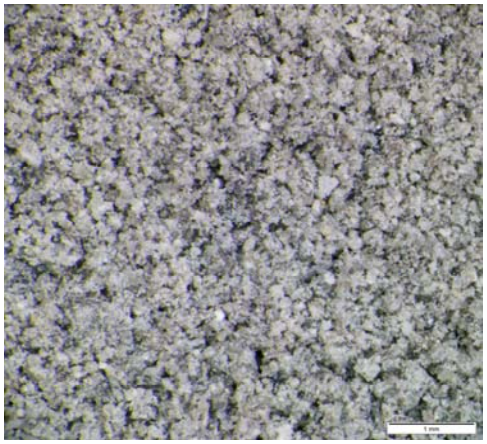

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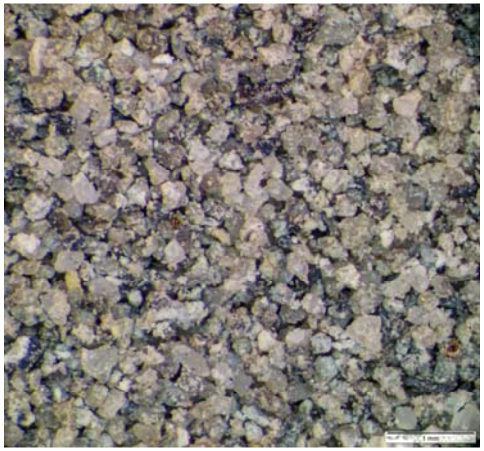

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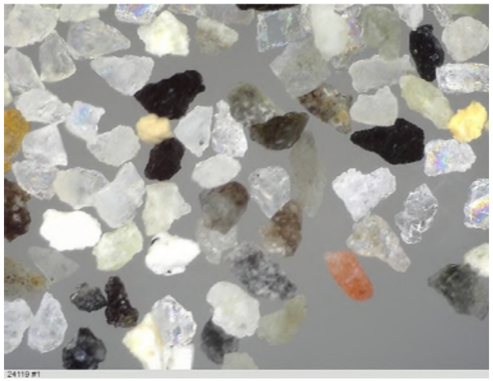
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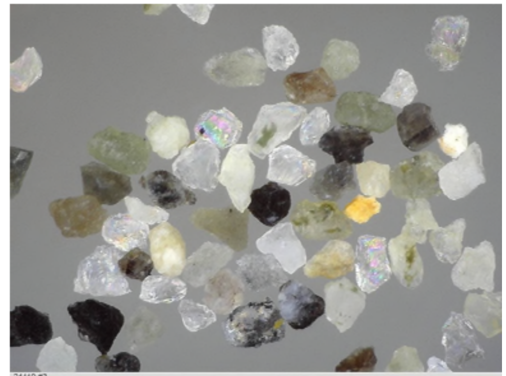
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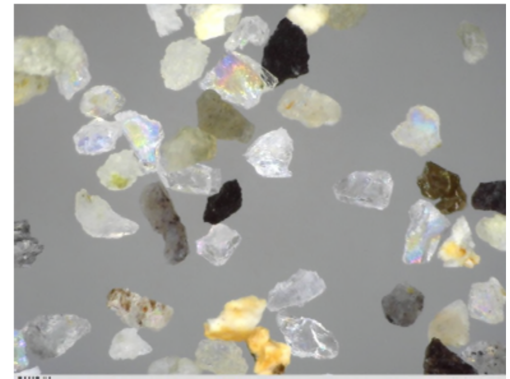
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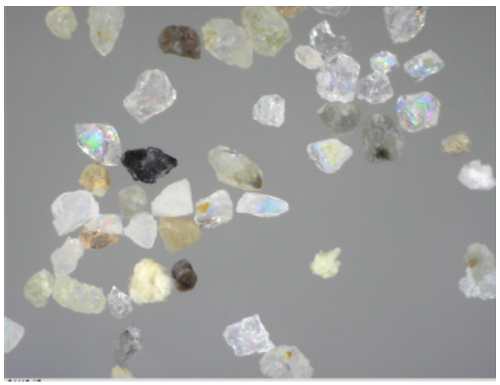
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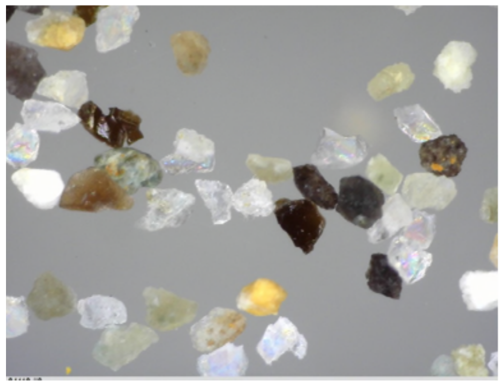
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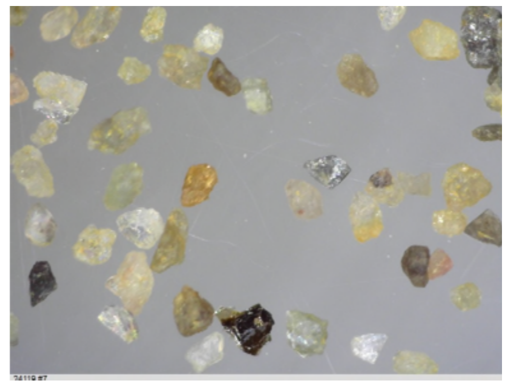
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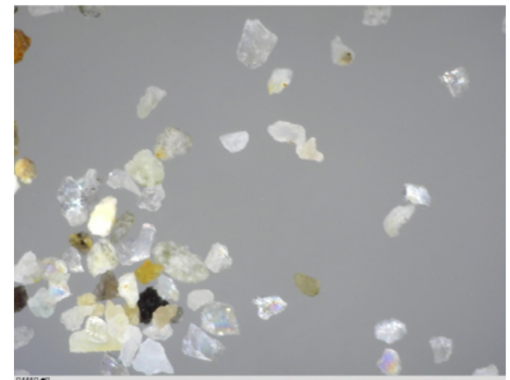
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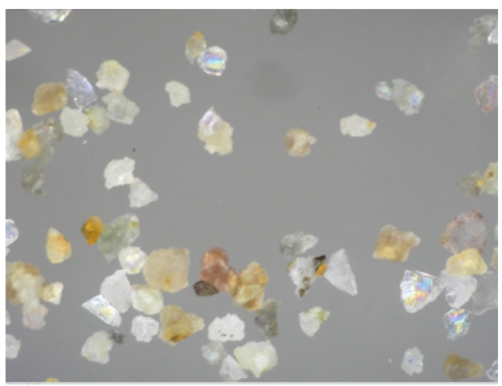
Plate II. Sand grain monolayer photomicrographs of sandstone samples (70/140) from the Sentinel Butte (Sample Nos. 1 - 6, 8, & 10 - 11) and Bullion Creek Formations (Sample Nos. 7 & 9) in western North Dakota. Quartz grains tend to be clear to opaque, dark-colored grains can be feldspars, detrital lignite, or sand grains from the weathering of igneous and metamorphic rocks.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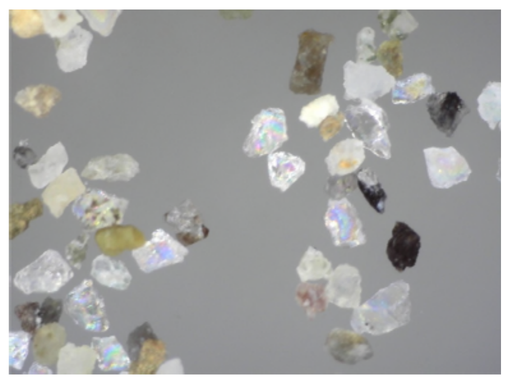
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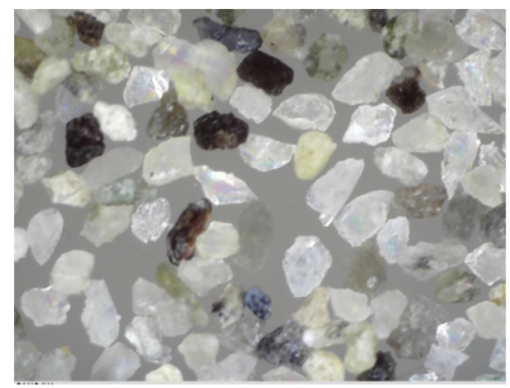
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Appendix A - XRD Mineralogical Analysis of Sandstone from the Sentinel Butte and Bullion Creek Formations in North Dakota

XRD-Methodology

Sand (unwashed) from each sample of sandstone was ground with a mortar and pestle, loaded into a standard sample cup, and placed into a Panalytical X'pert MPD diffractometer using Cu radiation at 45KV/40ma. Scanning was run over the range of 5° - 80° with a step size of 0.0131° and a counting time of 250 sec/step. After the diffraction pattern was obtained, phase identification was completed with the aid of the Powder Diffraction File published by the International Centre for Diffraction Data or the Inorganic Crystal Structure Database. These databases represent the world's largest collection of XRD data. The experimental patterns shown in Figures A-1-a through A-11-a are all at full scale along with stick patterns representing the identified mineralogical phases (Table 1). Expanded views, shown in Figures A-1-b through A-11-b, more clearly display the minor mineralogical phases. The identified crystalline phases were further quantified with the aid of a Rietveld refinement which more precisely defines the diffraction phase diagram peak heights and position which provides for greater accuracy in the mineralogical quantification (Table 2).

It should be noted that these sandstones were found to contain relatively large amounts of clay minerals which result in diffraction patterns with a high degree of preferred orientation and poorly defined peaks. The specific identification and quantification of clay mineralogy requires additional orientation and glycolation procedures that were not performed as a part of this work. Consequently, the clay mineralogy values presented here should be viewed as qualitative mineralogical estimates as, for example, muscovite could also be biotite or another form of mica.

Table 1. Qualitative Bedrock Sandstone Mineralogy by XRD Analysis[1]

Major	Moderate	Minor	Trace	Comments
Quartz	*Albite* *Orthoclase*	*Dolomite* *Muscovite* *Clinochlore* *Montmorillonite* *Calcite*	*Quintinite*	*Glycolation and specific orientation procedures not performed for clay mineralogy*

[1] *Unwashed (Bulk) Sample Analysis.*

Table 2. Quantitative Bedrock Sandstone Mineralogy by XRD Analysis[2]

Sample No.	Quartz	Dolomite	Calcite	Quintinite	Albite	Orthoclase	Montmorillonite	Muscovite	Clinochlore
1	50	5	1	--	18	7	6	9	4
2	49	2	1	<1	26	9	4	7	2
3	42	8	<1	5	24	11	4	4	2
4	45	2	<1	--	32	10	3	7	1
5	54	4	<1	--	28	8	2	2	2
6	51	2	<1	--	31	9	2	3	2
7	63	13	4	--	5	5	--	7	3
8	55	14	1	--	12	7	3	4	4
9	55	16	8	--	4	7	--	5	5
10	41	3	3	8	9	10	--	8	18
11	57	2	--	--	21	6	3	7	4

[2] *Unwashed (Bulk) Sample Analysis on Crystalline Phase Constituents in Weight % with Rietveld Refinement.*

-- Mineral not detected.

Mineral Name	Chemical Composition
Quartz	SiO_2
Dolomite	$CaMg(CO_3)_2$
Calcite	$Ca(CO_3)$
Quintinite	$((Mg_4Al_2(OH)_{12})(CO_3)(H_2O)_3$
Montmorillonite	$Al_2Si_4O_{10}(OH)_2(H_2O)_{10}$
Albite	$NaAlSi_3O_8$
Orthoclase	$KAlSi_3O_8$
Muscovite	$KAl_2(AlSi_3O_{10})(F,OH)_2$
Clinochlore	$(Mg_5Al)(AlSi_3)O_{10}(OH)_8$

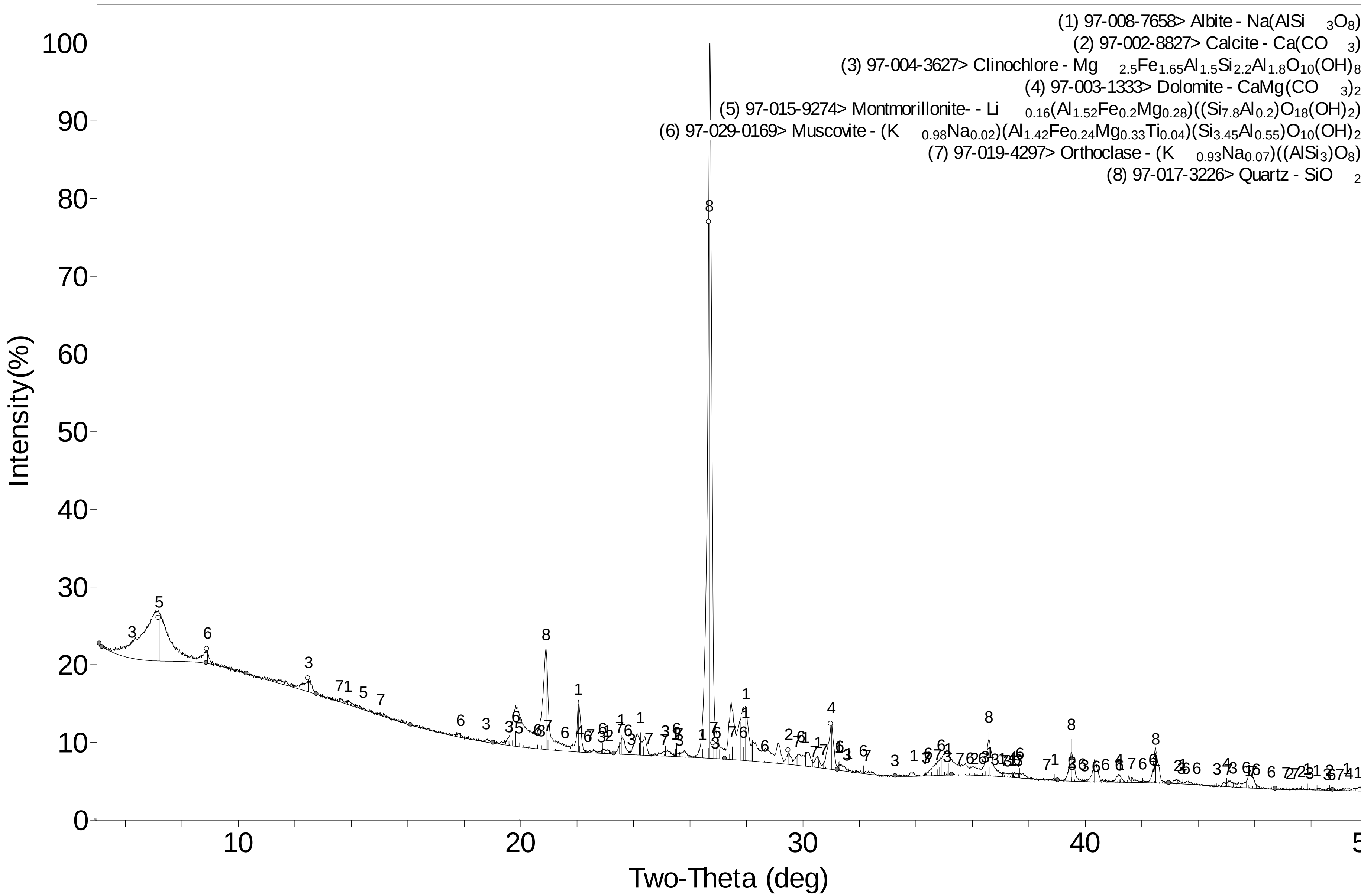

Figure A-1. XRD phase diagram for sandstone sample no. 1 from the Sentinel Butte Formation in northwestern Billings County, North Dakota.

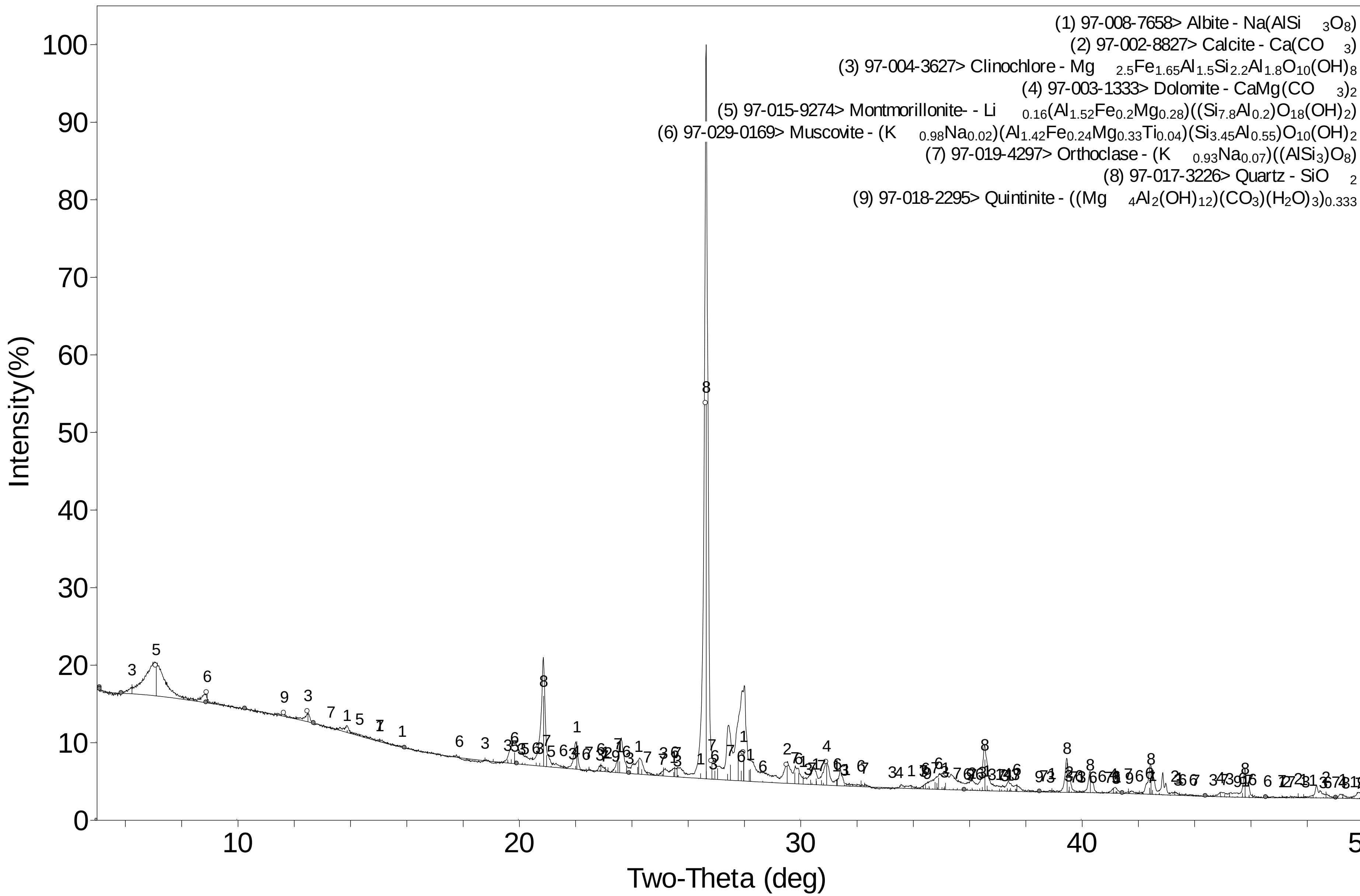

Figure A-2. XRD phase diagram for sandstone sample no. 2 from the Sentinel Butte Formation in northwestern Billings County, North Dakota.

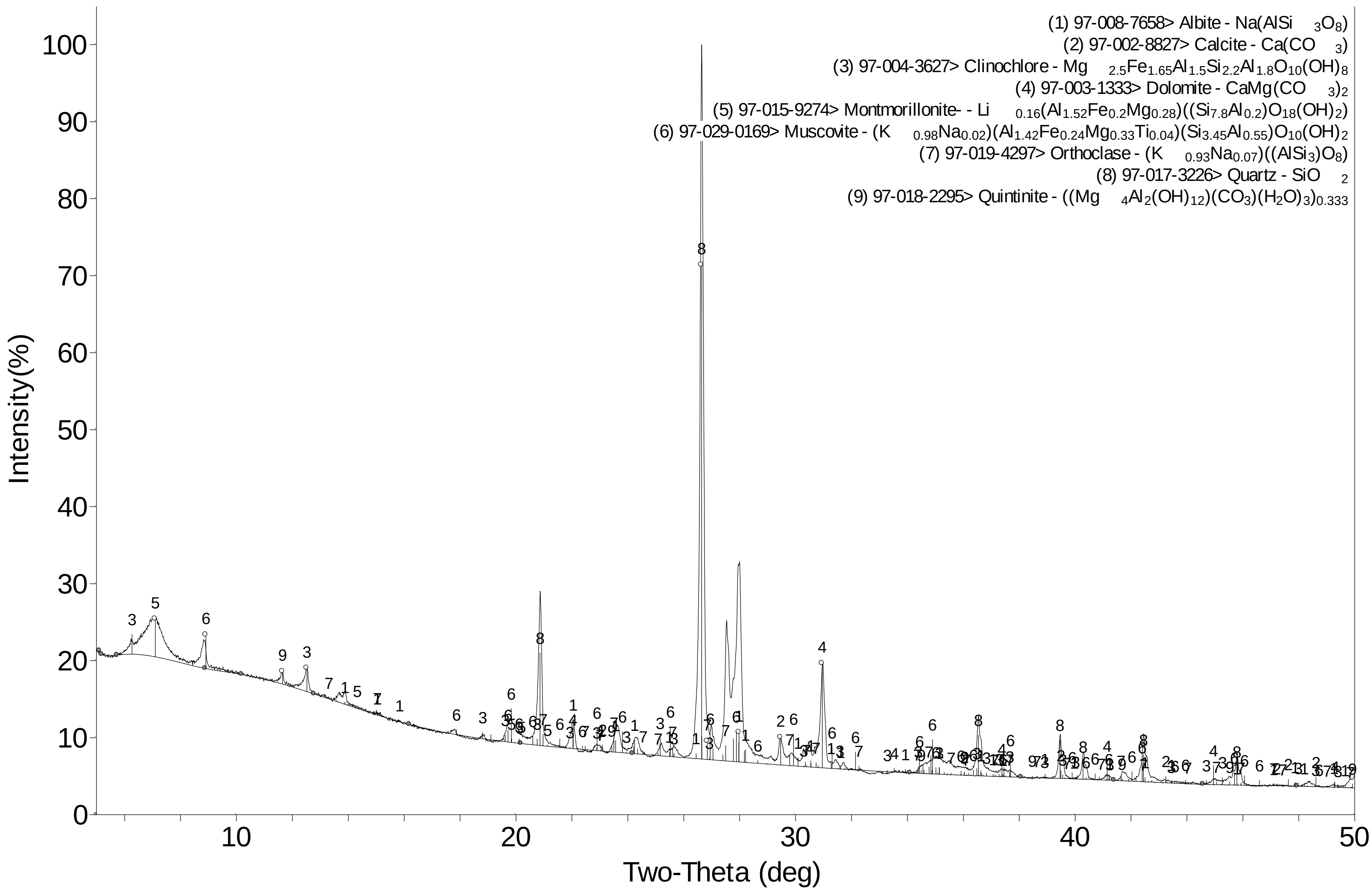

Figure A-3. XRD phase diagram for sandstone sample no. 3 from the Sentinel Butte Formation in northwestern Billings County, North Dakota.

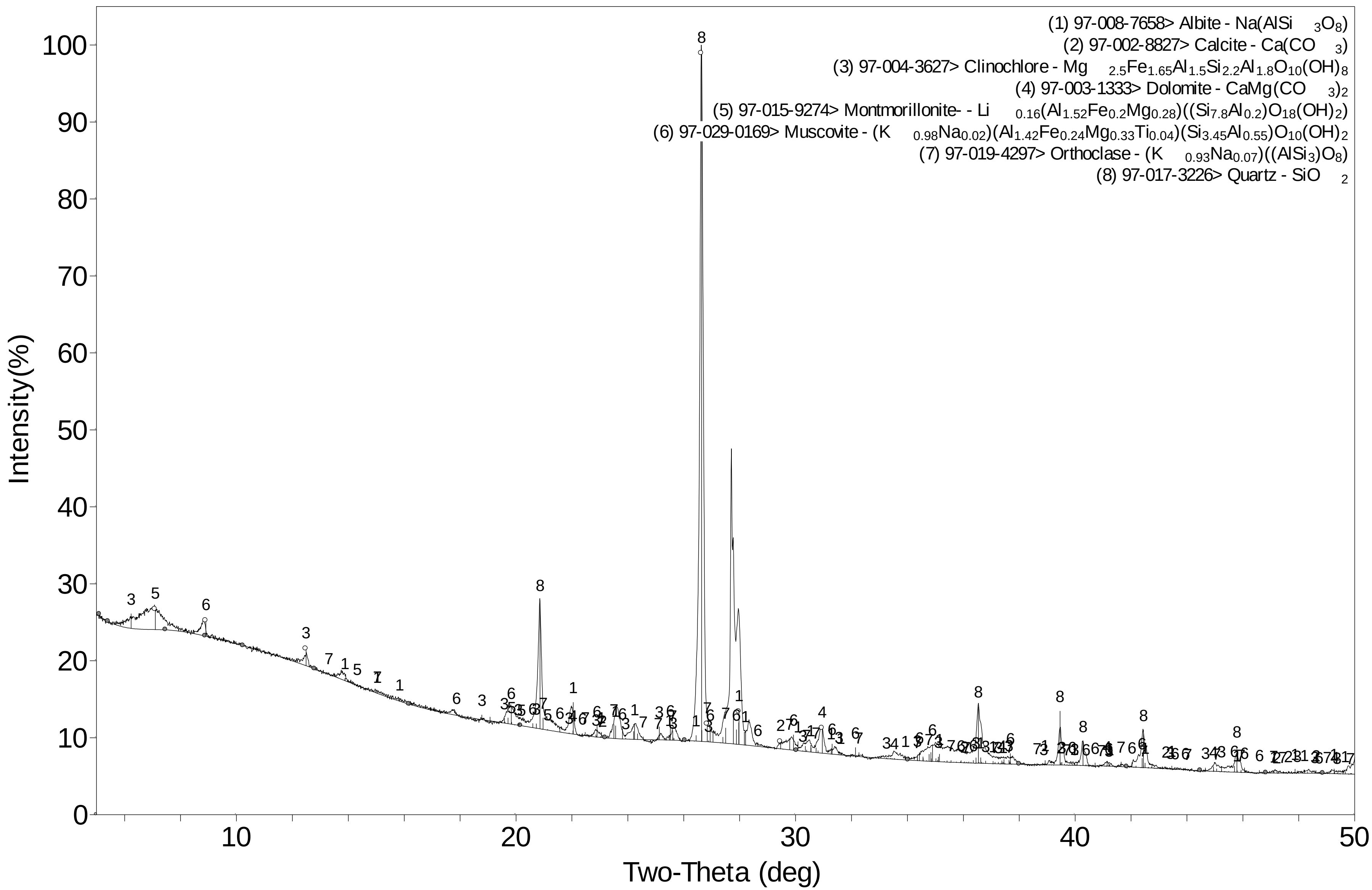

Figure A-4. XRD phase diagram for sandstone sample no. 4 from the Sentinel Butte Formation in northwestern Billings County, North Dakota.

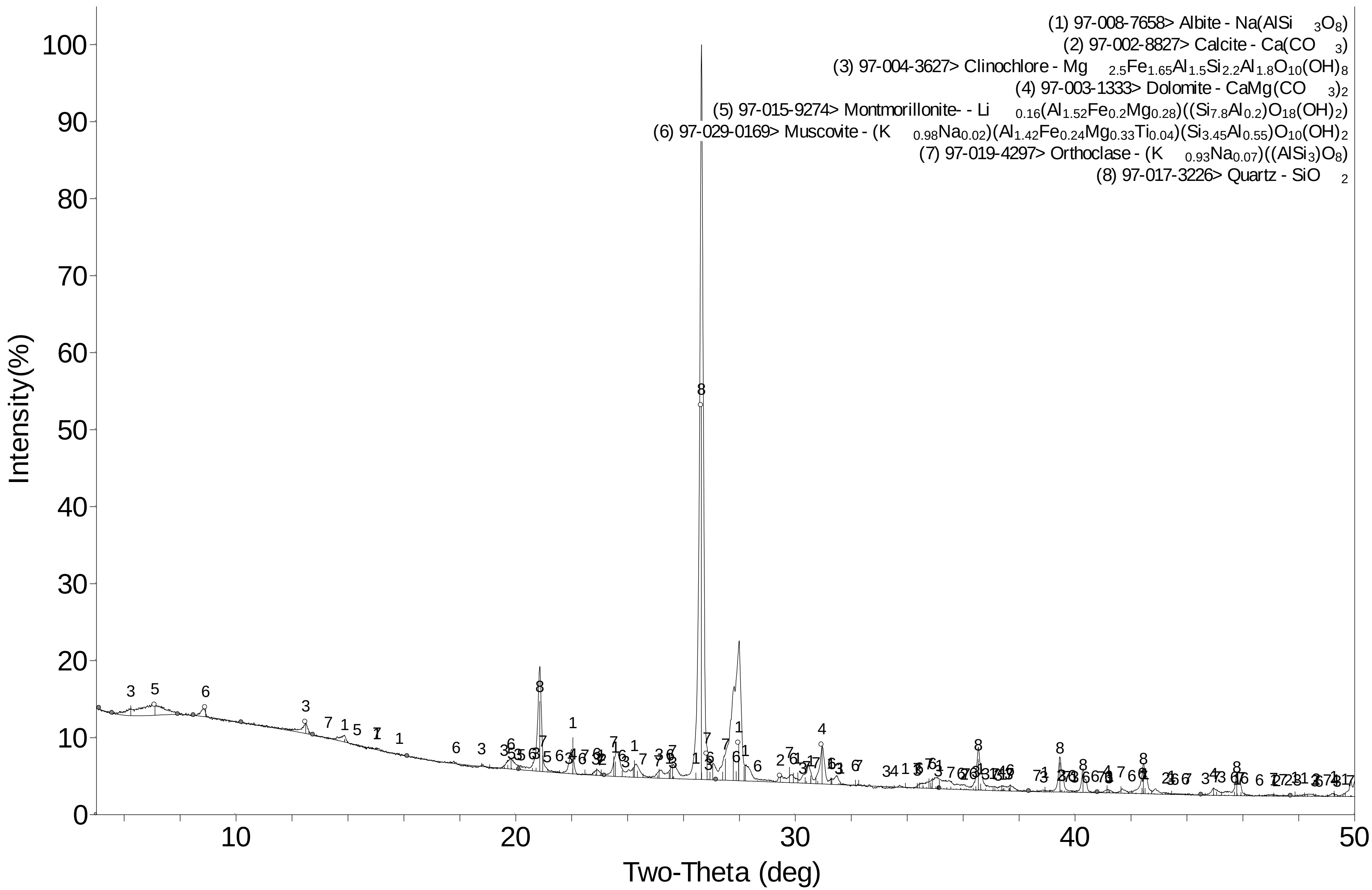

Figure A-5. XRD phase diagram for sandstone sample no. 5 from the Sentinel Butte Formation in northwestern Billings County, North Dakota.

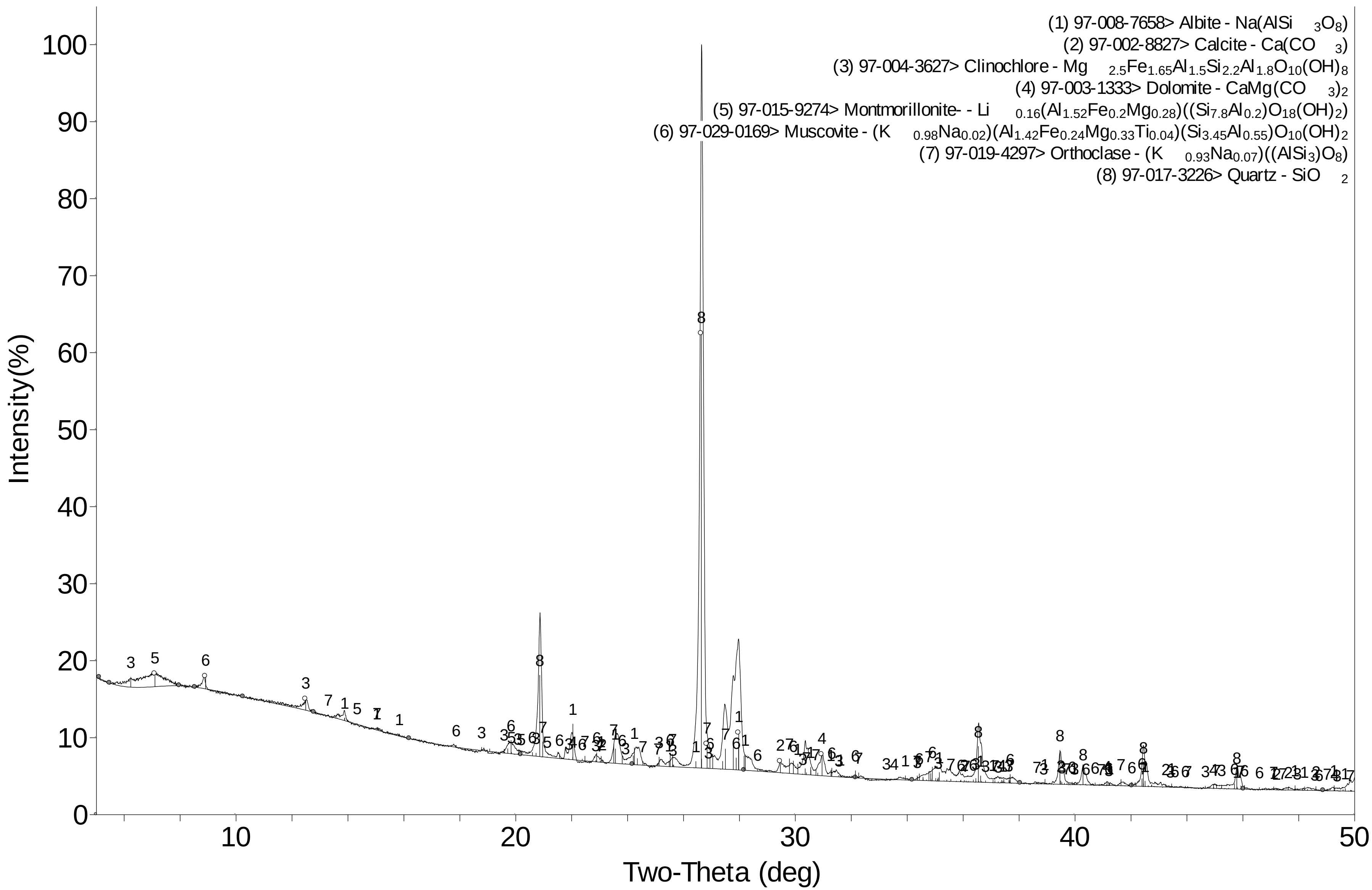

Figure A-6. XRD phase diagram for sandstone sample no. 6 from the Sentinel Butte Formation in northwestern Billings County, North Dakota.

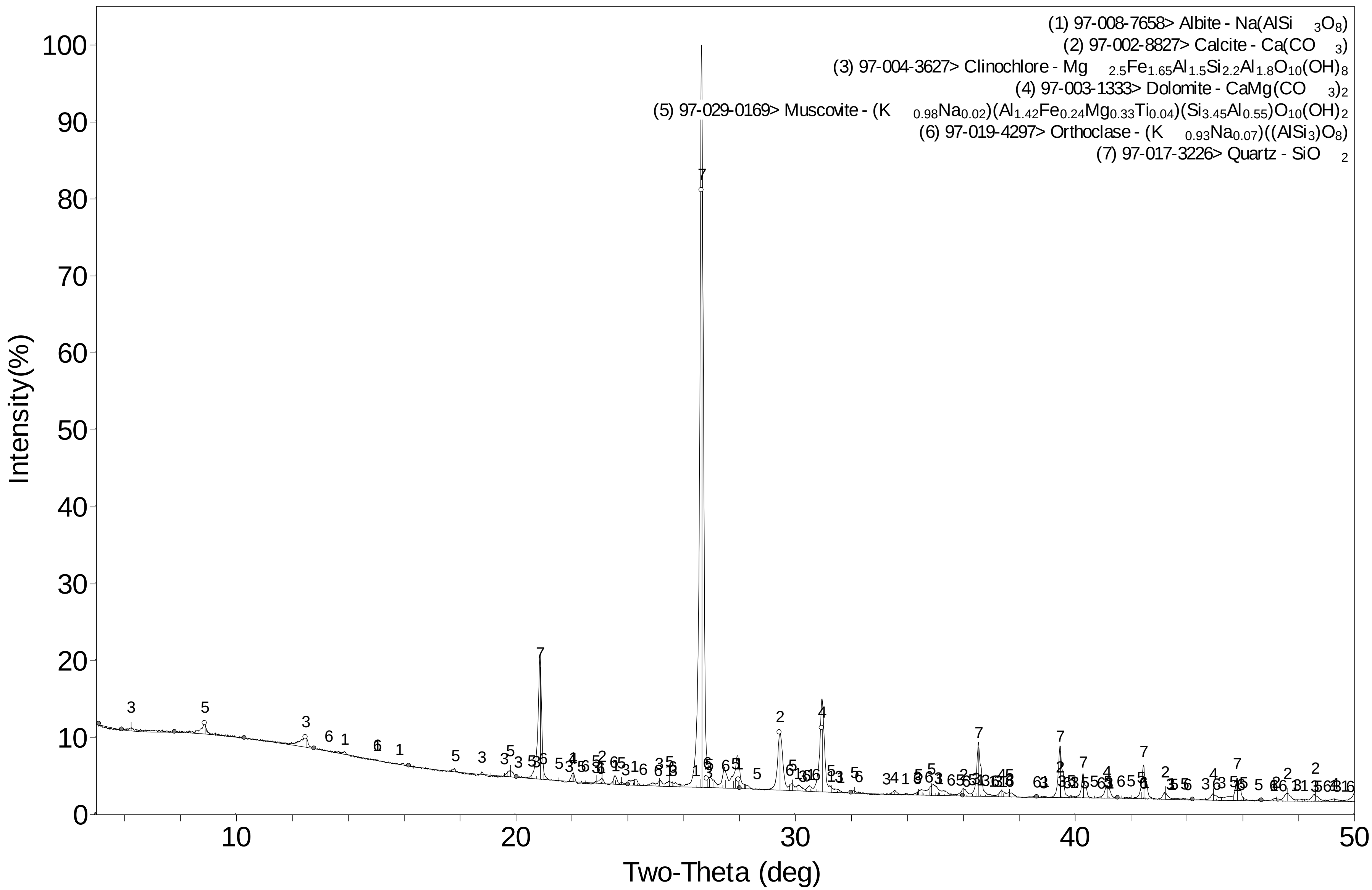

Figure A-7. XRD phase diagram for sandstone sample no. 7 from the Bullion Creek Formation in northwestern Billings County, North Dakota.

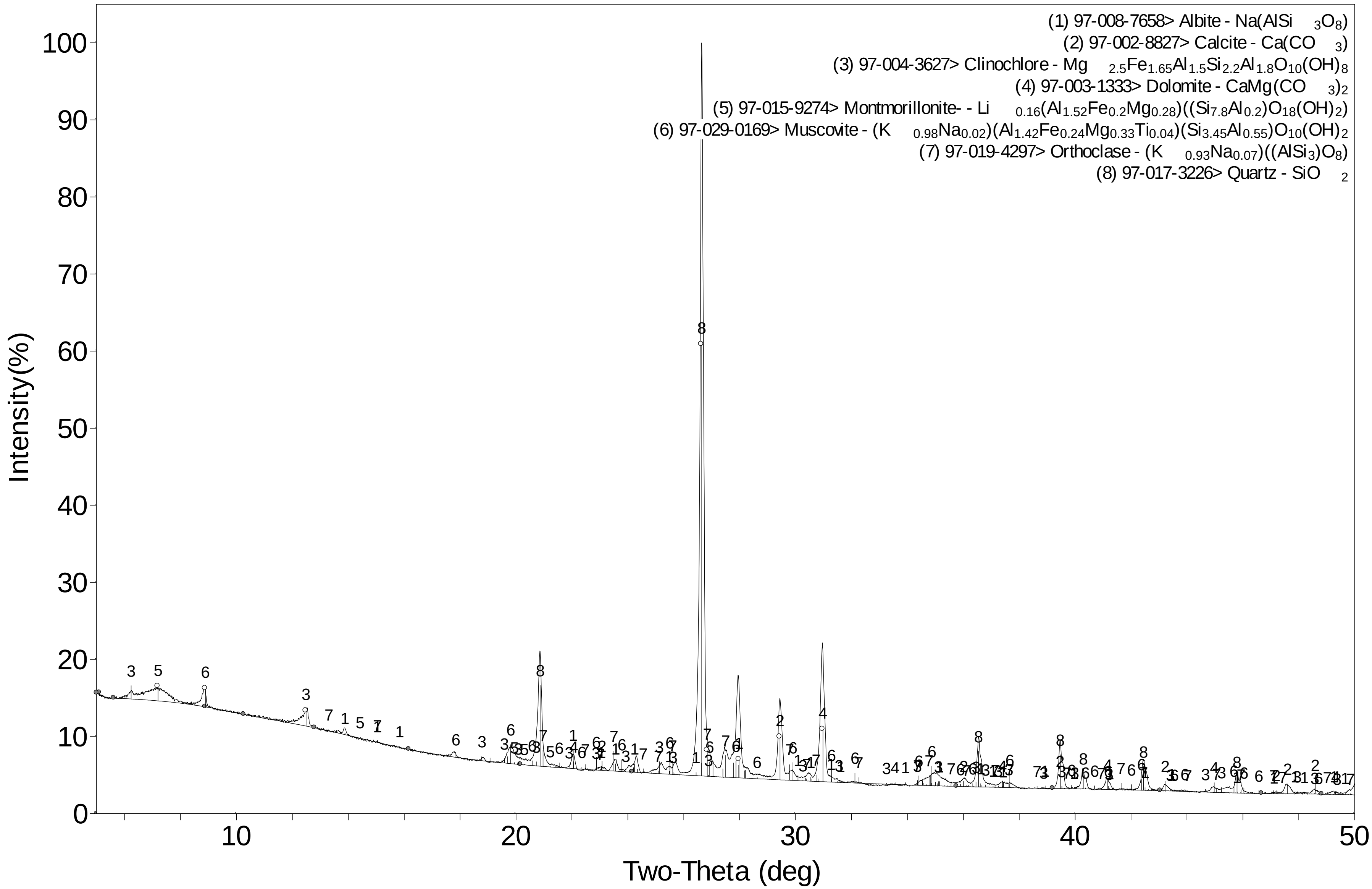

Figure A-8. XRD phase diagram for sandstone sample no. 8 from the Sentinel Butte Formation in central McKenzie County, North Dakota.

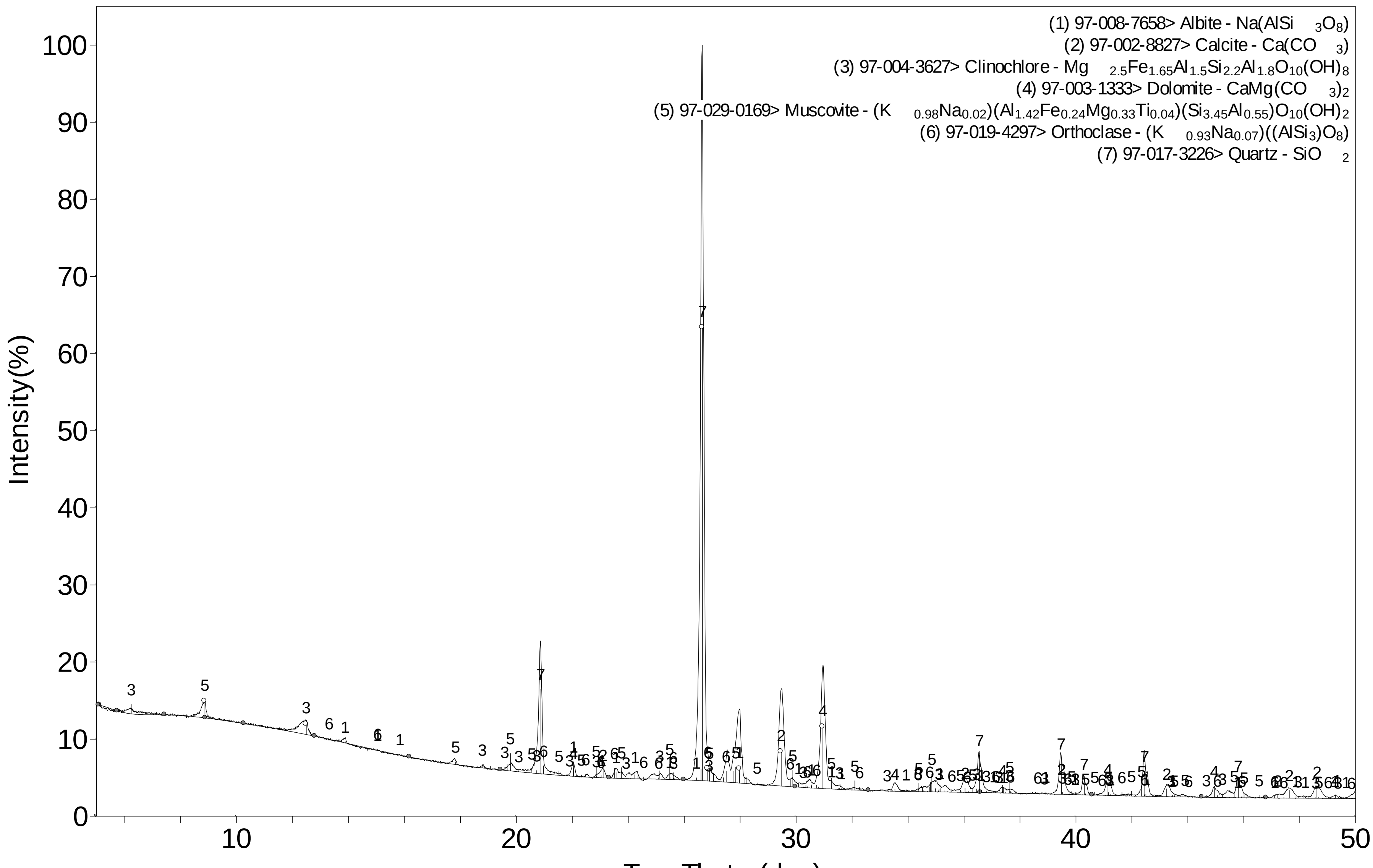

Figure A-9. XRD phase diagram for sandstone sample no. 9 from the Bullion Creek Formation in northwestern Slope County, North Dakota.

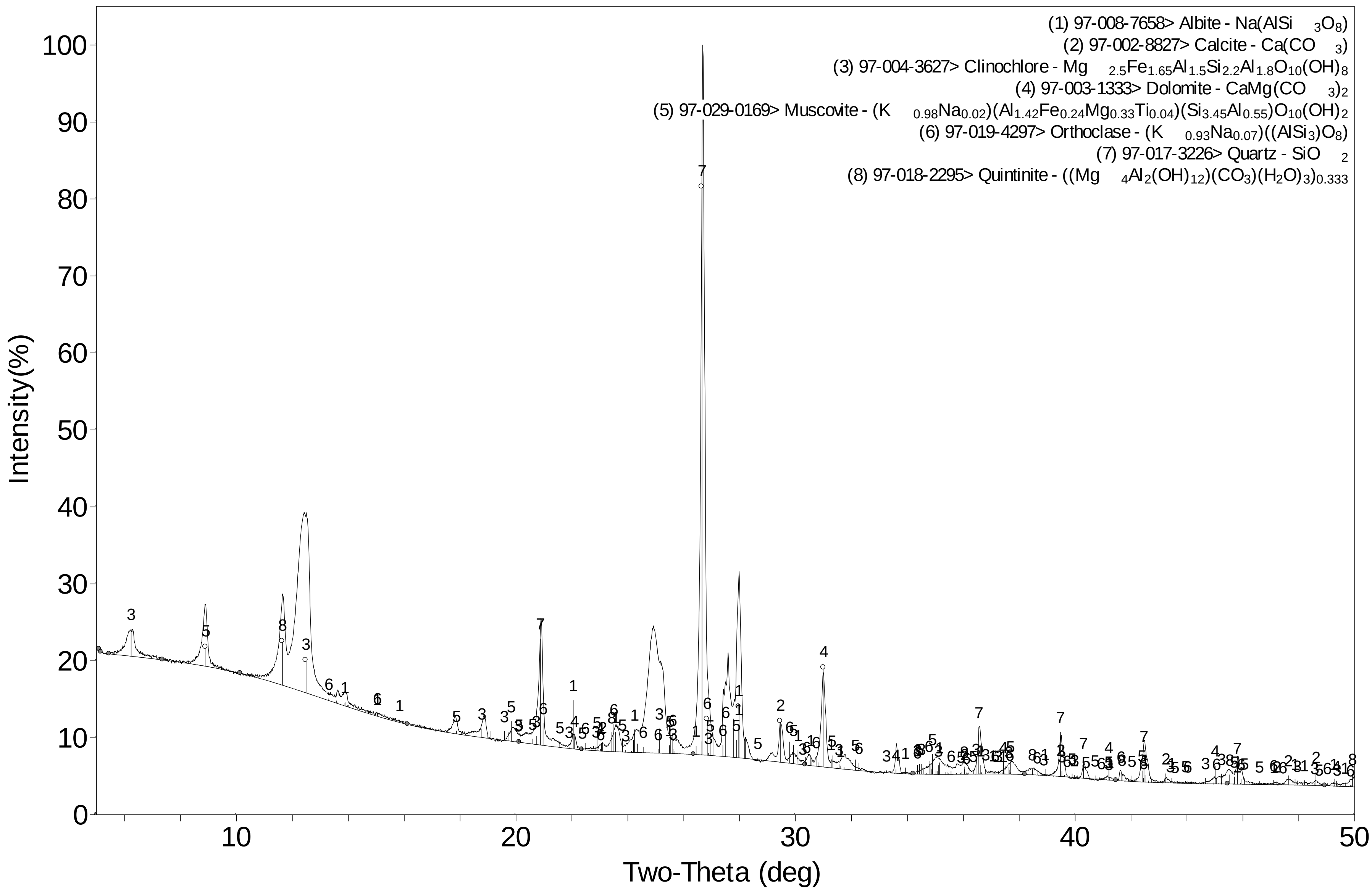

Figure A-10. XRD phase diagram for sandstone sample no. 10 from the Sentinel Butte Formation in southeastern McLean County, North Dakota.

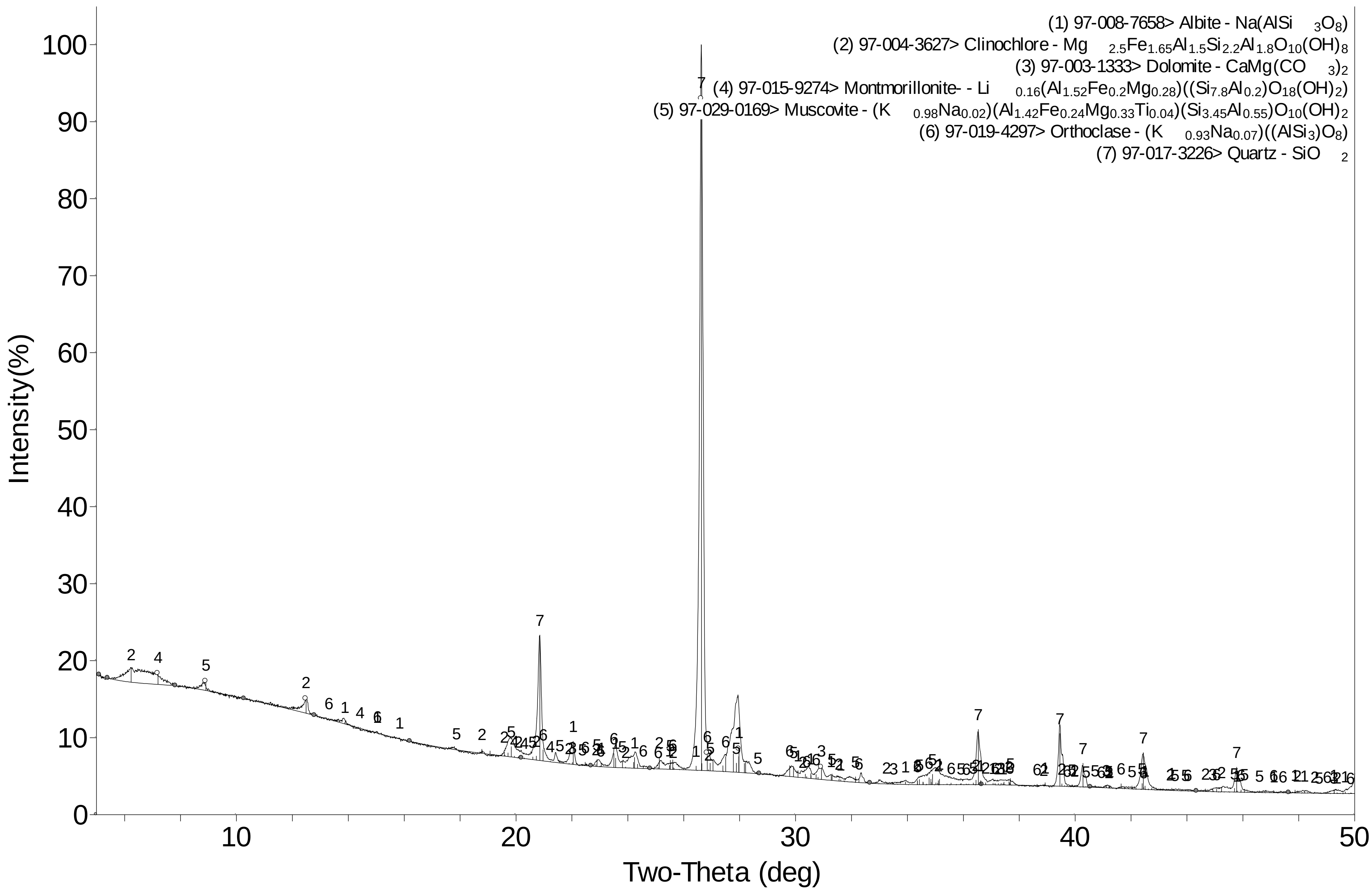

Figure A-11. XRD phase diagram for sandstone sample no. 11 from the Sentinel Butte Formation in central Mercer County, North Dakota.