SANDSTONES OF THE FOX HILLS AND HELL CREEK FORMATIONS IN NORTH DAKOTA:
Proppant Testing and Characterization

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NORTH DAKOTA GEOLOGICAL SURVEY

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ABSTRACT

Sampling and testing of sandstone deposits of the Fox Hills and Hell Creek Formations in southwestern and south-central North Dakota were completed in 2022 to characterize these deposits for potential use as proppant in the hydraulic fracturing of oil and gas wells in the Williston Basin. Proppant sand testing was performed on eight samples from four areas where sandstones of these formations are best exposed. Sandstones of the Colgate and Bullhead Members of the Fox Hills Formation and from the lower Hell Creek Formation were tested as these represented the most likely deposits for proppant development which are consistently well sorted, medium to very-fine grained, with quartz contents averaging 67%, with crush resistance ranging from 1,000 psi (1K) to 5,000 psi (5K) in the 70/140, and 40/140 size classes. Acid solubility averaged 9.1% and turbidity 106 NTU with a very low loss on ignition (LOI) values averaging 0.7% suggesting a low presence of detrital lignite and deleterious mineralogy. Sand grain particle shape factors approach but are below the desired ranges specified for proppant use and average 0.5 for roundness and 0.8 for sphericity. Mineralogically, carbonates were not found in the washed and sized samples which averaged 60% quartz, with 20% feldspars (albite), and 10% clays (illite). Based on current industry proppant sand requirements these sandstones would be of lower overall quality as compared to the windblown sand deposits currently being produced for proppant sand use from the Hazen-Stanton and Denbigh Dunes.

ACKNOWLEDGMENTS

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AUTHOR'S NOTE

Recently the oil and gas industry has relaxed proppant testing specifications in parts of the U.S. in favor of more regional or local proppant sand source utilization. In-basin proppant sand is currently being produced from windblown sands in the Hazen-Stanton Dunes in Mercer County and Denbigh Dunes in McHenry County. This report characterizes selected sandstones of the Fox Hills and Hell Creek Formations which were considered potential candidates for development as proppant sand.

Cover photo: Outcrop of the Fox Hills and Hell Creek Formations along the Little Missouri River in western Bowman County, North Dakota. Sandstone samples Nos. 6 – 8 from this report were collected at this outcrop.

INTRODUCTION

A renewed interest in the possibility of locating in-basin proppant sand resources in North Dakota started in 2018 as falling oil prices forced companies to take a closer look at all aspects of the development equation. Local proppant sand sourcing was identified as an area for potential savings in the Williston Basin, as was currently being realized in other shale basins in the U.S., e.g., the Permian Basin in Texas. As a result, the Survey embarked on renewed exploration for potential proppant sand deposits that were closer to the heart of oil development activity across North Dakota.

Sandstone deposits in the Fox Hills Formation, in particular the Colgate Member, were originally considered by field geologists to have a high potential for use as natural proppant sand based on the sedimentologic characteristics exhibited on the outcrop. These sandstones, best exposed in southwestern and south-central North Dakota, have commonly been described as massive, well-sorted, fine-grained sandstones with seemingly high quartz contents at the macroscopic scale. Upon closer inspection, these sandstones are of lesser overall quality, with respect to proppant use, than the windblown deposits identified by recent investigations in the Hazen-Stanton and Denbigh Dunes in Mercer and McHenry Counties, respectively (Anderson, 2020). Since most of the bedrock sandstones deemed to be viable candidates for proppant development were previously investigated (Anderson, et al., 2019), it was determined that finishing out the evaluation of North Dakota sandstones by evaluating samples from the Fox Hills and Hell Creek Formations would be logical and worthwhile. At a minimum, additional sedimentologic characterization of these sandstones helps to better understand the geologic history and depositional environments of these deposits.

Sampling and testing efforts for these units were concentrated in western Bowman County and southeastern Morton Counties as these are the areas where the units are best exposed and readily accessible. Eight sandstone samples from eight different locations were collected during the 2022 field season and submitted to Lonquist & Co., LLC's frac sand testing lab in College Station, Texas, for proppant characterization. Bulk sand samples were collected on the outcrop and placed into five-gallon buckets at selected sandstone outcrop locations (Samples No. 1 - 8, this report). Samples were collected from within selected outcrops (generally two to five feet beneath surface exposure) using a battery-powered hand drill with a sand bucket auger attachment.

Sandstone samples were evaluated for proppant suitability in accordance with testing standards and specifications published by the American Petroleum Institute (API): API STD-19C (API, 2018) and the International Organization for Standardization (ISO): ISO 13503-2 (ISO, 2006). Testing included: gross sample inspection and field description (including field acid reactivity), sample washing and comparison, stereo microscope photomicrography, particle shape factors (roundness and sphericity) analysis, qualitative and quantitative mineralogy via X-ray diffraction (XRD), crush resistance, acid solubility, turbidity, loss on ignition, and sand density testing. Tests were again performed on the most abundant size classes, as determined by sieve analysis, which typically fell into the 40/140 and 70/140 size classes. Over the past decade, desired sand specifications have changed with continued refinements in the hydraulic fracturing process. Sand in the coarser size classes (e.g., 30/50) was originally preferred, which over time has changed to the finer sand size classes in the 40/70 and 70/140 ranges which generally demonstrate much higher crush resistance than coarser fractions. Also broadening the testing size class range, such as in a 40/140 or 50/140 cut continues to result in higher crush resistance values in some samples and provides useful data over a larger amount of the deposit.

FOX HILLS AND HELL CREEK FORMATION SANDSTONES IN NORTH DAKOTA

Although the majority of the bedrock (and thus the sandstone) exposed at the surface in western North Dakota belongs to the Fort Union Group (Paleocene), older Cretaceous sandstones are exposed in the far southwestern corner of the state (the Little Missouri badlands in western Bowman and Slope counties), as well as the south-central region of the state, with the best outcrops occurring along the Missouri and Cannonball River valleys (Figure 1). Late Cretaceous strata is present in the subsurface across the majority of the Williston basin but is overlain by over a thousand feet (hundreds of meters) of younger strata in the center of the basin and is beneath varying thicknesses of glacial till around its northern and eastern margins, putting its sandstones economically out of reach for any potential mining outside of the aforementioned regions of near-surface exposure.

The Cretaceous section exposed in these areas preserves a late regressive phase of the Western Interior Seaway. Depositional environments transition upward from offshore marine (shale of the Pierre Formation) to nearshore deltaic and shoreline (sandy shales and sandstones of the Fox Hills Formation) to fully terrestrial river and floodplain (sandstones and mudstones of the Hell Creek Formation) (Figure 2). The contact of the Pierre and Fox Hills Formations is gradational, and thus the lowermost intervals of Fox Hills are typically fine-grained (sandy shales and siltstones). Poorly consolidated brown and green-gray clayey sandstones become dominant higher through the basal Trail City Member, containing frequent gray carbonate concretions (Cvancara, 1976). The overlying Timber Lake Member is primarily yellow to reddish-brown clayey sandstone, poorly consolidated but concretions are abundant and well-cemented sandstone and claystone beds become common in upper portions of the member. In central North Dakota, the Bullhead Member consists of buff and brown “banded beds” of sandstone and shale. The Colgate member, a prominent white to light gray medium-grained muddy sandstone, is the uppermost member in western North Dakota and extends as far east as northern Sioux County (Feldmann, 1972). East of there, the Linton Member is the uppermost unit and often forms a heavily indurated gray or brown silty sandstone which caps many buttes through Emmons County (Klett and Erickson, 1976).

The Fox Hills Formation reaches a thickness up to 391 feet (119 meters) in the subsurface in northwestern Hettinger County (Cvancara, 1976), but thins toward the margins of the basin. Cvancara lists outcrop thicknesses as high as 75 feet (23 meters) for the Trail City Member, 128 feet (39 meters) for the Timber Lake Member, 112 feet (34 meters) for the Bullhead lithofacies, 40 feet (12 meters) for the Colgate lithofacies, and 23 feet (7 meters) for the Linton member. The Bullhead and Colgate lithofacies are now recognized as members and the Iron Lightning Member is not recognized in North Dakota (Murphy et al., 2009).

The contact of the Fox Hills with the overlying Hell Creek Formation is generally placed at the first significant carbonaceous bed, denoting the change from marine to terrestrial deposition, but locally channel sandstones may be the first bed present above contact. These sandstones in the Hell Creek Formation range up to 100 feet (30 meters) thick, are typically light gray to light brown and poorly cemented (Murphy et al., 2002). The sandstones analyzed in this report are among these basal channels, but thick channel sandstones also occur near the upper contact with the Ludlow and were investigated by Anderson et al., (2019).

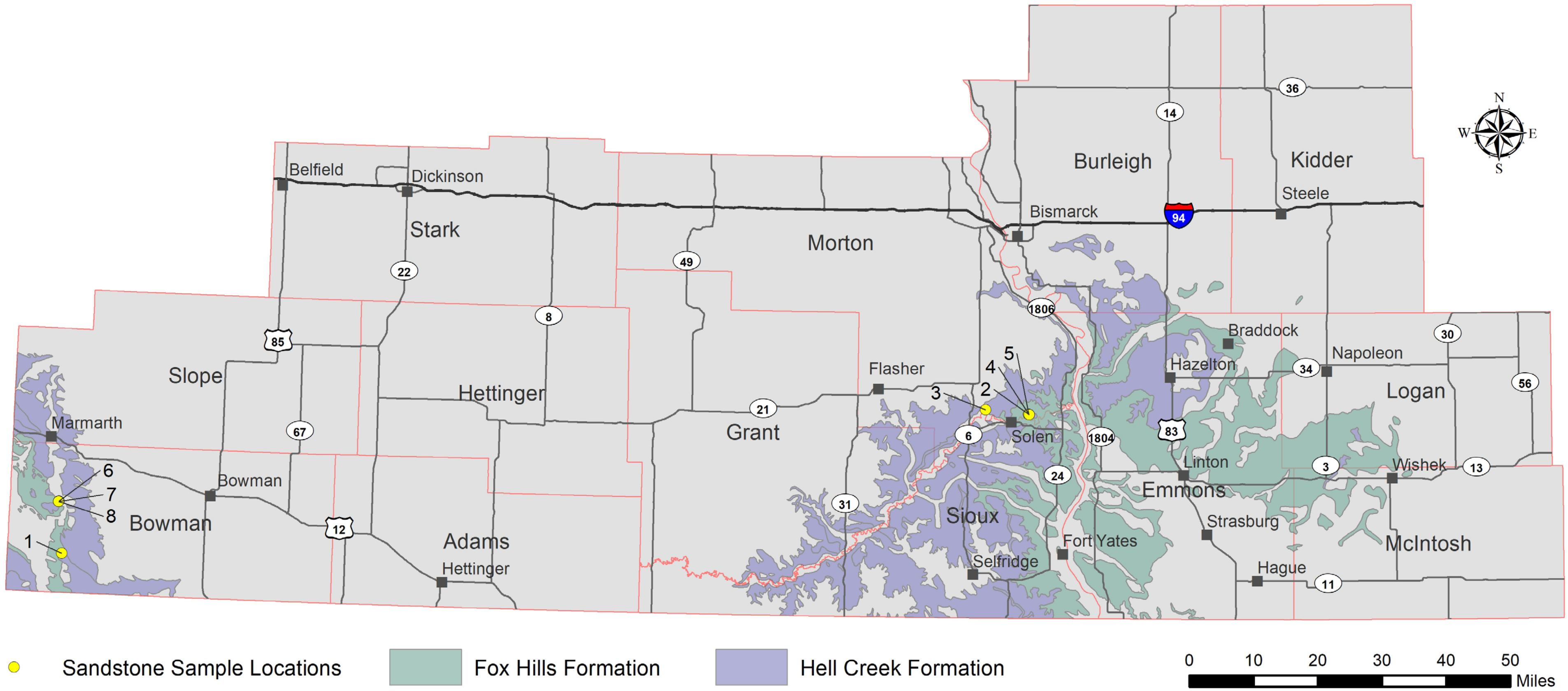


FIGURE 1.

Location and extent of the Fox Hills and Hell Creek Formations in southwestern and south-central North Dakota. Sandstone samples Nos. 1, and 6 - 8 were collected from outcrops in western Bowman County. Sandstone samples Nos. 2 - 5 were collected in southeastern Morton County. County scale mapping work was preformed in Adams and Bowman, Grant and Sioux, and Morton counties by Carlson (1979, 1982, and 1983), Burleigh County by Kume and Hansen (1965), Emmons County by Bluemle (1984), and Logan and McIntosh Counties by Clayton (1962).

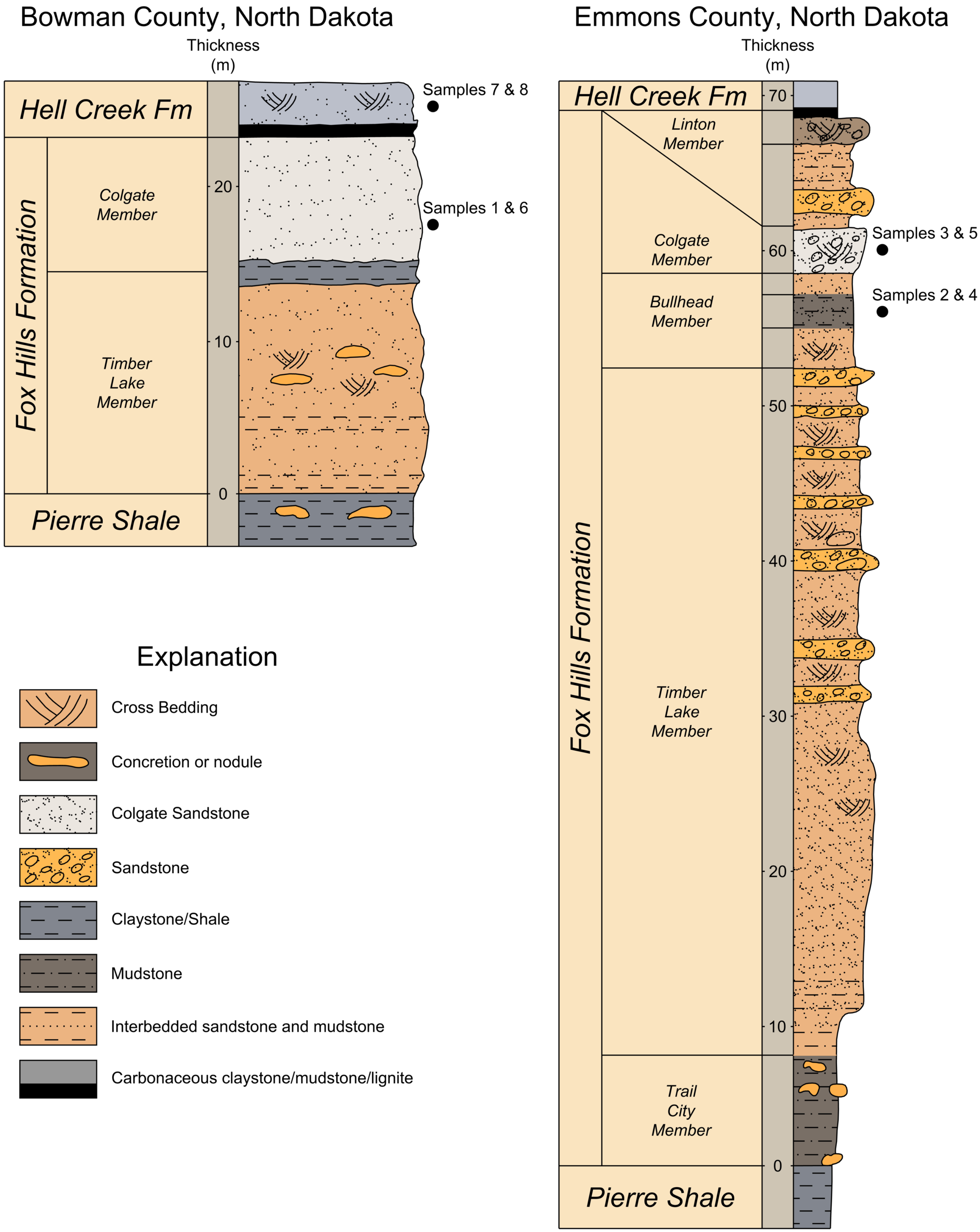


FIGURE 2.
Stratigraphic columns and relative positions of samples collected from the Fox Hills and Hell Creek Formations in southwestern and south-central North Dakota. The Colgate and Timber Lake members are the only two members of the Fox Hills Formation present in Bowman County. The Trail City (lower Fox Hills Formation), Iron Lightning (including the Colgate Sandstone), and Linton members are present in south-central North Dakota (Hoganson and Erickson, 2005).

SANDSTONE SAMPLING LOCATIONS

Bulk samples of sandstone were collected in five-gallon buckets from prominent outcrops in southwestern and south-central North Dakota during the 2022 field season. Each of the eight samples collected were submitted to Lonquist & Co. LLC for proppant (frac sand) testing (Figure 1). Sandstone sampling requires the use of a battery-powered bucket auger drill to collect samples from behind the most weathered zone approximately two to three feet into the outcrop. One of the sandstone samples collected from the Fox Hills Formation, Sample No. 2 reacted vigorously to dilute (10%) HCL in the field suggesting sandstone with an abundance of carbonate matrix or other potentially deleterious acid-reactive minerals.

Samples No. 1 – Fox Hills Formation, Colgate Member

Sample No. 1 was collected from a prominent area of northeast-facing outcrops in southwestern Bowman County (Figure 3) located 18 miles (29.4 km) south of Marmarth (Table 1).

Sample No. 2 – Fox Hills Formation, Bullhead Member

Sample No. 2 was collected along a large southwest-facing outcrop section located on the Strommen Ranch in southeastern Morton County (Table 1). This sample location is located approximately 3 miles (4.8 km) northeast of Solen on the northern side of the Cannonball River (Figure 4).

Sample No. 3 – Fox Hills Formation, Colgate Member

Sample No. 3 was collected along a westward-facing outcrop of sandstone on the Tschider Ranch in southern Morton County (Table 1). This sample location is located approximately 3.5 miles (5.6 km) northeast of Breien just north of the Cannonball River. This area of outcrop displays the characteristic castellate weathering patterns described for the Colgate Member by previous workers (Figure 5).

Sample No. 4 – Fox Hills Formation, Bullhead Member

Sample No. 4 was collected along the large southwest-facing outcrop section located on the Strommen Ranch in southeastern Morton County (Table 1). The sample was collected approximately midway (~15 feet) up-section on the outcrop which is approximately the mid-portion of the Bullhead Member at this location. This expansive outcrop area is located approximately 3 miles (4.8 km) northeast of Solen on the northern side of the Cannonball River (Figure 6).

Sample No. 5 – Fox Hills Formation, Colgate Member

Sample No. 5 was collected further up-section into the sandstone of the Colgate Member, which directly underlines the carbonaceous layer of the Hell Creek Formation around 40 feet above the base of the outcrop (Table 1). Again, this sample location is within the expansive outcrop area located approximately 3 miles (4.8 km) northeast of Solen on the northern side of the Cannonball River (Figure 6).

Sample No. 6 – Fox Hills Formation, Colgate Member

Sample No. 6 was collected from a western-facing outcrop of Fox Hills and Hell Creek Formation Sandstone along the Little Missouri River in western Bowman County (Table 1). This sample location is located ten miles south of Marmarth and 12.5 miles southwest of Rhame (Figure 7).

Sample No. 7 – Hell Creek Formation, Lower Section

Sample No. 7 was collected from the same outcrop as Sample No. 6, but on the backside (Figure 8) higher up in the section into the Hell Creek portion of the outcrop (Table 1).

Sample No. 8 – Hell Creek Formation, Lower Section

Sample No. 8 was collected from an outcrop that is further west (Figure9 9) than outcrops shown in Figures 7 and 8 (Table 1).

TABLE 1.
Sample Location Stratigraphic Summary

Sample No.	Stratigraphic Unit	County	Location (PLSS)	Description	Analytical Testing Completed		Field Acid Reactivity (10% HCl)
					Bulk Sand XRD Mineralogy	API 19C Proppant (wXRD)	
1*	Fox Hills Formation, Colgate Member	Bowman	130-106-36-SE	Sandstone; Fine-Grained	NT	X	NR
2	Fox Hills Formation, Bullhead Member	Morton	134-80-21-SE	Siltstone; Sandy, with Concretions	X	X	Yes
3	Fox Hills Formation, Colgate Member	Morton	134-80-21-SE	Sandstone; Fine-Grained	X	X	NR
4	Fox Hills Formation, Bullhead Member	Morton	134-80-21-SE	Siltstone; Sandy, Very-Fine Grained	X	X	NR
5	Fox Hills Formation, Colgate Member	Morton	134-80-21-SE	Siltstone; Sandy, Very-Fine Grained	X	X	NR
6	Fox Hills Formation, Colgate Member	Slope	131-106-24-SW	Sandstone; Fine-Grained	X	X	NR
7	Hell Creek Formation, Lower	Slope	131-106-24-SW	Sandstone; Fine-Grained	X	X	NR
8	Hell Creek Formation, Lower	Slope	131-106-24-SW	Sandstone; Fine-Grained	X	X	NR

** = Sample locations on ND State Trust Lands.*

NR = Non-Reactive.

NT = Not Tested.

FIGURE 3.
Light gray to buff sandstones of the Colgate Member (Fox Hills Formation) exhibiting a distinct castellate weathering pattern in southwestern Bowman County, North Dakota. Sample No. 1 was collected nearby from the relatively well-sorted, weakly indurated interval of fine-grained sandstone along the base of this exposure. This sandstone, making up roughly the lower half of the outcrop pictured, is at least 30 feet (9 meters) thick. The darker brown mudstone of the lower Hell Creek Formation is visible across the top of the outcrops. The view is to the southwest.

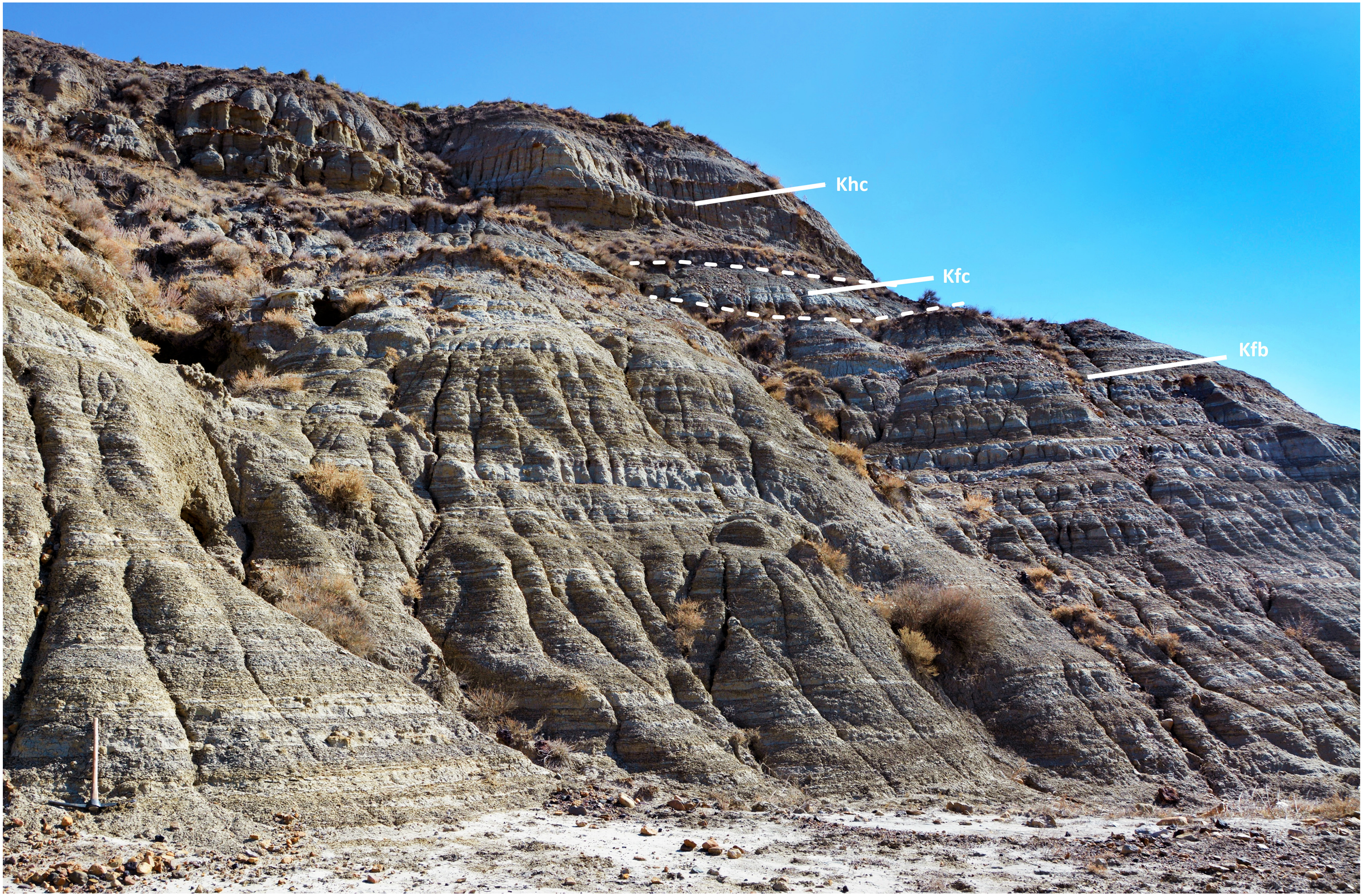


FIGURE 4.
View to the east of outcrops of the Fox Hills Formation and Hell Creek Formations on the Strommen Ranch in southeastern Morton County, North Dakota. This outcrop is about 220 feet high from base to top. Stratigraphy at this location (Clausen and Kihm, 1986) consists of 50 feet of the Hell Creek Formation capping the ridge, underlain by 20 feet of the Colgate Sandstone, and 75 feet of the Bullhead Member. Sample No. 2 was collected from the sandy siltstones of the Bullhead member of the Fox Hills Formation along the outcrop at the level of the pick in the lower left foreground.

FIGURE 5.
View to the east of an outcrop of the Colgate Sandstone Member of the Fox Hills Formation on the Tschider Ranch in southeastern Morton County, North Dakota. Sample No. 3 was collected from this sandstone which exhibits the characteristic castellate weathering pattern at the outcrop. Survey geologist Benjamin York is operating a battery-operated drill with a sand auger bucket attachment in order to collect sandstone deep from behind the outcrop weathering zone (~2-ft).

FIGURE 6.
View to the southwest across the expansive outcrop of the Fox Hills and Hell Creek Formations on the Strommen Ranch in southeastern Morton County, North Dakota. Samples No. 4 and 5 were collected from the Bullhead and Colgate silty sandstone members, respectively on this outcrop (black arrows).

FIGURE 7.
Interbedded light gray sandstone and gray-brown sandy mudstone of the Colgate Member (Fox Hills Formation) underlies yellow-brown sandstone and sandy mudstone of the Hell Creek Formation along the Little Missouri River in western Bowman County, North Dakota. Sample No. 6 (black arrow) was collected from a relatively well-sorted interval of sandstone near the base of the outcrop. Ophiomorpha burrows were abundant within the lower half of this section, suggesting nearshore marine deposition. The outcrop pictured is 170 feet (52 meters) tall. The view is to the southwest.

FIGURE 8.

Yellow-brown sandstone grades upwards into sandy mudstone of the lowermost Hell Creek Formation in western Bowman County, North Dakota. The outcrop pictured (view is to the northeast) is roughly the upper half of the taller outcrop pictured in Figure 7 from the opposite side. The sample was collected near the base of this photo (black arrow) from a weakly indurated, cross-bedded yellow-brown sandstone with minor organic laminae. The outcrop pictured is 65 feet (20 meters) tall.

FIGURE 9.
Light brown silty sandstone of the Hell Creek Formation underlying dark brown mudstone in western Bowman County, North Dakota. Sample No. 8 (black arrow) was collected near the base of this outcrop in a cross-bedded, very fine-grained silty sandstone, which was weakly indurated between stringers of more moderately indurated, lighter-colored sandstone. The outcrop pictured is 30 feet (9 meters) tall. The view is to the southeast.

DESCRIPTION OF TESTING RESULTS

Sandstone samples from the Fox Hills and Hell Creek Formation were tested in accordance with recommendations and specifications for proppant sand published by the American Petroleum Institute (API) and the International Organization for Standards (ISO). Testing included: particle size distribution (sieve analysis), 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 testing. As in past investigations, long-term conductivity testing was not performed during this investigation due to budgetary considerations. Testing and analyses were completed by Lonquist & Co., LLC, located in College Station, Texas in August of 2022.

SAMPLE PREPARATION

All samples submitted for testing were prepared for analysis by washing on the #200 sieve, drying, and disassociation (Table 2). An initial gradational analysis was first performed on prepared samples to determine the dominant sand size fraction. The remaining tests were then performed on the dominant sand size fraction, in this case, sands falling within either the 40/140 or 70/140 size classification (Figure 10). Bulk samples were tested on samples No. 4 & 5 since the wash loss on these samples was >80% (Table 2).

PARTICLE SIZE DISTRIBUTION – TEXTURAL (SIEVE) ANALYSIS

Sieve analyses were conducted to quantify the different-sized sand grains within an individual sample. A series of stacked wire-mesh sieves of standard sizes was 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) was recorded and reported as tabular data (Table 4) or in graphical form on a grain-size distribution diagram (Figure 10).

The resulting graph and grain-size curves depict the volume of 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 4), resulting in a very steep curve on the grain size distribution diagram (Figure 8). All samples selected for testing were well-sorted (poorly graded) sands falling into the medium to very-fine-grained size classes (Appendix I).

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 different types of sediment (e.g. sand and gravel). The Modified Wentworth system was used in this study.

All samples fall into the grain size ranges for classification as “sand” according to the Modified Wentworth classification scheme (Figure 10) and can be further characterized as well-sorted (poorly graded) to very well-sorted, medium to very-fine-grained sands. All the samples had most grains fall in the 40/140 and 70/140 or “100 mesh” sand size ranges (Table 4).

Statistical analyses can be performed 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). The average mean and median particle sizes were 0.15 and 0.14 respectively, in sandstones with similar gradations. Sample no. 7 from the lower Hell Creek Formation returned values of 0.3 both for mean and median particle size, which was somewhat larger than the other values.

TABLE 2.
Wash Loss on Bulk Sample Summary

Sample No.	Stratigraphic Name	Wash Loss (%)	Tested Size Class	(%) In-Size
1	Fox Hills Formation - Colgate Member	37	70/140	64
2	Fox Hills Formation - Bullhead Member	50	40/140	52
3	Fox Hills Formation - Colgate Member	23	40/140	94
4	Fox Hills Formation - Bullhead Member	90	Bulk	All
5	Fox Hills Formation - Colgate Member	81	Bulk	All
6	Fox Hills Formation - Colgate Member	41	70/140	76
7	Hell Creek Formation - Lower	23	40/140	90
8	Hell Creek Formation - Lower	39	70/140	55

[1] Wash Loss includes % material passing the #200 sieve (effectively the sand/silt cutoff). *Bulk: Bulk sample XRD Mineralogy only.*

All: Remaining gradations after wash loss tested from XRD Mineralogy only.

TABLE 3.

Proppant Testing Analytical Summary of Fox Hills-Hell Creek Formation Sandstones

Sample No.	Stratigraphic Unit	Lithology	Wash Loss (%)	Tested Size Class[1]	Quartz Content (%)	Crush Resistance (K-Value)	Acid Solubility (%)	Shape Factors		ISO Mean Particle Dia. (mm)	Median Particle Dia. (mm)	Turbidity (FTU)	Loss on Ignition (%)	Bulk Density (pcf)	Apparent Density (g/cm^3)
								Roundness	Sphericity						
1	Fox Hills Formation, Colgate Member	Sandstone	36.6	70/140	64.0	3K	9.8	0.5	0.8	0.196	0.185	147	NT	79	NT
2	Fox Hills Formation, Bullhead Member	Silty Sandstone (c)	49.5	40/140	13.5	NT	NT	0.5	0.8	0.153	0.114	95.8	NT	69.0	NT
3	Fox Hills Formation, Colgate Member	Sandstone	23.1	40/140	49.8	4K	10.3	0.5	0.8	0.168	0.164	78.6	1.10	77.9	2.67
4	Fox Hills Formation, Bullhead Member	Sandy Siltstone	89.8	Bulk	59.9	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
5	Fox Hills Formation, Colgate Member	Sandy Siltstone	80.8	Bulk	55.5	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
6	Fox Hills Formation, Colgate Member	Silty Sandstone	40.6	70/140	75.0	3K	9.8	0.5	0.8	0.134	0.129	86.2	0.90	75.2	2.62
7	Hell Creek Formation, Lower	Sandstone	22.9	40/140	83.0	5K	9.9	0.5	0.8	0.302	0.301	105.0	1.20	80.1	2.61
8	Hell Creek Formation, Lower	Sandstone	38.5	70/140	79.0	1K	5.8	0.5	0.8	0.110	0.110	121.0	0.60	73.8	2.62

NT = Not Tested.

[1] *Washed tested gradation.*

(c) concretionary

TABLE 4.

Sample Sieve Analysis Results Summary (Weight % Retained)

Sample No.		1	2	3	4	5	6	7	8
US Sieve No.	6	0.0	0.0	0.0	No Gradation Performed - Full Sample Tested	No Gradation Performed - Full Sample Tested	0.0	0.0	0.0
	8	0.0	0.0	0.0			0.0	0.0	0.0
	16	0.0	0.0	0.0			0.0	0.0	0.0
	30	0.0	0.9	0.1			0.0	0.3	0.0
	40	0.3	1.7	0.1			0.0	7.3	0.0
	45	1.7	2.1	0.1			0.0	16.0	0.0
	50	5.6	4.3	0.3			0.2	27.1	0.0
	60	9.8	5.4	3.0			1.1	22.4	0.1
	70	13.8	5.2	8.2			2.8	9.8	0.4
	80	23.0	6.6	22.6			5.6	5.6	2.0
	100	21.7	8.9	31.2			16.4	4.0	7.0
	120	13.1	8.8	19.3			29.2	2.8	17.8
	140	5.9	11.1	9.2			24.6	2.2	28.5
	170	2.9	12.1	3.1			11.2	1.4	19.2
	200	1.9	21.4	2.1			6.8	1.0	16.5
	Pan	0.3	11.4	0.6			2.1	0.1	8.4
Total		100.0	100.0	100.0	NA	NA	100.0	100.0	100.0

TABLE 5.
Crush Resistance Testing Data Summary

Sample No.	Size Class	K-Value	Test Stress (psi)						
			1000	2000	3000	4000	5000	6000	7000
			Fines Generated (%)						
1	70/140	3K	--	--	6.65	11.95	--	--	--
2	40/140	NT	--	--	--	--	--	--	--
3	40/140	4K	--	--	--	9.51	13.29	--	--
4	NT		--	--	--	--	--	--	--
5	NT		--	--	--	--	--	--	--
6	70/140	3K	--	--	9.23	16.70	--	--	--
7	40/140	5K	--	--	--	--	8.58	10.60	--
8	70/140	1K	7.14	11.45	--	--	--	--	--

-- Stress point not tested.

NT: Not Tested

TABLE 6.
X-ray diffraction (XRD) Mineralogy Analytical Summary

Sample ID	Tested Size Class	Quartz (%)	Feldspars (%)			Carbonates (%)			Clays (%)				Other Minerals (%)	
			Albite	Microcline	Feldspars*	Calcite	Dolomite	Carbonates*	Illite	Montmorillonite	Kaolinite	Clays*	Biotite	Chlorite
1 - Kfc	70/140	63.5	--	--	30.1	--	--	--	4.1	--	--	4.1	--	1.6
2 - Kfb	BULK	25.8	26.4	--	26.4	36.6	0.0	36.6	7.2	3.6	0.4	11.2	--	--
	40/140	13.5	9.8	--	9.8	75.9	0.9	76.8	0.0	0.0	0.0	0.0	--	--
3 - Kfc	BULK	60.6	39.4	--	39.4	--	--	--	--	--	--	--	--	--
	40/140	49.8	19.9	--	19.9	10.9	19.4	--	--	--	--	--	--	--
4 - Kfb	BULK	73.2	19.2	--	19.2	--	--	--	7.6	--	--	7.6	--	--
	Washed	59.9	29.1	--	29.1	--	--	--	11.0	--	--	11.0	--	--
5 - Kfc	BULK	45.2	35.0	--	35.0	--	--	--	19.8	--	--	19.8	--	--
	Washed	55.5	44.5	--	44.5	--	--	--	--	--	--	--	--	--
6 - Kfc	BULK	66.8	8.4	--	8.4	--	--	--	13.8	--	5.2	18.0	5.7	--
	70/140	75.0	12.6	--	12.6	--	--	--	7.4	--	2.5	9.9	2.5	--
7 - Khl	BULK	82.0	8.9	--	8.9	--	--	--	--	--	--	--	4.1	--
	40/140	83.0	5.7	--	5.7	--	--	--	--	--	3.9	3.9	2.0	--
8 - Khl	BULK	80.8	5.3	--	5.3	--	--	--	6.8	--	--	6.8	7.0	--
	70/140	79.3	6.6	--	6.6	--	--	--	14.0	--	--	14.0	--	--

** Undifferentiated*

-- Mineral not detected

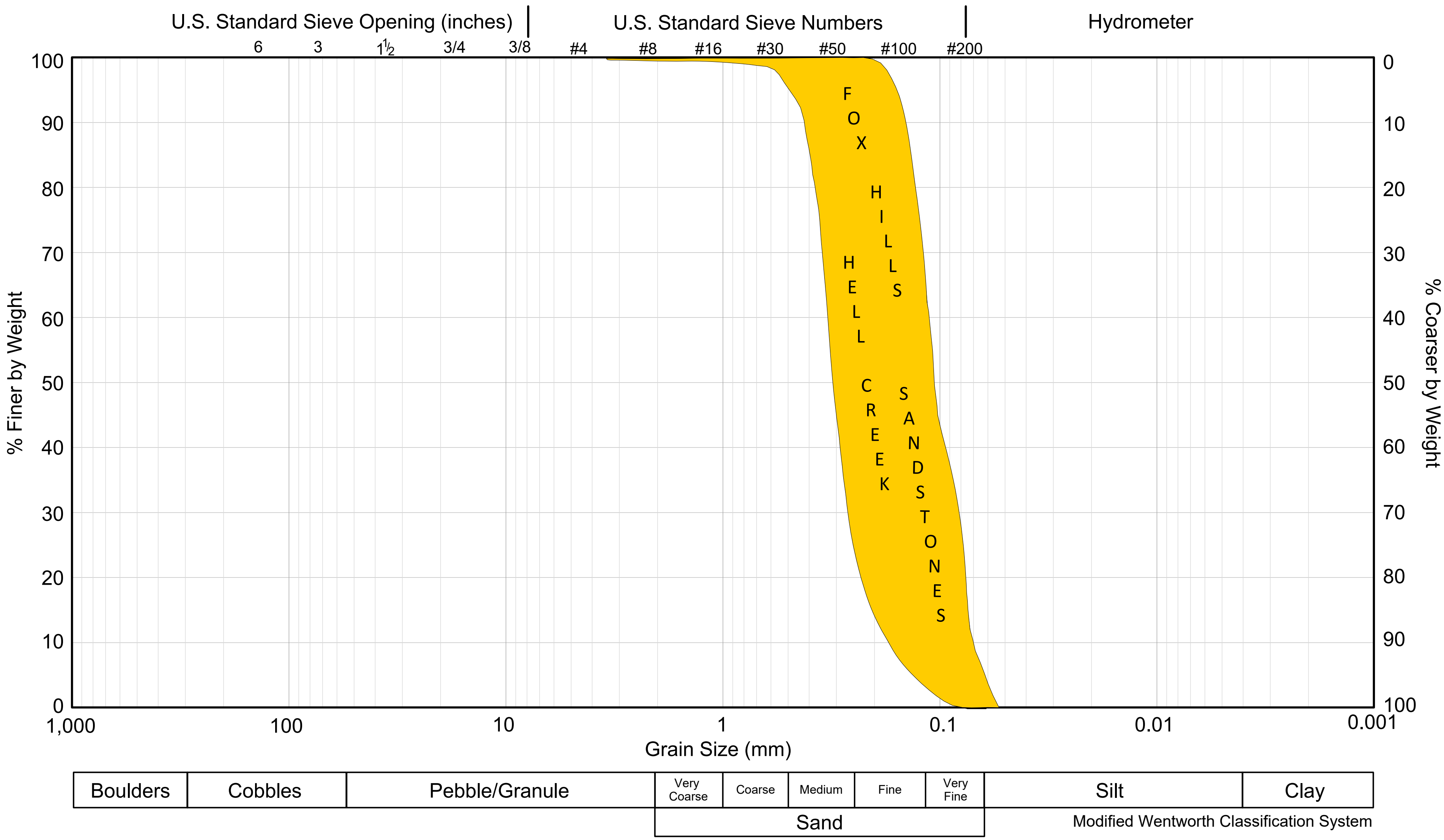


FIGURE 10.
Range of 8 grain-size distribution curves (shaded-yellow) from the washed gradations of sandstone of the Fox Hills and Hell Creek Formations in south-central and southwestern North Dakota. The sand fractions of these sandy siltstones are well sorted and mostly fine to very-fine grained according to the Modified Wentworth Classification of grain sizes.

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 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 shape of the corners of an individual sand grain. Sand with high sphericity and roundness is desirable for proppant use. The recommended sphericity and roundness values are 0.6 or greater, with values of 0.7 or greater characteristic of high-strength proppants (API, 2018). Samples tested in this group had consistent sphericity values of 0.8 and roundness values of 0.5 (Figure 12). These values approach desired specifications, particularly for sphericity factors, but are much more angular in overall grain shape (Plate I).

ACID SOLUBILITY

The volume of 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 at or below the recommended acid solubility threshold (Figure 13) but are within the lower range of other sandstones tested from western North Dakota (Anderson et al., 2019). Acid solubility on these sandstones was generally near 10% (Table 3).

SILT AND CLAY FINES TESTING (TURBIDITY)

Turbidity tests measure the optical properties of water samples containing suspended sediment and are commonly used to determine the percentage of fine materials (e.g. silts and clays) present. 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 the initial processing of raw product into proppant sand. Turbidity is measured and commonly reported in Formazin Turbidity Units (FTU). The recommended turbidity limit for proppant sand is less than 250 FTU. All eight samples were generally 100 FTU below the recommended limit of 250 FTU (Figure 14). The samples ranged from 78.6 to 147 FTU (Table 3), like other tested sandstones in western North Dakota (Anderson et al., 2019), and are considerably higher than windblown sand tested from north-central North Dakota (Anderson, 2020).

CRUSH RESISTANCE

Sand compositions can be quite variable as can the resultant strength. 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 the material from the largest size class on four of the samples that contained sufficient volumes after sample preparation. K-Values were determined by subjecting the samples to one set of two pre-determined stresses, that were determined by the size range of the samples. A K-value is defined as the highest stress level that will generate no more than 10% crushed material (rounded down to the nearest 1,000 psi). Crush resistance in these samples was low and ranged from 1,000 psi (1K) to 5,000 psi (5K) within the tested size classes (Table 6). For comparison, Ottawa White (preferred) sands typically generate K-values between 7 and 15K, and the windblown sands tested in North Dakota range from 4 to 8K (Anderson, 2019). Bakken oil and gas wells have fracture closure stresses in the reservoir that range typically around 9,500 psi.

LOSS ON IGNITION TESTING

Loss on ignition (LOI) testing was performed on four of the sized samples (sample nos. 3, and 6 – 8) to serve as a proxy for the determination of the potential detrital lignite content. The LOI values were all low, ranging from 0.6 to 1.2%, which is probably to be expected from a washed, sized sample cut (Table 3, Figure 12, Figure 14). This suggests a very low amount of potential organic-based deleterious constituents.

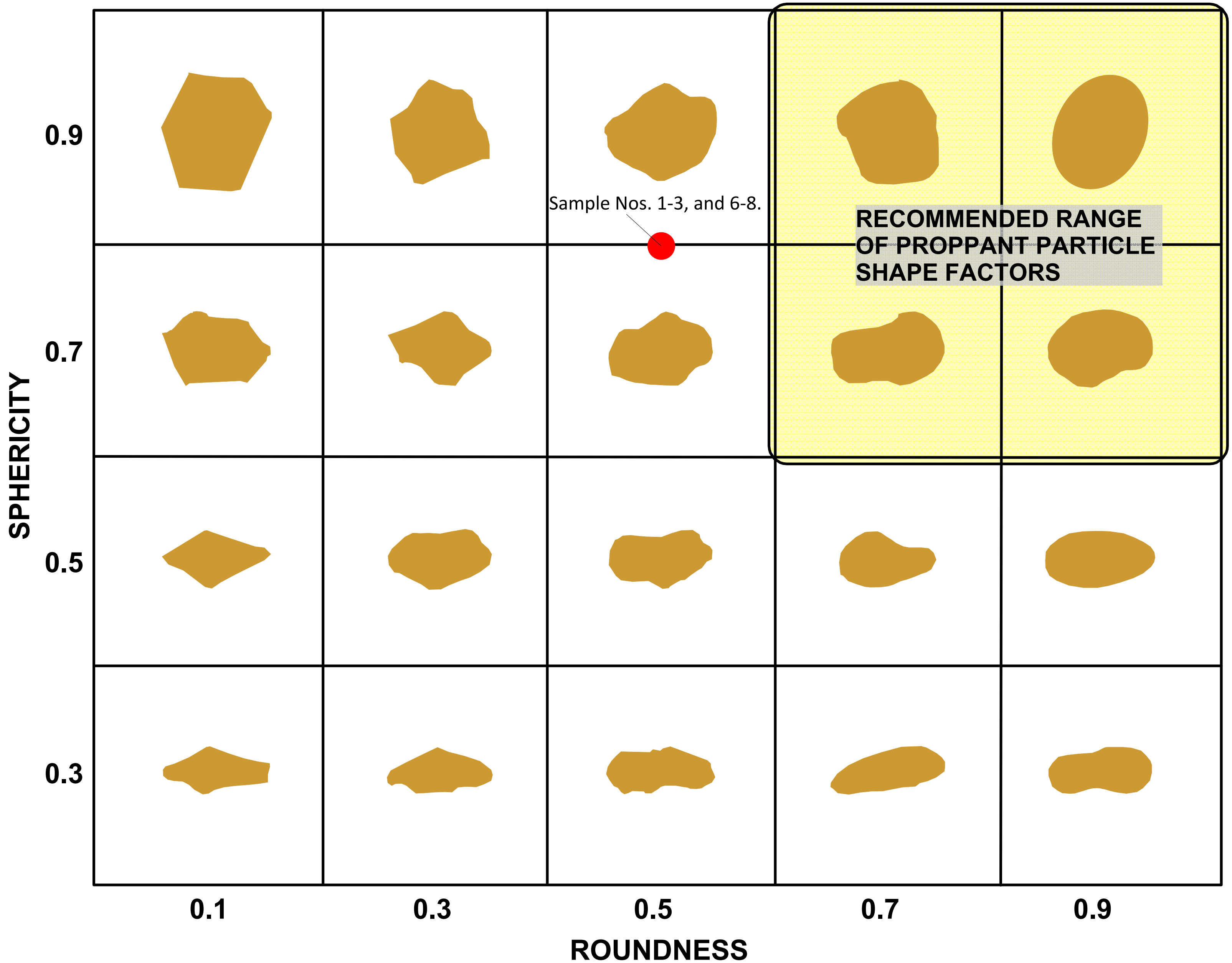


FIGURE 11.
Comparison of sphericity and roundness values for the sand fraction of silty sandstones from the Fox Hills and Hell Creek Formations in North Dakota. Samples from these deposits all have similar particle shape factor values (red circle) that approach but are below the range of desired specifications (shaded yellow) for frac sand as compared to Ottawa White silica sands.

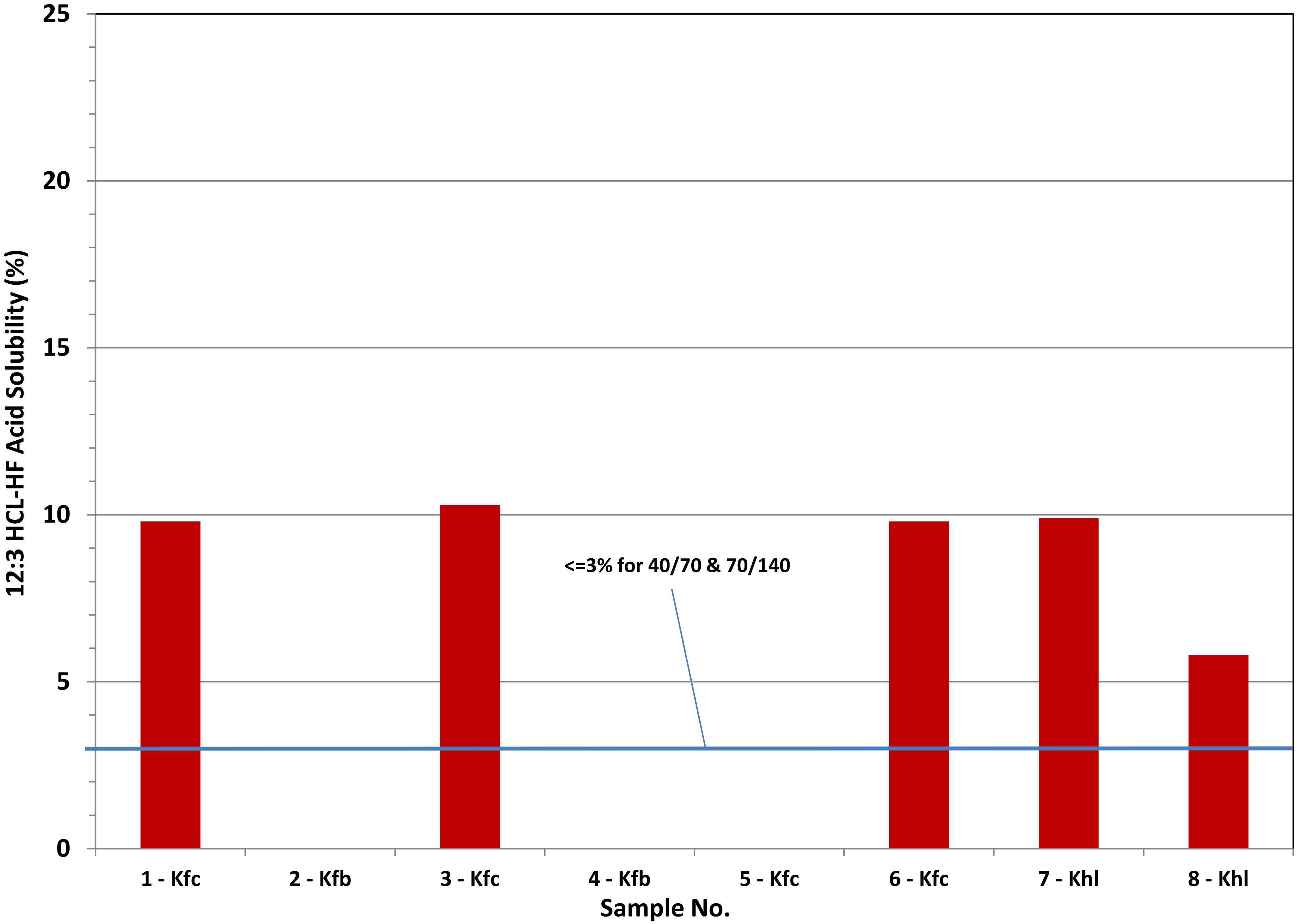


FIGURE 12.
Comparison of hydrochloric:hydrofluoric acid solubility results for sandstones of the Fox Hills and Hell Creek Formations in North Dakota. Recommended specifications for acid solubility on 40/70 & 70/140 sands are less than or equal to 3%.

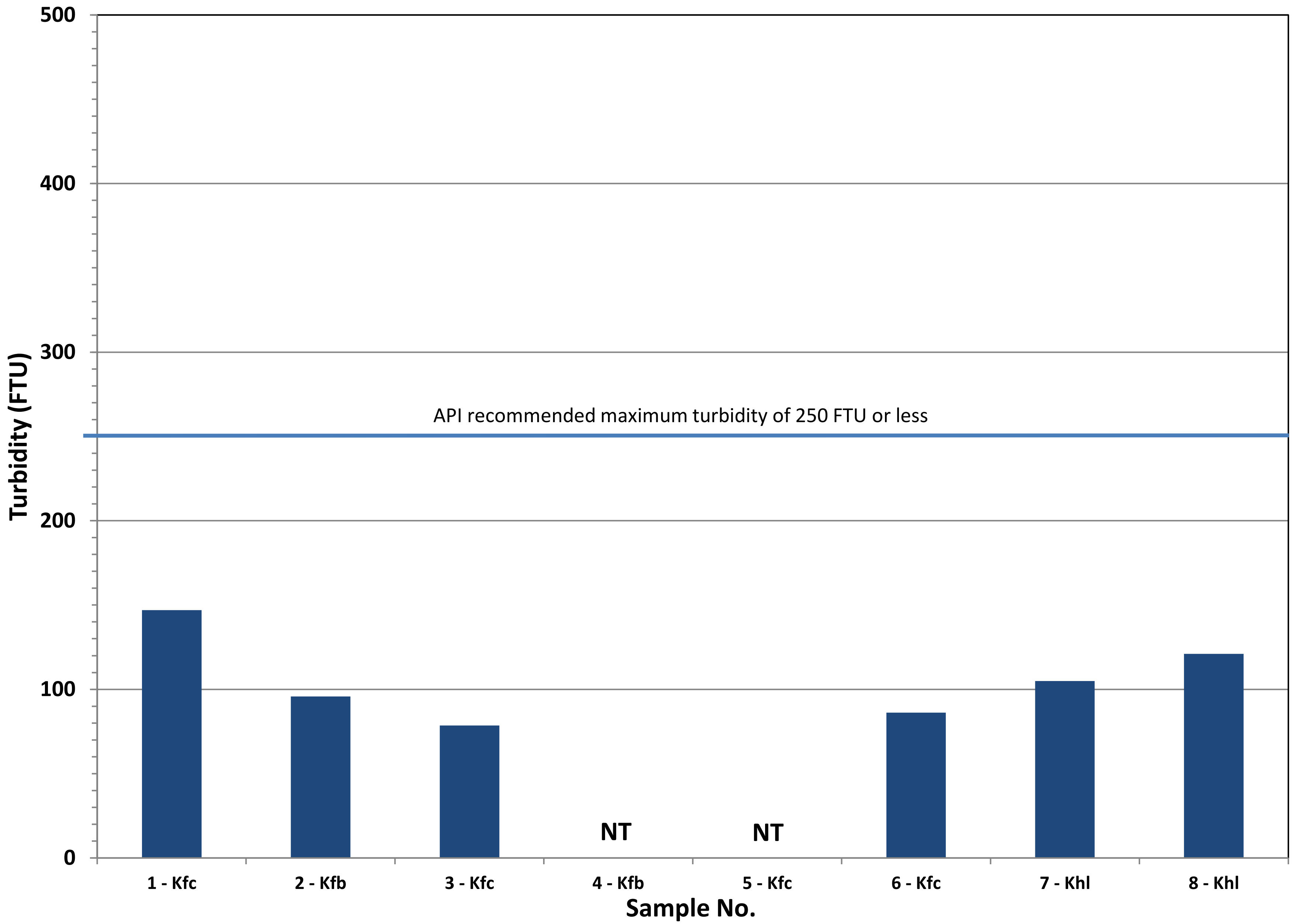


FIGURE 13.
Comparison of turbidity results for washed sandstone samples from the Fox Hills and Hell Creek Formations in North Dakota. Recommended API specifications for turbidity are 250 FTU or less (blue line). All samples tested fall well below this value. NT = Not tested due to low sample volume after initial wash loss during sample prep.

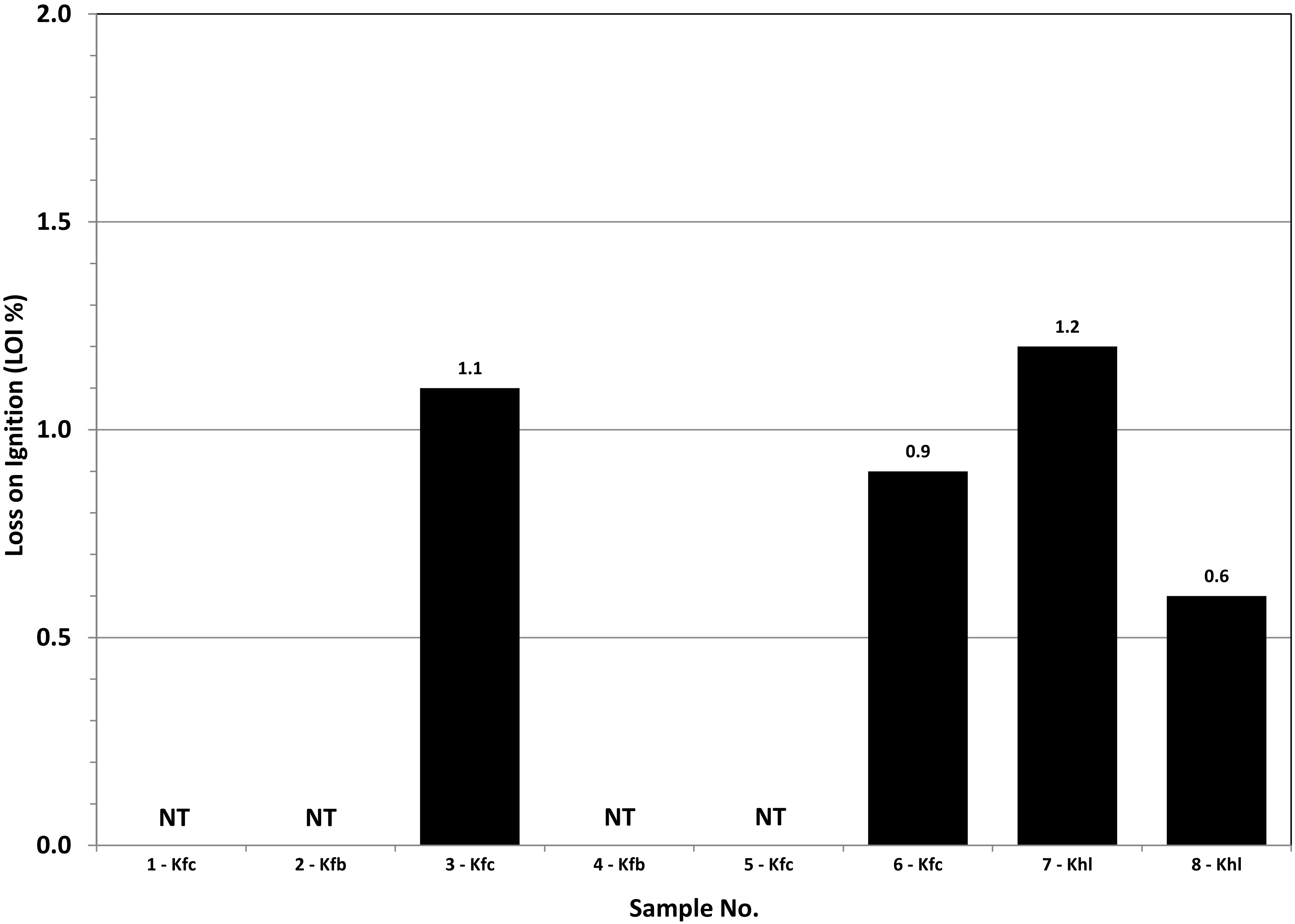


FIGURE 14.
Graph of loss on ignition (LOI) values of sandstones from the Fox Hills and Hell Creek Formations. These sands are from the 40/140 and 70/140 (washed) size classes. NT = Not tested (Small Sample Volume after Wash-Loss).

MINERALOGY (X-RAY DIFFRACTION)

Sample geochemistry was determined using qualitative and quantitative X-ray diffraction (XRD) on all washed and sized sand samples reported in weight percent (Table 6). XRD analysis is commonly used to determine the mineralogy of fine-grained lithologies, particularly clays. In general, the samples had similar overall mineralogical compositions (except for sample no. 2 which was a highly calcareous sample) with some variability in the lower percentages of feldspars and clays. Quartz percentages ranged from 50 to 83%, feldspar ranged from 6 to 45%, and clay contents ranged from 0 to 14% (Figure 15). In comparison, Ottawa White silica sands are commonly 99% quartz. XRD phase diagrams for all samples tested are included in Appendix II. These deposits are generally similar in mineralogical character (Figure A-II-15) but the sandstones tested from Bowman County had higher quartz contents that were closer to their windblown counterparts in central North Dakota.

Additional XRD work was also performed on bulk sandstone samples (i.e. samples that have not been washed and sized) from these units to provide additional information on the overall mineralogical character of deposits in situ (Appendix II). In these analyses, clay contents were low from 7 to 20%, and micas were reported at 6% from samples collected only in Bowman County (Figure 16). Quartz and feldspar contents were somewhat lower in the bulk samples, averaging 62% and 20% respectively. No iron-containing minerals were detected in the bulk samples, further suggesting a low potential for the existence of potentially deleterious mineralogy, other than clays and carbonates to be present in the sandstones. Clay content speciation was predominantly illite, a non-expansive clay mineral. Although all clays are considered deleterious components of a potential proppant source, the especially problematic expansive clay species (montmorillonites) were only identified in one sample from these sandstones.

BULK DENSITY

In regard to proppants, 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 sandstone deposits (Table 3) ranged from 75 to 80 pounds per cubic foot (PCF) with an average of 76 PCF.

APPARENT DENSITY

The apparent density measures sand density by using a low-viscosity fluid that wets particle surfaces and includes pore spaces inaccessible to the fluid. The apparent density of these sandstones ranged from 2.61 to 2.67 grams per cubic centimeter (g/cm^3) (Table 3). Apparent density values are used in the design of hydraulic fracturing applications. A summary of proppant testing specifications is provided in Appendix III.

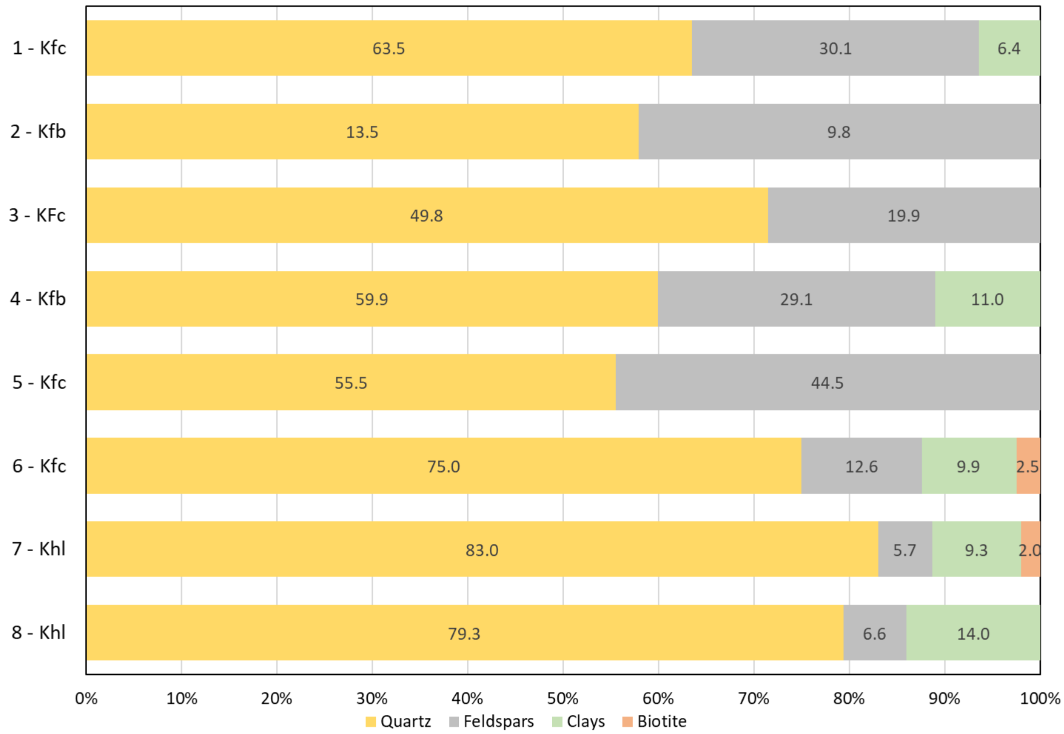


FIGURE 15.

X-Ray Diffraction (XRD) mineralogic analysis of washed sandstone from the Fox Hills and Hell Creek Formations. Sample Nos. 1 through 6 are from the Fox Hills Formation (Bullhead and Colgate Members) in southwestern and southcentral North Dakota. Sample Nos. 7 and 8 are from the Hell Creek Formation (lower) in southwestern North Dakota.

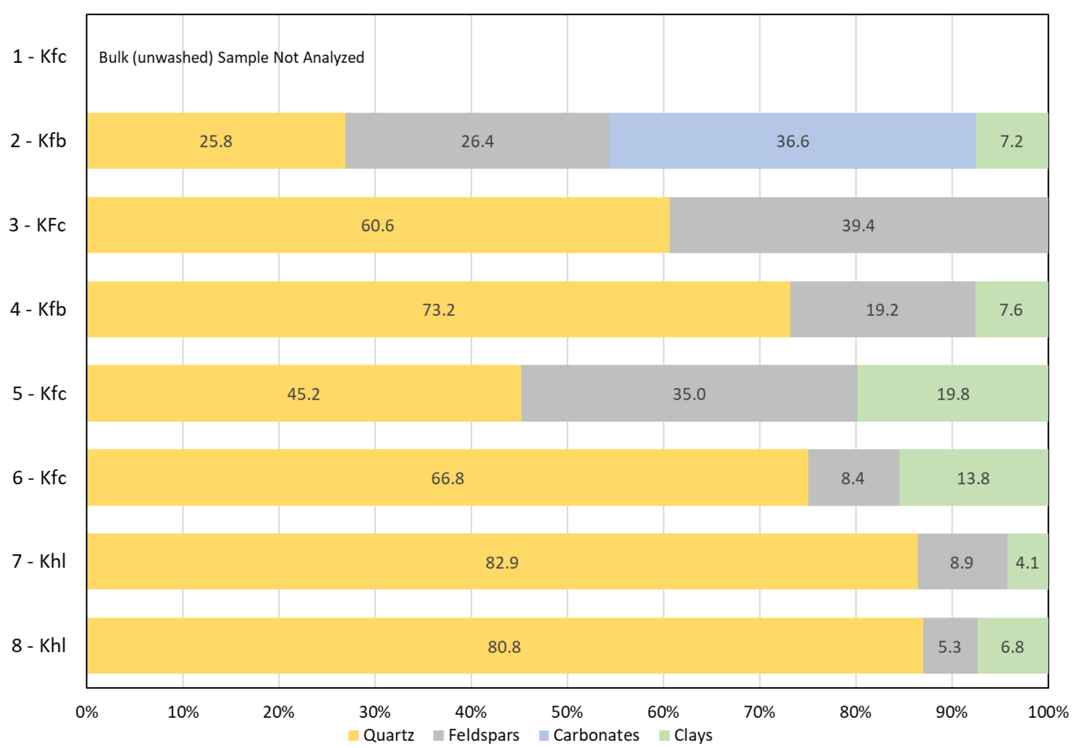
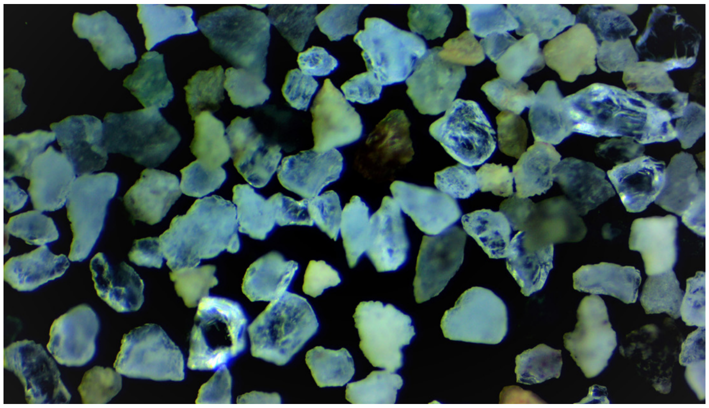
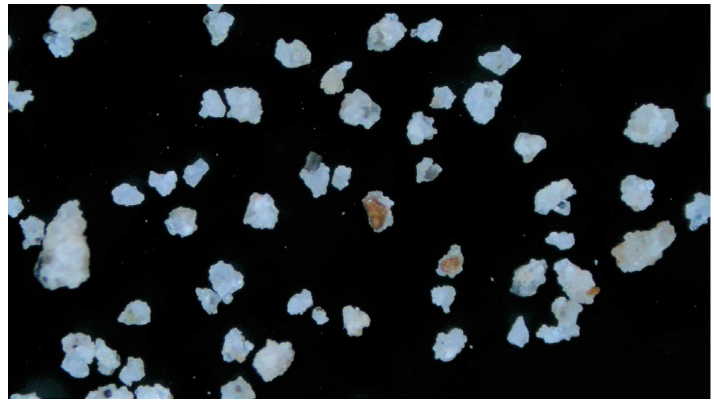


FIGURE 16.

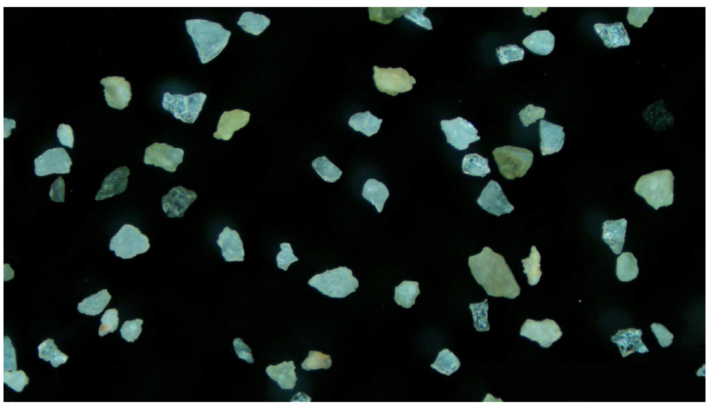
X-Ray Diffraction (XRD) mineralogic analysis of bulk (unwashed) sandstone from the Fox Hills and Hell Creek Formations. Sample Nos. 1 through 6 are from the Fox Hills Formation (Bullhead and Colgate Members) in southwestern and southcentral North Dakota. Sample Nos. 7 and 8 are from the Hell Creek Formation (lower) in southwestern North Dakota.



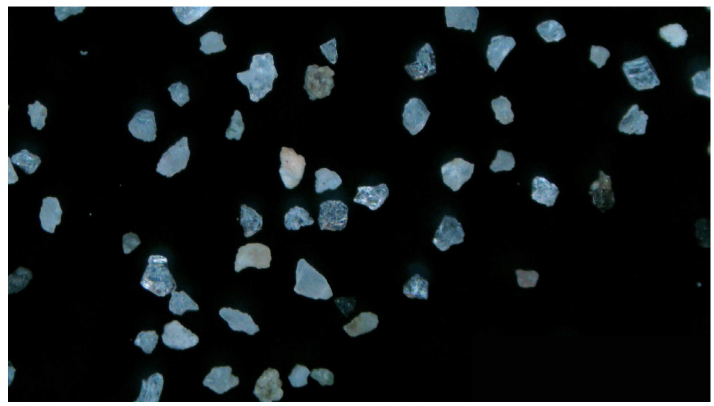
1. Fox Hills Formation, Colgate Member, Bowman County - 70/140.
Sample No. 1, this report.



2. Fox Hills Formation, Bullhead Member, Morton County - 40/140.
Sample No. 2, this report.

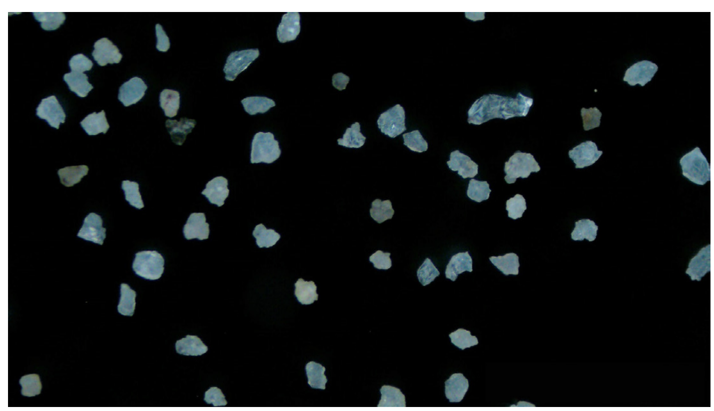


3. Fox Hills Formation, Colgate Member, Morton County - 40/140.
Sample No. 3, this report.



4. Fox Hills Formation, Colgate Member, Morton County - 40/140.
Sample No. 6, this report.

5. Hell Creek Formation, Lower, Bowman County - 40/140.
Sample No. 7, this report.



6. Hell Creek Formation, Lower, Bowman County - 70/140.
Sample No. 8, this report.

PLATE I.

Monolayer photomicrographs (40x) of sandstone from the Fox Hills and Hell Creek Formations in southwestern and southcentral North Dakota. No photomicrographs were collected for samples 4 & 5 in this report due to very low sample volume after wash-loss preparation.

CONCLUSIONS

Sandstone deposits within the Cretaceous Fox Hills and Hell Creek Formation in southwestern and south-central North Dakota have geologic characteristics observable in the field, such as uniformity of grain size and apparent consistent mineralogy which suggests these sandstones may have some potential for use as proppant sand. However, upon closer examination and laboratory testing, it is found that these sandstones do not possess the characteristics favored by the industry for use as proppant sand. Sandstone deposit quality is considerably variable across the deposits tested as no regionality was observed in the testing data. It is supposed that considerable additional material processing would likely be needed to increase favorable testing results for these sandstones which would likely be economically unfavorable.

Although these sandstones sampled and tested have visually appealing characteristics, such as uniformity of grain size as a fine grain sand (fig. 6) and a somewhat higher than average quartz content for most bedrock sandstones (Anderson et al., 2019), further testing has revealed that these sandstones are not likely to be viable candidates for use due to the high-volume loss on washing and resultant low resistance to crush stresses at around 3,000 psi (3K).

However, due to its somewhat distinct buff-to-white color and general uniformity of grain size as a fine-grained sandstone and relatively simple mineralogy, this material may be suitable for other applications such as landscaping or other decorative sand uses. The testing data included in this report should prove valuable for other potential industrial sand uses as well as future sedimentological research in clastic environments.

REFERENCES

Anderson, F.J., 2021, Evaluation of Windblown Sand Deposits in Eastern North Dakota for Potential Use as Proppant, North Dakota Geological Survey, Report of Investigation No. 127, 66 p.

Anderson, F.J., 2020a, Evaluation of Windblown Sand Deposits in South-Central North Dakota for Potential Use as Proppant, North Dakota Geological Survey, Report of Investigation No. 126, 76 p.

Anderson, F.J., 2020b, Evaluation of Windblown Sand Deposits in North-Central North Dakota for Potential Use as Proppant, North Dakota Geological Survey, Report of Investigation No. 124, 109 p.

Anderson, F.J., 2020c, Mineralogy of Windblown Sand Deposits in McHenry County, North Dakota Geological Survey, Geologic Investigation No. 243, 1:150,000.

Anderson, F.J., 2019a, The Potential of the Hazen-Stanton Dunes for use as Natural Sand Proppant, North Dakota Geological Survey, Geologic Investigation No. 216, 15 p.

Anderson, F.J., 2019b, Evaluation of Eolian Sand Deposits in North and South-Central North Dakota for Potential Use as Proppant, North Dakota Geological Survey, Report of Investigation No. 123, 32 p.

Anderson, F.J., 2018, Eolian Sands in North Dakota Evaluated for use as Natural Sand Proppant for Oil & Gas Wells, North Dakota Geological Survey, Geologic Investigation No. 207, 64 p.

Anderson, F.J., 2011, Investigation of Sand Resources in North Dakota: Sedimentological Characterization of Surficial Sand Deposits for Potential Use as Proppant, North Dakota Geological Survey, Report of Investigation No. 110, 67 p.

Anderson, F.J., Moxness, L.D., Kruger, N.W., Murphy, E.C., and Maike, C.A., 2019, Evaluation of Tertiary (Paleocene) Bedrock Sandstone of the Sentinel Butte and Bullion Creek Formations for Potential Use as Proppant, North Dakota Geological Survey, Report of Investigation No. 121., 43 p.

API, 2018, Measurement of and Specifications for Proppants Used in Hydraulic Fracturing and Gravel-packing Operations, American Petroleum Institute, API Standard 19C, 2nd edition, August 2018, 47 p.

Bluemle, J.P., 1984, Geology of Grant and Sioux Counties, North Dakota, North Dakota Geological Survey, Bulletin 66 - Part I, 69 p.

Boggs, S., Jr., 1995, Principles of Sedimentology and Stratigraphy (2nd ed): Prentice Hall, 774 p.

Carlson, C.G., 1983, Geology of Morton County, North Dakota, North Dakota Geological Survey, Bulletin 72 – Part I, 37 p.

Carlson, C.G., 1982, Geology of Grant and Sioux Counties, North Dakota, North Dakota Geological Survey, Bulletin 67 - Part I, 32 p.

Carlson, C.G., 1979, Geology of Adams and Bowman Counties, North Dakota, North Dakota Geological Survey, Bulletin 65 – Part I, 29 p.

Clausen, E. N., and Kihm, A. J., eds., 1986, Tertiary and Upper Cretaceous of south-central and western North Dakota Field Trip Guidebook, North Dakota Geological Society. 47 p.

Clayton, L., 1962, Geology of Logan and McIntosh Counties, North Dakota, North Dakota Geological Survey, Bulletin 37 - Part I, 84 p.

Clayton, L., Moran, S.R., Bluemle, J.P., and Carlson, C.G., 1980, Geologic Map of North Dakota, U.S. Geological Survey-N.D. Geological Survey, 1:500,000.

Cvancara, A. M., 1976, Geology of the Fox Hills Formation (Late Cretaceous) in the Williston Basin of North Dakota, with reference to uranium potential: North Dakota Geological Survey Report of Investigations no. 55, 16 p.

Feldmann, R. M., 1972, Stratigraphy and paleoecology of the Fox Hills Formation (Upper Cretaceous) of North Dakota: North Dakota Geological Survey Bulletin 61, 65 p.

Hoganson, J. W., and Erickson, J. M., 2005, A new species of Ischyodus (Chondrichthyes: Holocephali: Callorhynchidae) from Upper Maastrichtian Shallow marine facies of the Fox Hills and Hell Creek Formations, Williston basin, North Dakota, USA. Palaeontology, vol. 48, p. 709–721.

ISO, 2006, International Standard, Petroleum and Natural Gas Industries-Completion Fluids and Materials, Part 2: Measurement of properties of proppants used in hydraulic fracturing and gravel-packing operations, 28 p.

Klett, M. C., and Erickson, J. M., 1976, Type and reference sections for a new member of the Fox Hills Formation, Upper Cretaceous (Maestrichtian) in the Missouri Valley region, North and South Dakota: Proc. North Dakota Academy of Sciences v. 28, pt. II.

Kume J., and Hansen D.E., 1965, Geology of Burleigh County, North Dakota, North Dakota Geological Survey, Bulletin 42 - Part I, 111 p.

Murphy, E. C., Hoganson, J. W., Johnson, K. R., 2002, Lithostratigraphy of the Hell Creek Formation in North Dakota. Geological Society of America Special Paper 361, p. 9–34.

Murphy, E.C., Nordeng, S.H., Juenker, B.J., and Hoganson, J.W. (2009). North Dakota Stratigraphic Column. North Dakota Geological Survey, Miscellaneous Series 91.

APPENDIX I.

Individual Grain-Size Distribution (Sieve) Diagrams of Windblown Sand Samples from Dune and Sheet Sand Deposits in Eastern North Dakota.

The gradation diagrams included in this appendix are presented in a traditional Unified Soil Classification System (USCS) style diagram along with Wentworth particle size classifications.

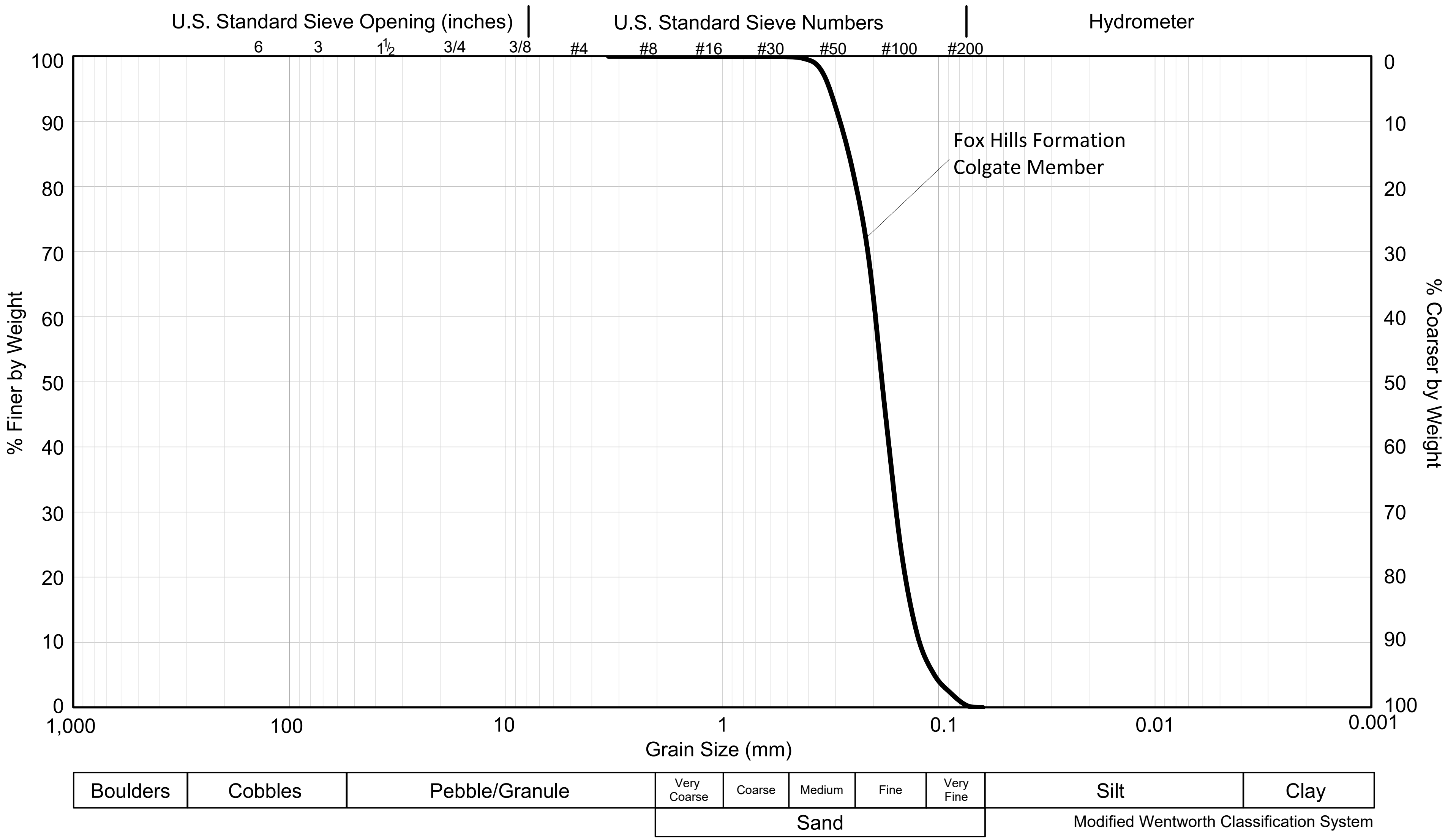


FIGURE A-I-1.
Grain-size distribution diagram (sieve analysis) for sandstone sample no. 1 from the Colgate Member of the Fox Hills Formation in southwestern Bowman County, North Dakota.

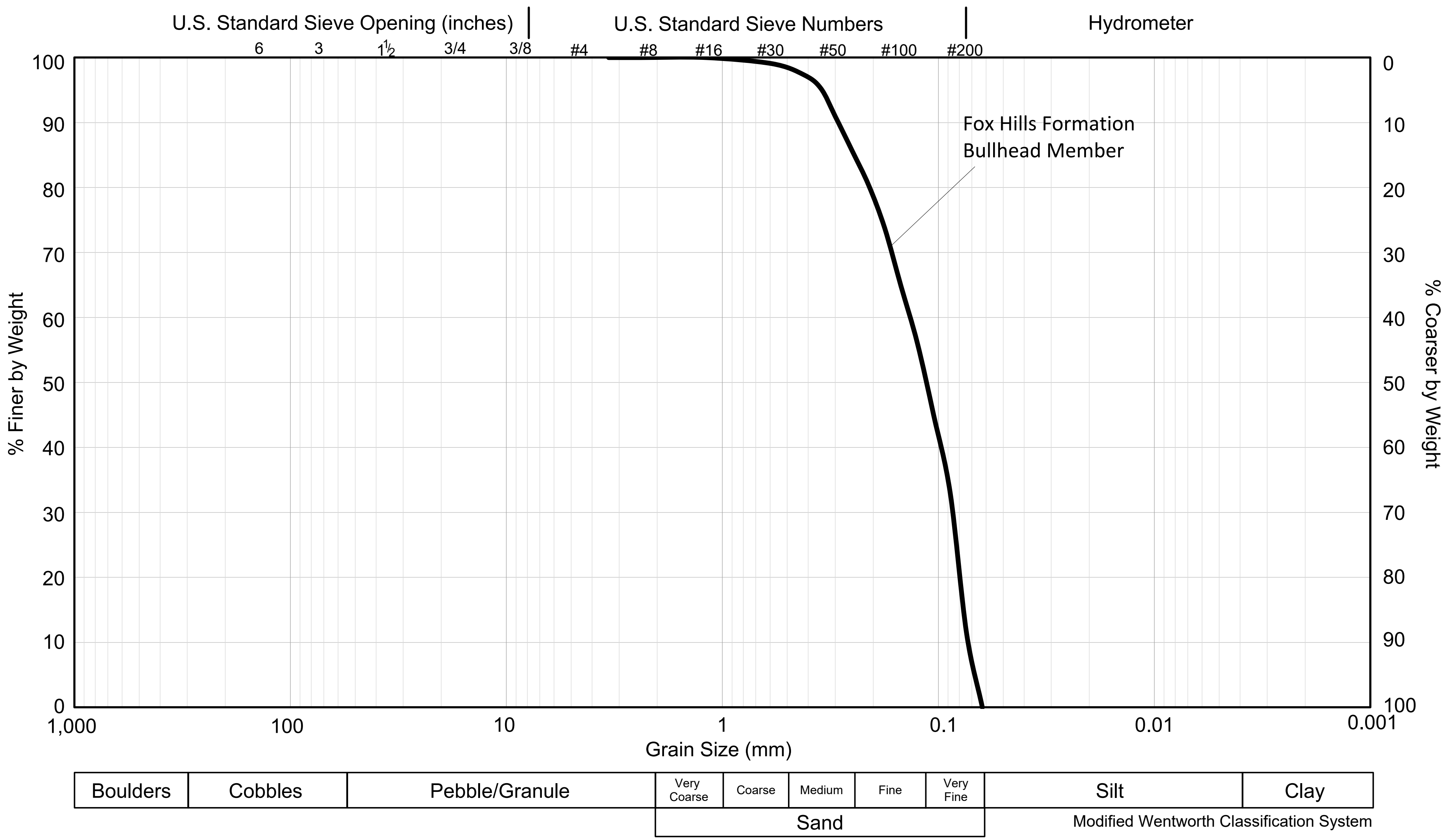


FIGURE A-I-2.
Grain-size distribution diagram (sieve analysis) for sandstone sample no. 2 from the Bullhead Member of the Fox Hills Formation in southeastern Morton County, North Dakota.

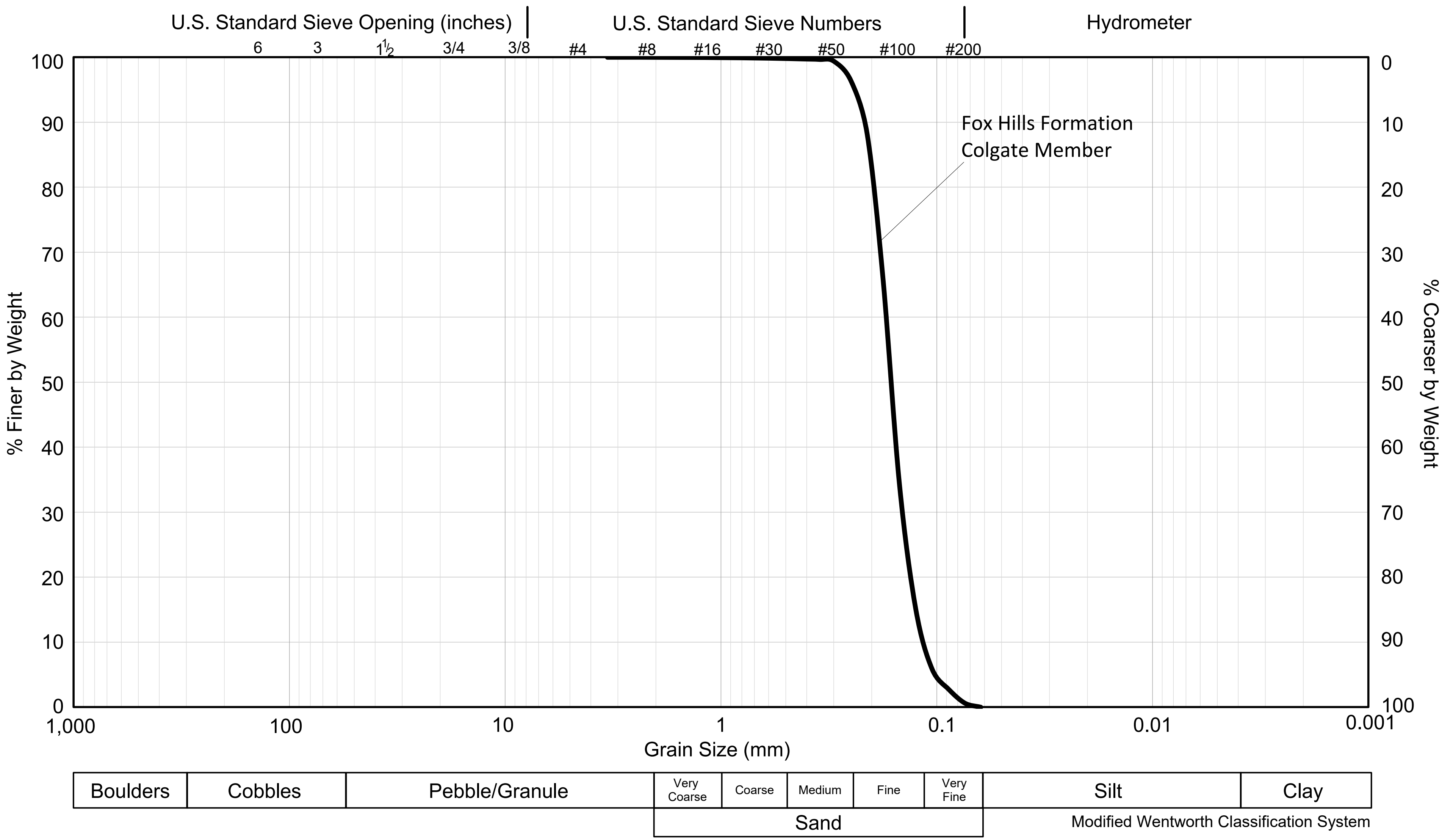


FIGURE A-I-3.
Grain-size distribution diagram (sieve analysis) for sandstone sample no. 3 from the Colgate Member of the Fox Hills Formation in southeastern Morton County, North Dakota.

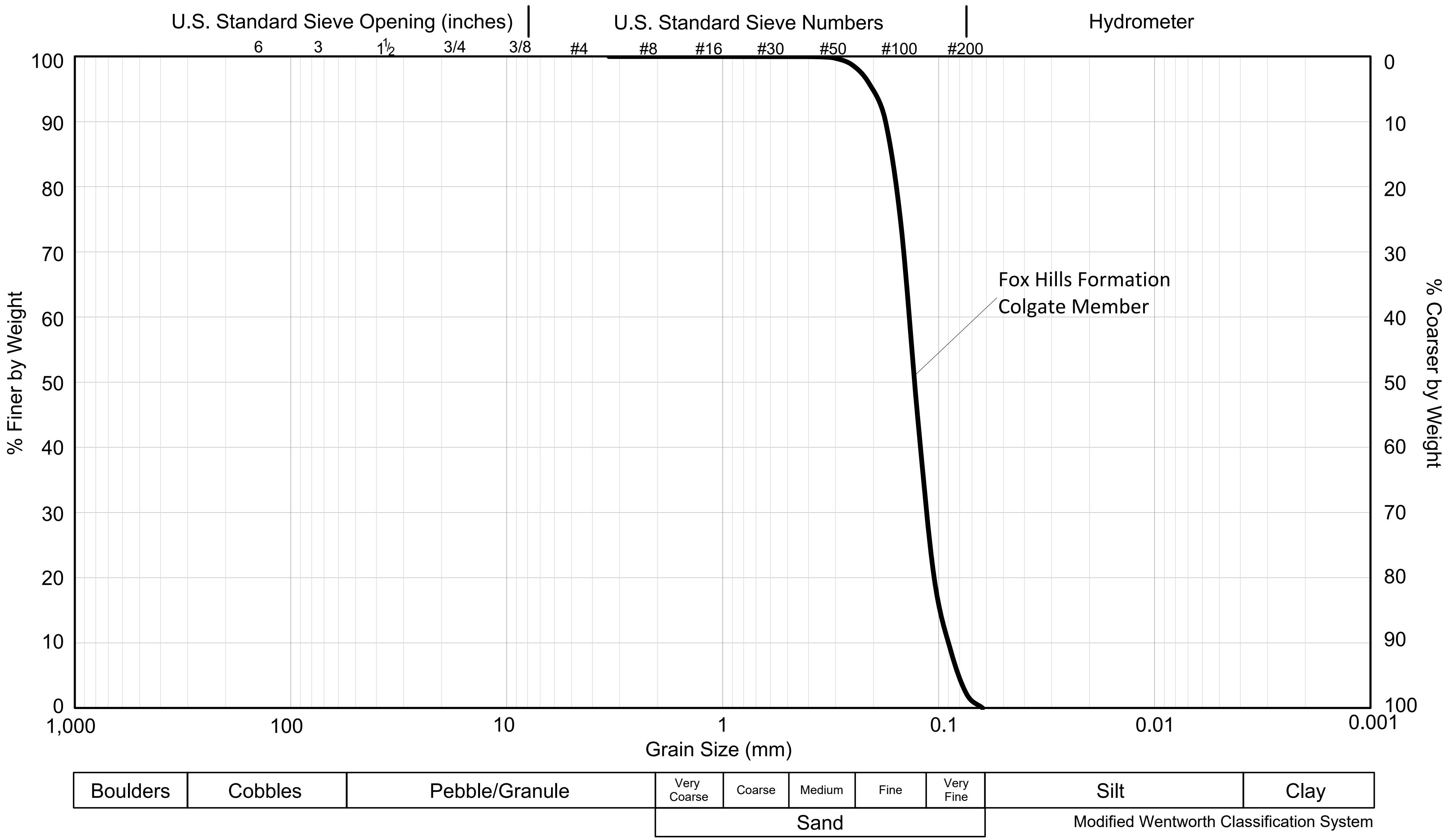


FIGURE A-I-4.
Grain-size distribution diagram (sieve analysis) for sandstone sample no. 6 from the Colgate Member of the Fox Hills Formation in southwestern Bowman County, North Dakota.

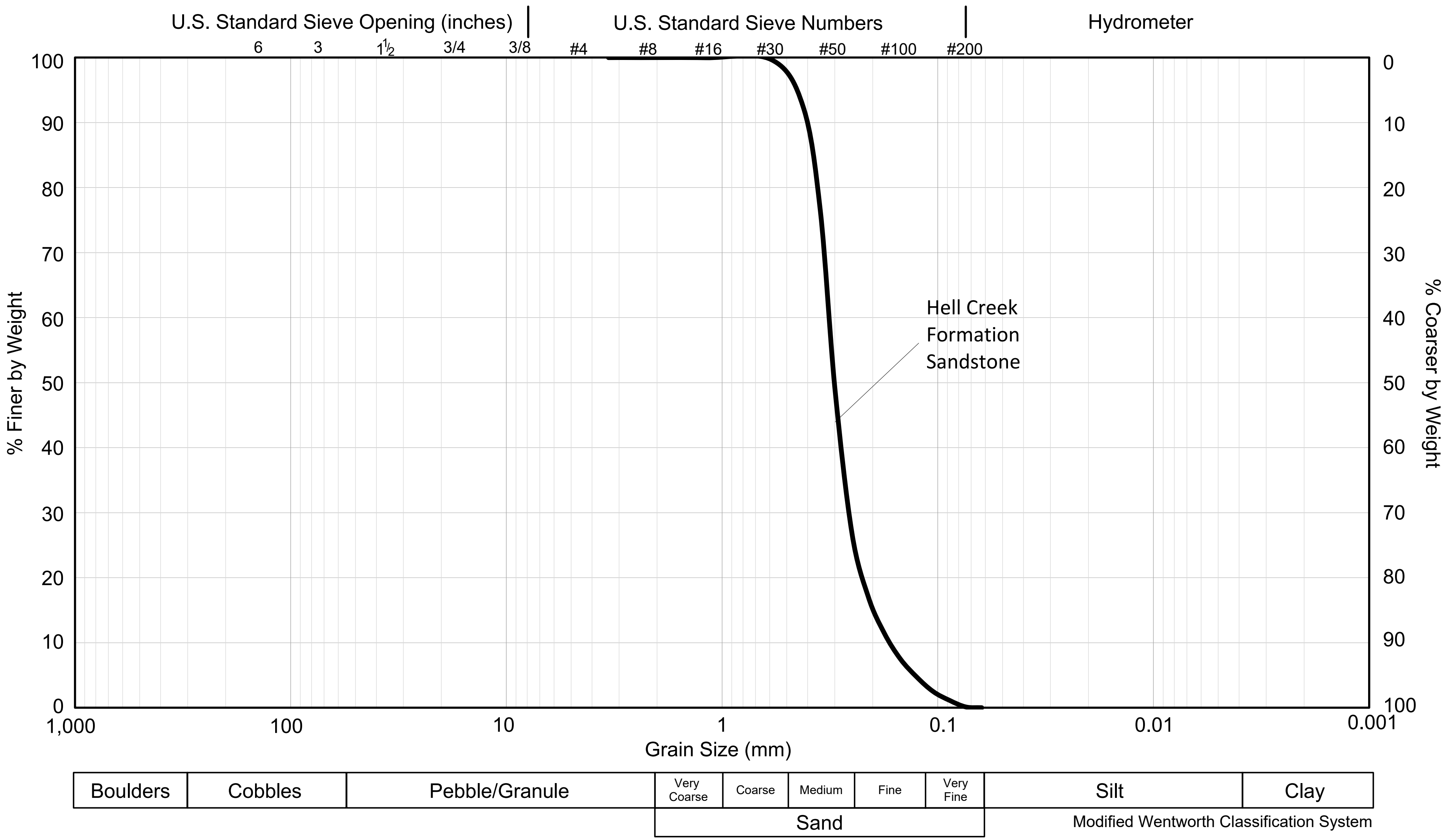


FIGURE A-I-5.
Grain-size distribution diagram (sieve analysis) for sandstone sample no. 7 from the Hell Creek Formation in Slope County, North Dakota.

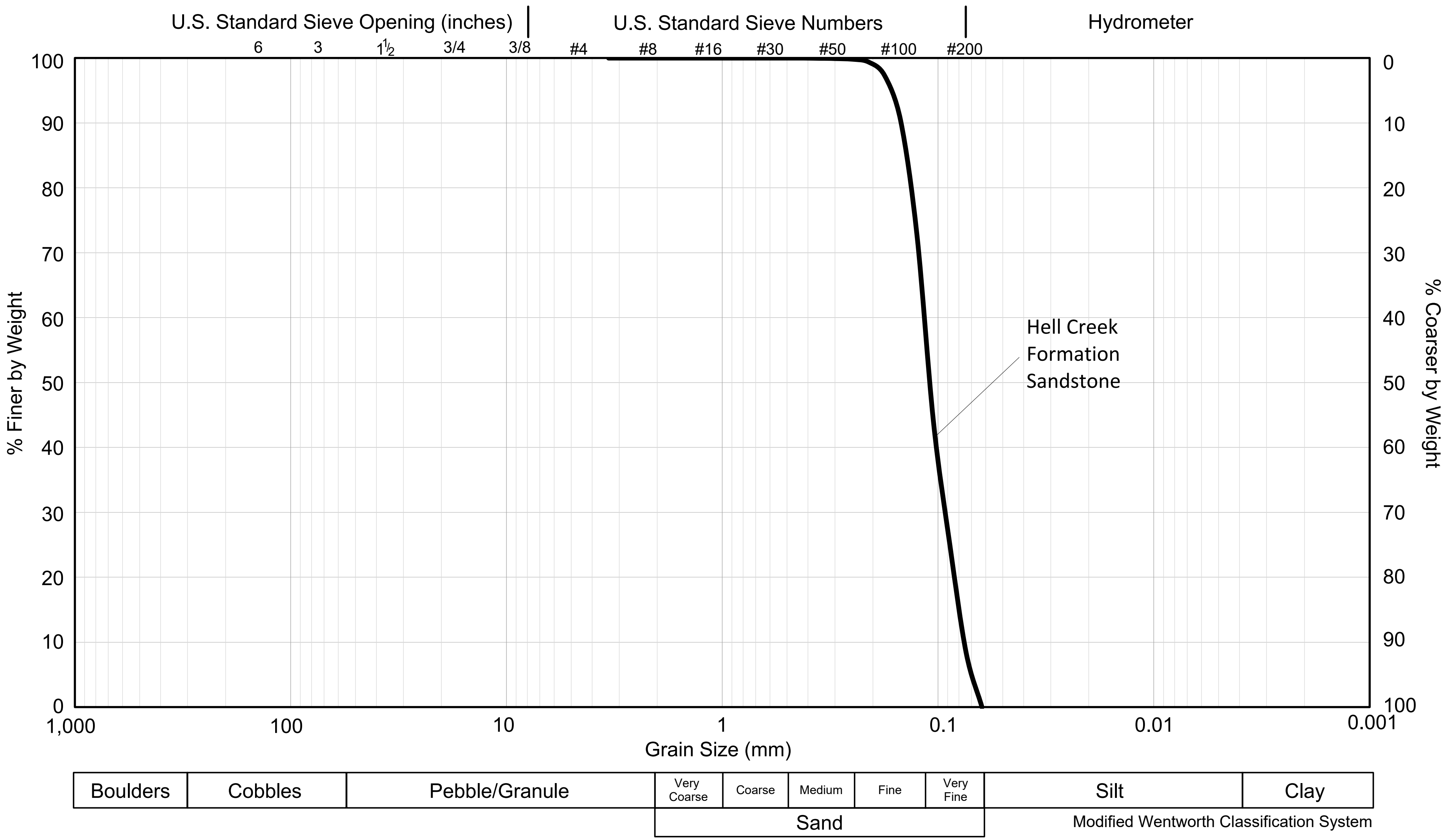


FIGURE A-I-6.
Grain-size distribution diagram (sieve analysis) for sandstone sample no. 8 from the Hell Creek Formation in Slope County, North Dakota.

APPENDIX II.

X-ray diffraction (XRD) Mineralogical Analysis of Sandstones from the Fox Hills and Hell Creek Formations in Southwestern and South-Central North Dakota

INTRODUCTION

Sampling and mineralogical analysis of sandstones of the Fox Hills and Hell Creek Formations from outcrops in southwestern and south-central North Dakota was conducted during the 2022 field season. A total of 15 samples from 8 locations were analyzed for bulk and washed-sized fraction mineralogy. X-ray diffraction (XRD) mineralogical analysis was completed by Lonquist Frac Sand Services in Austin, Texas.

XRD-METHODOLOGY

The analytical patterns shown in the phase diagrams in this appendix, Figures A-II-1 through A-II-15, represent the identified mineralogical phases contained within each sample analyzed. The identified phases were further quantified with the aid of a Rietveld refinement which more precisely defines the diffraction phase diagram peak heights and position, providing greater accuracy in the mineralogic quantification. For these samples, an XRD analysis prior to sample washing and size grading was performed to provide additional information on sandstone composition in situ.

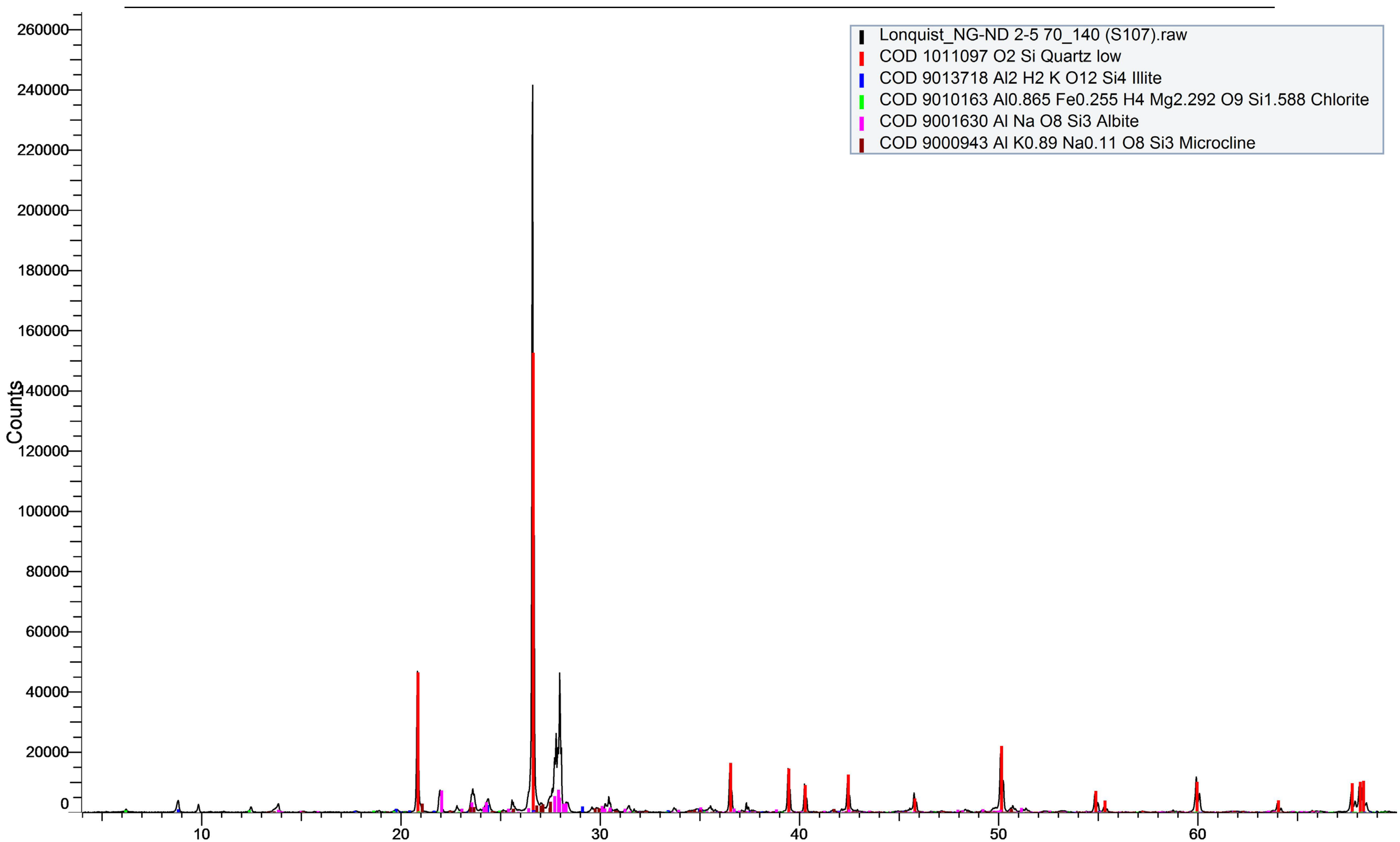


FIGURE A-II-1.
XRD phase diagram of sandstone sample no. 1 (washed 70/140) from the Colgate Member of the Fox Hills Formation in Bowman Co., N.D.

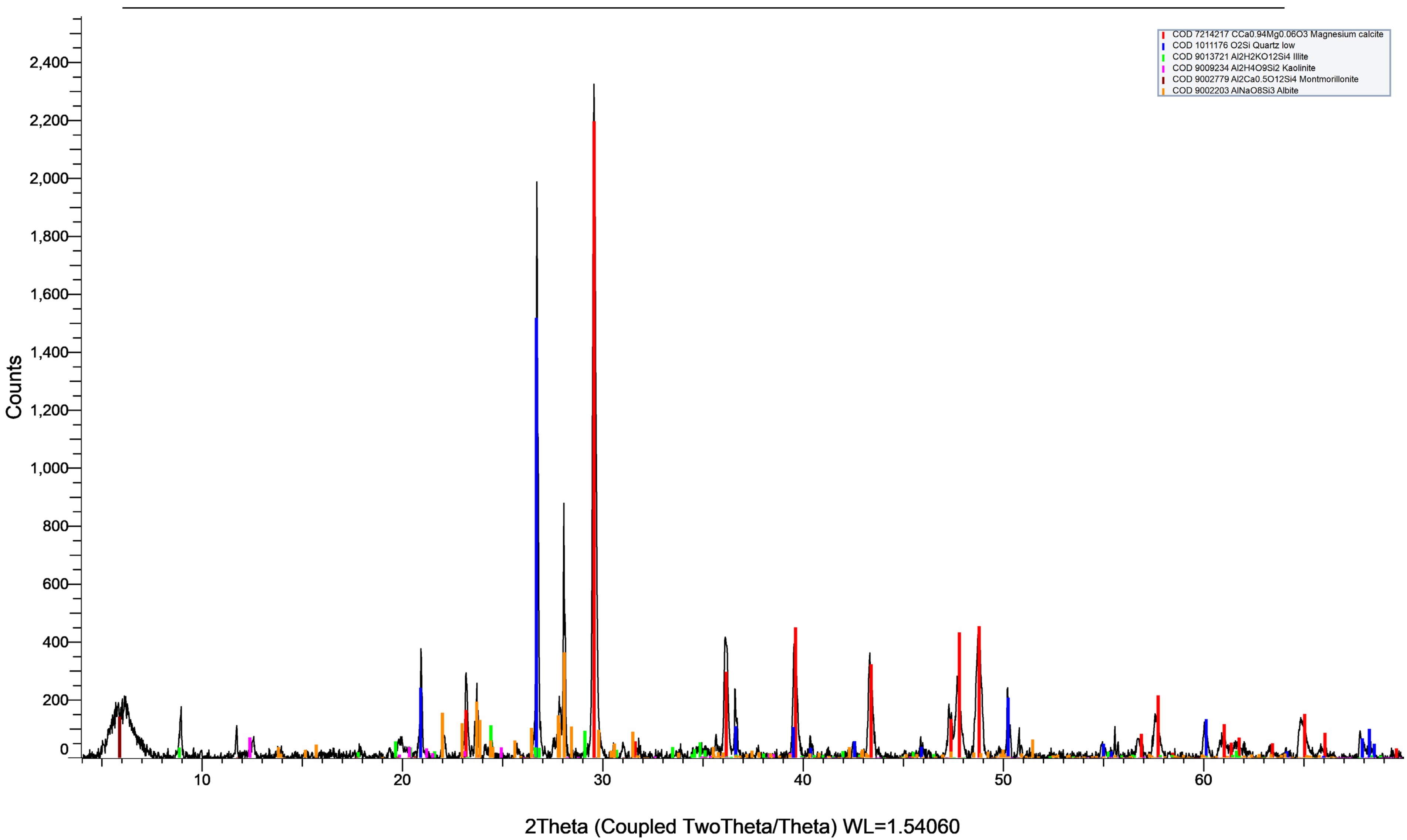


FIGURE A-II-2.
XRD phase diagram of sandstone sample no. 2 (bulk unwashed) from the Bullhead Member of the Fox Hills Formation in Southeastern Morton Co., N.D.

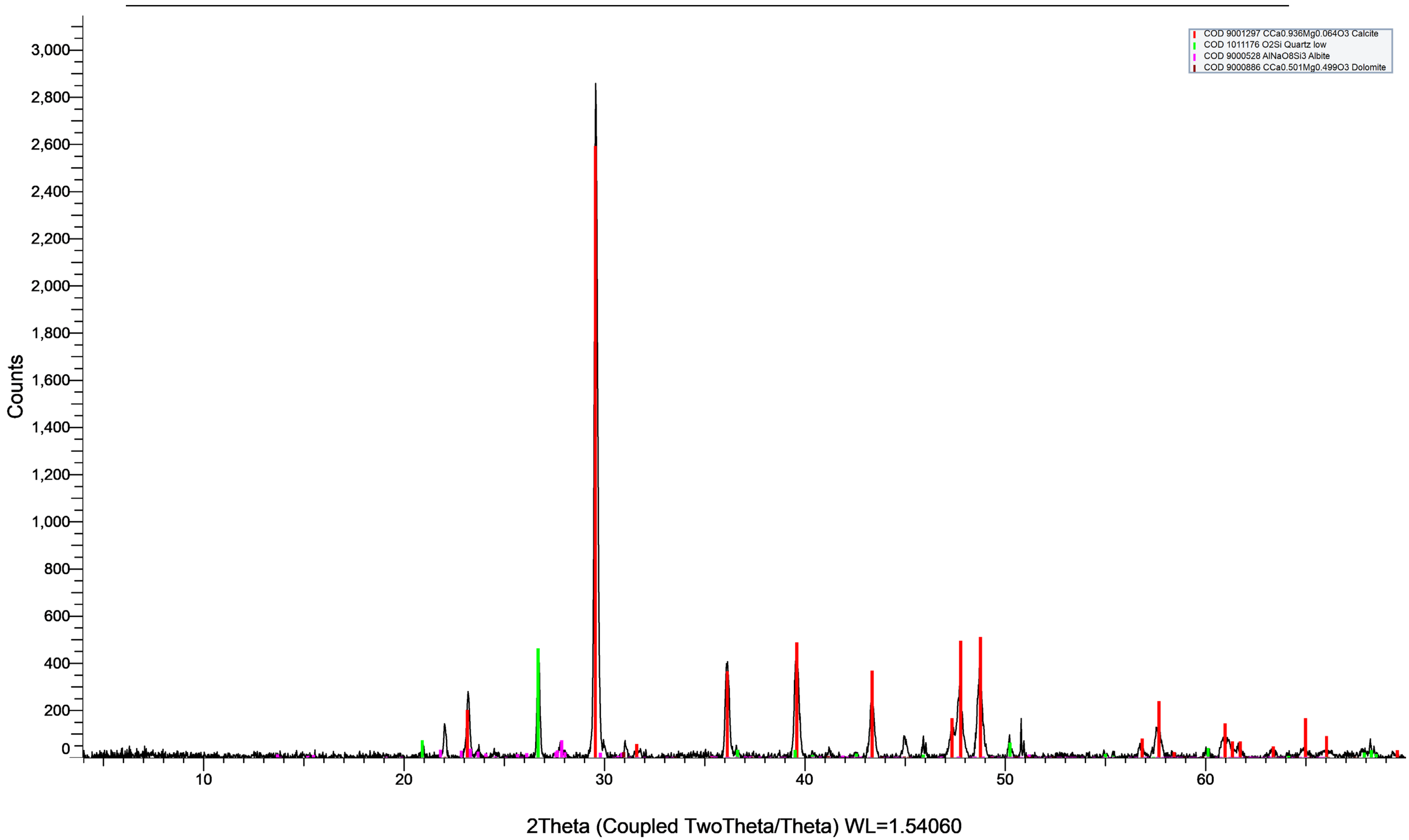


FIGURE A-II-3.
XRD phase diagram of sandstone sample no. 2 (washed 40/140) from the Bullhead Member of the Fox Hills Formation in Southeastern Morton Co., N.D.

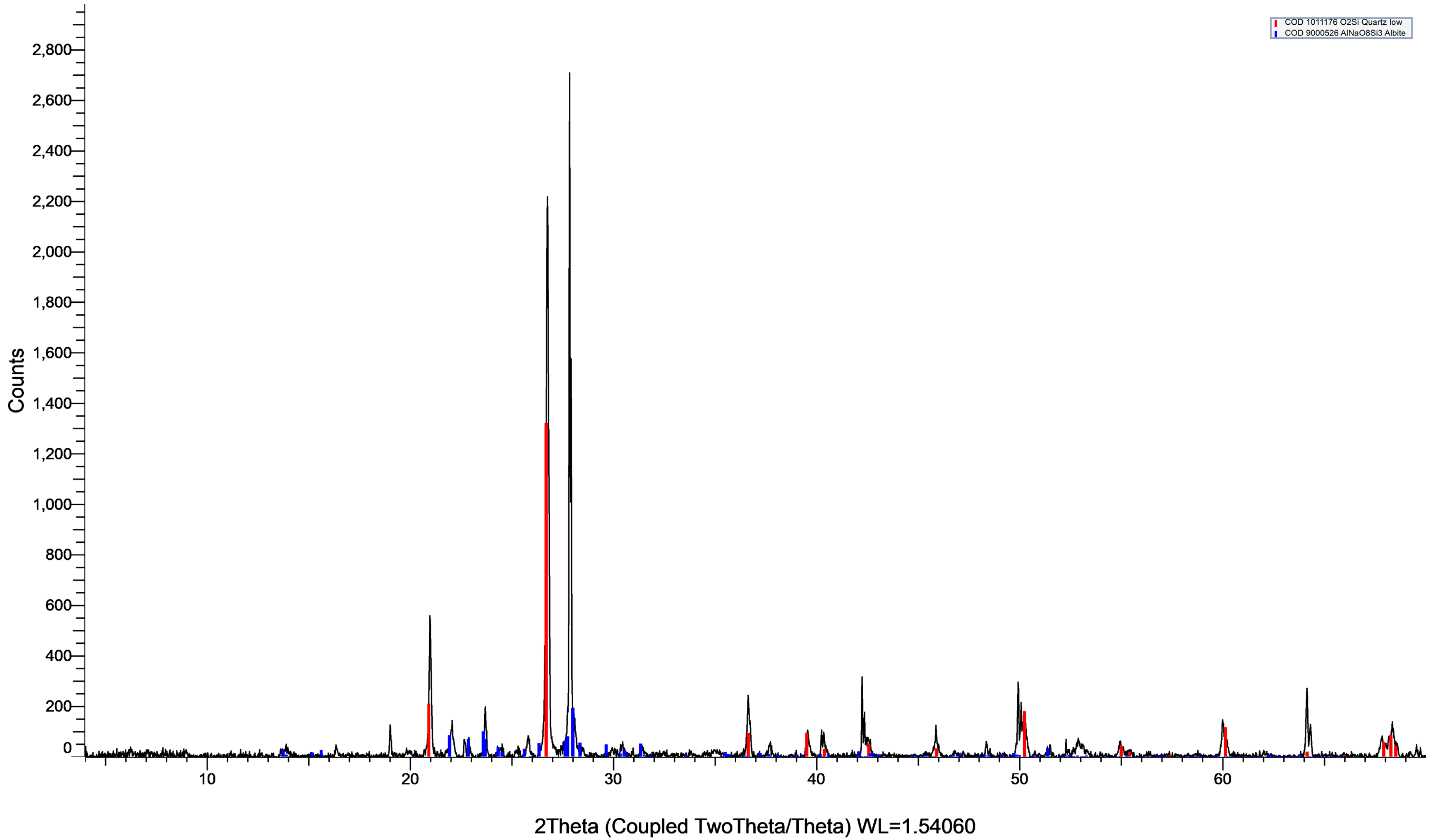


FIGURE A-II-4.
XRD phase diagram of sandstone sample no. 3 (bulk unwashed) from the Colgate Member of the Fox Hills Formation in Southeastern Morton Co., N.D.

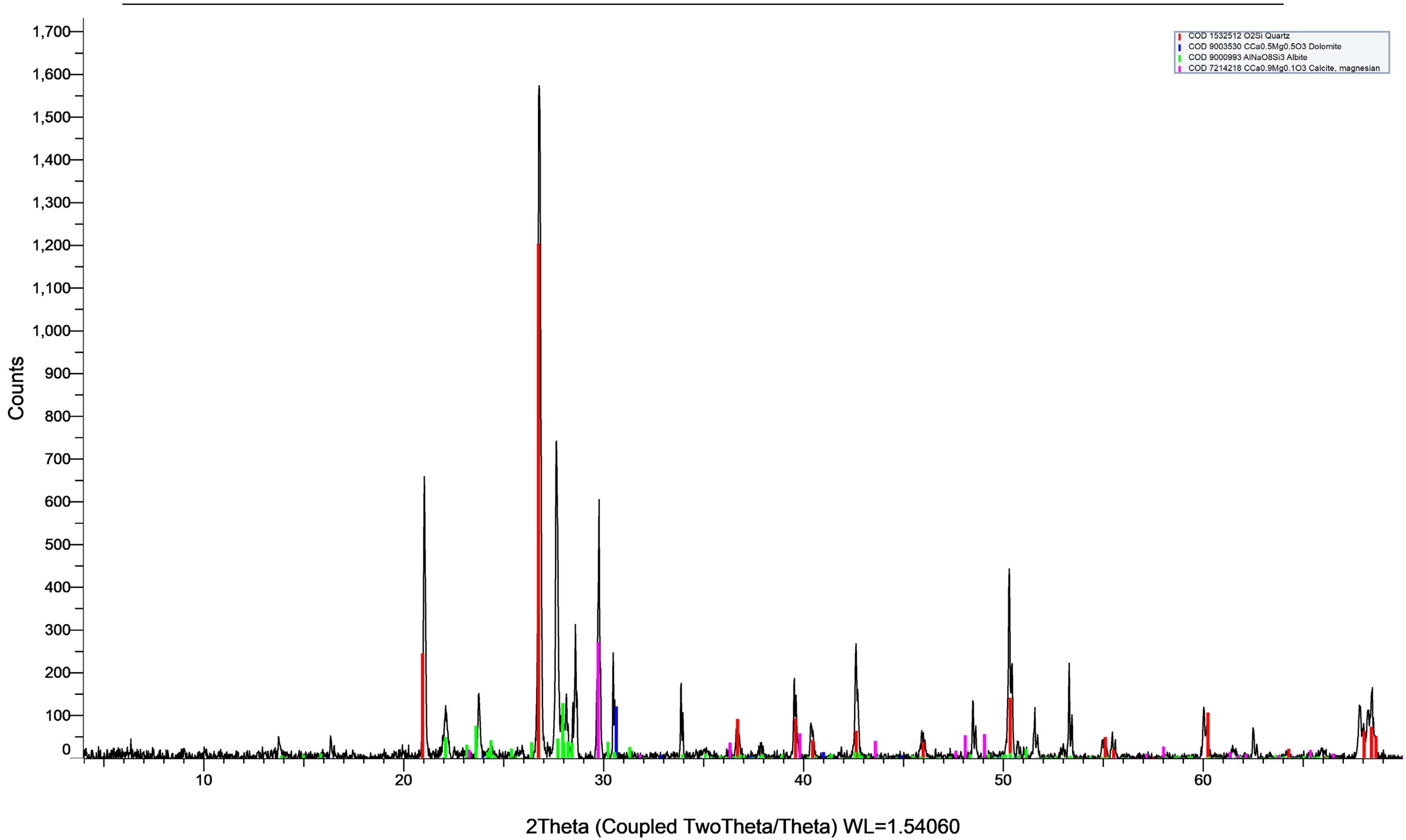


FIGURE A-II-5.
XRD phase diagram of sandstone sample no. 3 (washed 40/140) from the Colgate Member of the Fox Hills Formation in Southeastern Morton Co., N.D.

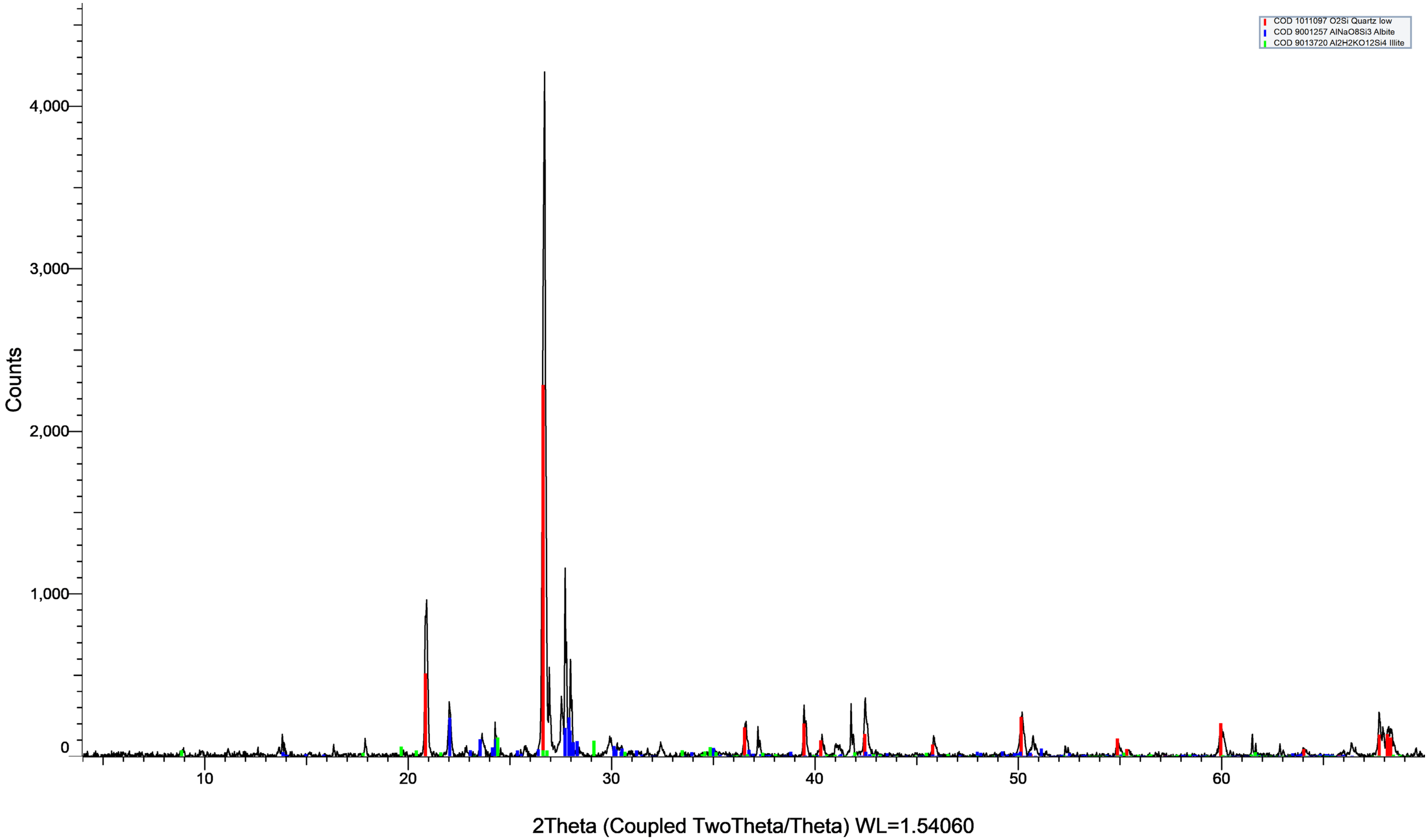


FIGURE A-II-6.
XRD phase diagram of sandstone sample no. 4 (bulk washed) from the Bullhead Member of the Fox Hills Formation in Southeastern Morton Co., N.D.

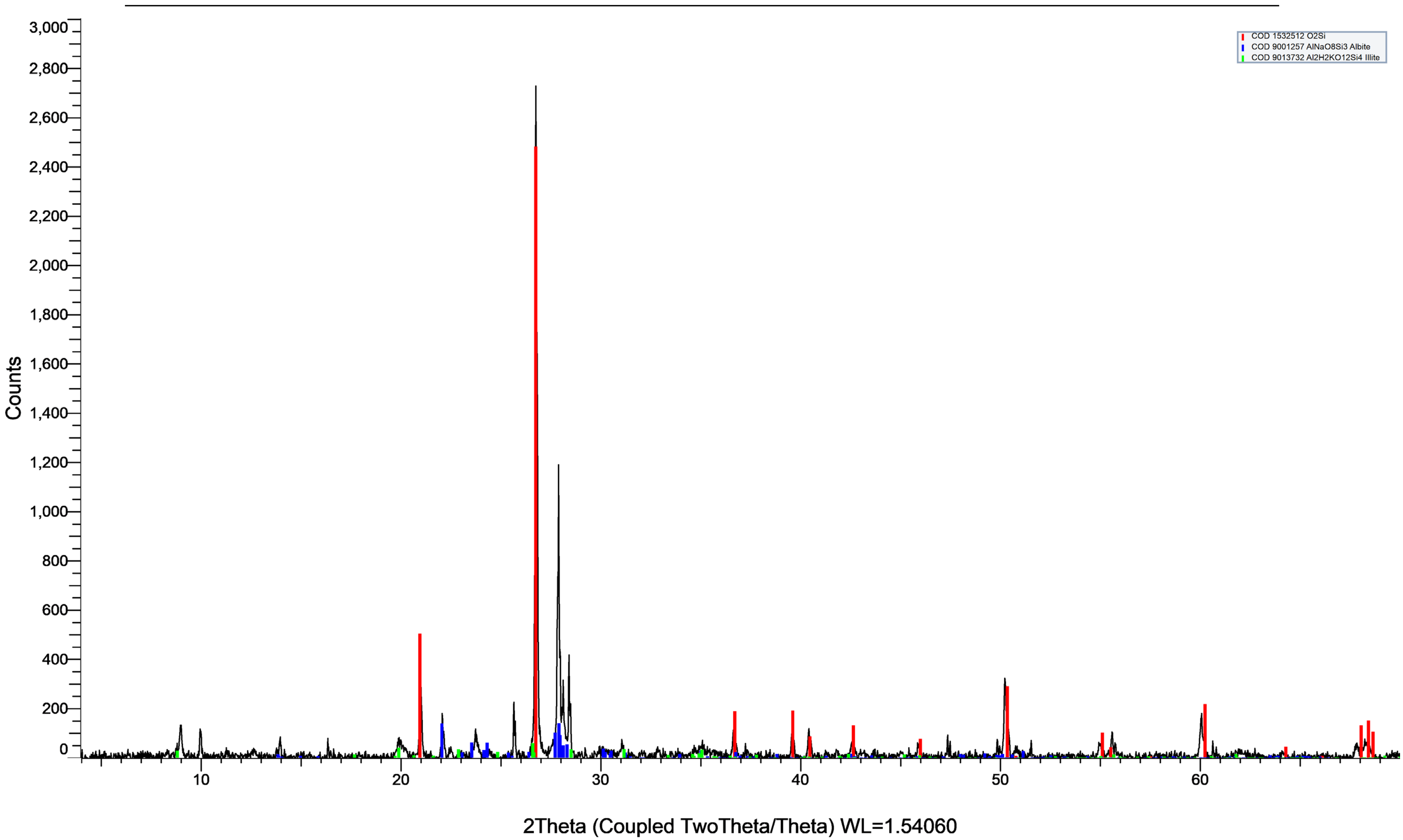


FIGURE A-II-7.
XRD phase diagram of sandstone sample no. 4 (bulk unwashed) from the Bullhead Member of the Fox Hills Formation in Southeastern Morton Co., N.D.

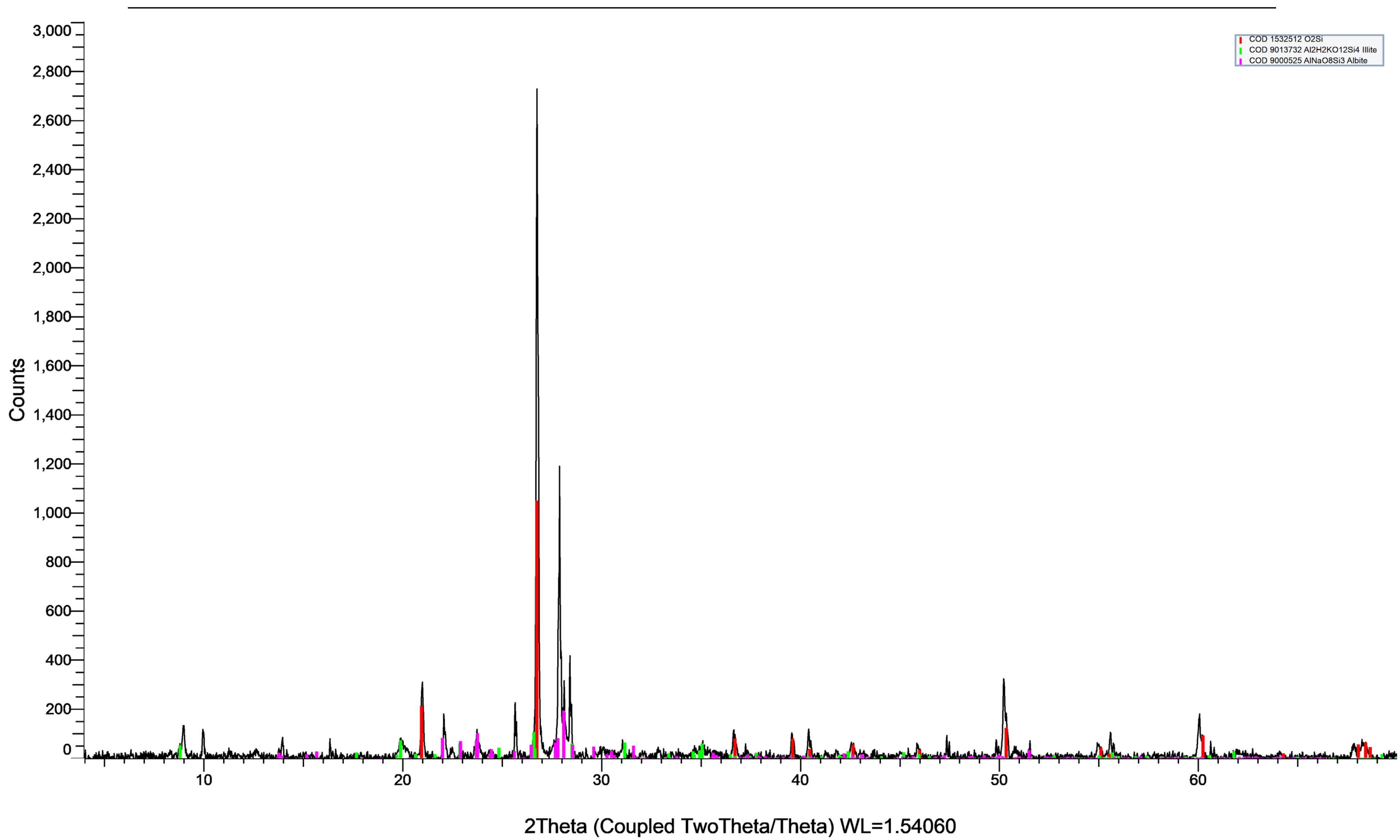


FIGURE A-II-8.
XRD phase diagram of sandstone sample no. 5 (bulk unwashed) from the Colgate Member of the Fox Hills Formation in Southeastern Morton Co., N.D.

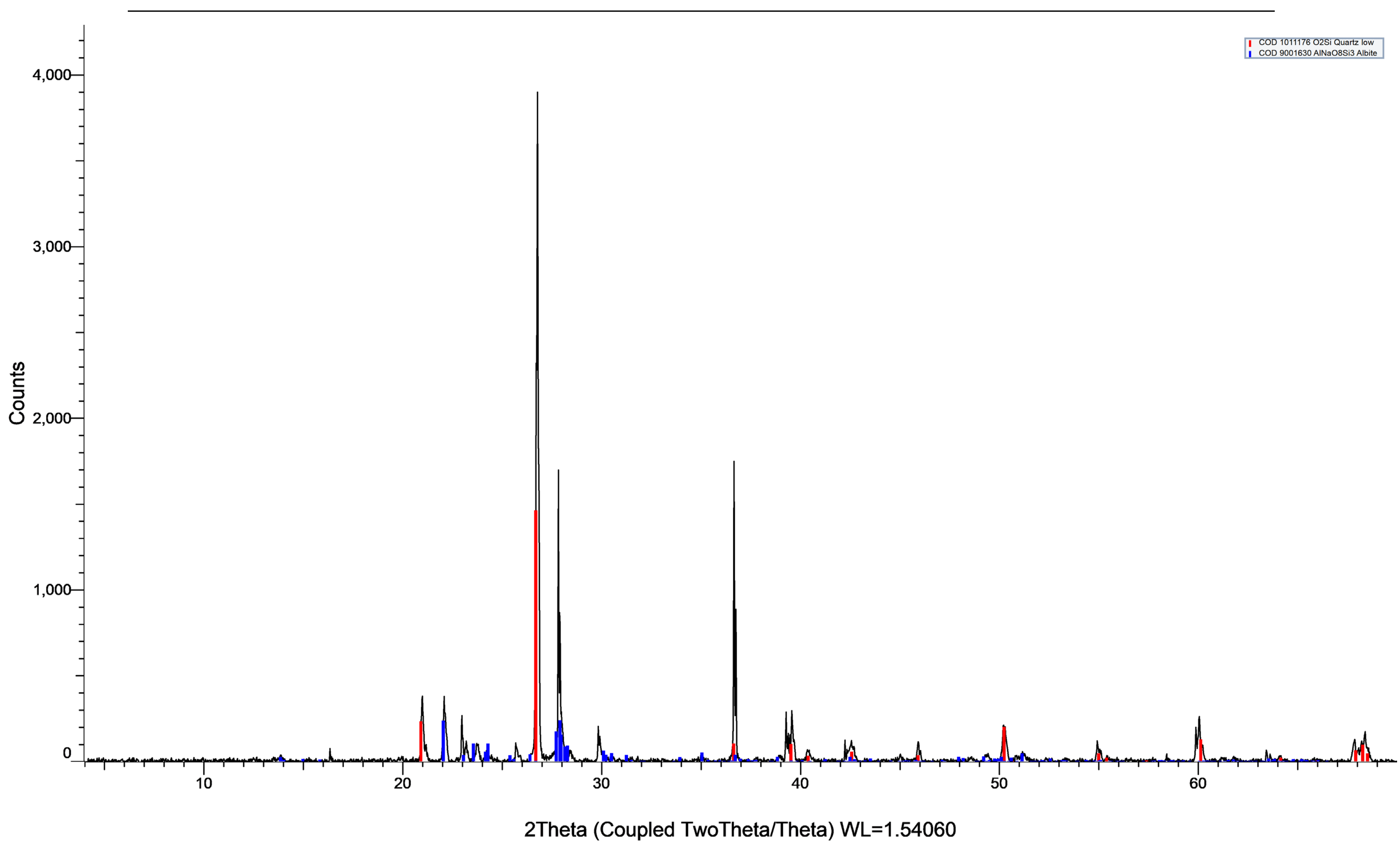


FIGURE A-II-9.
XRD phase diagram of sandstone sample no. 5 (bulk washed) from the Colgate Member of the Fox Hills Formation in Southeastern Morton Co., N.D.

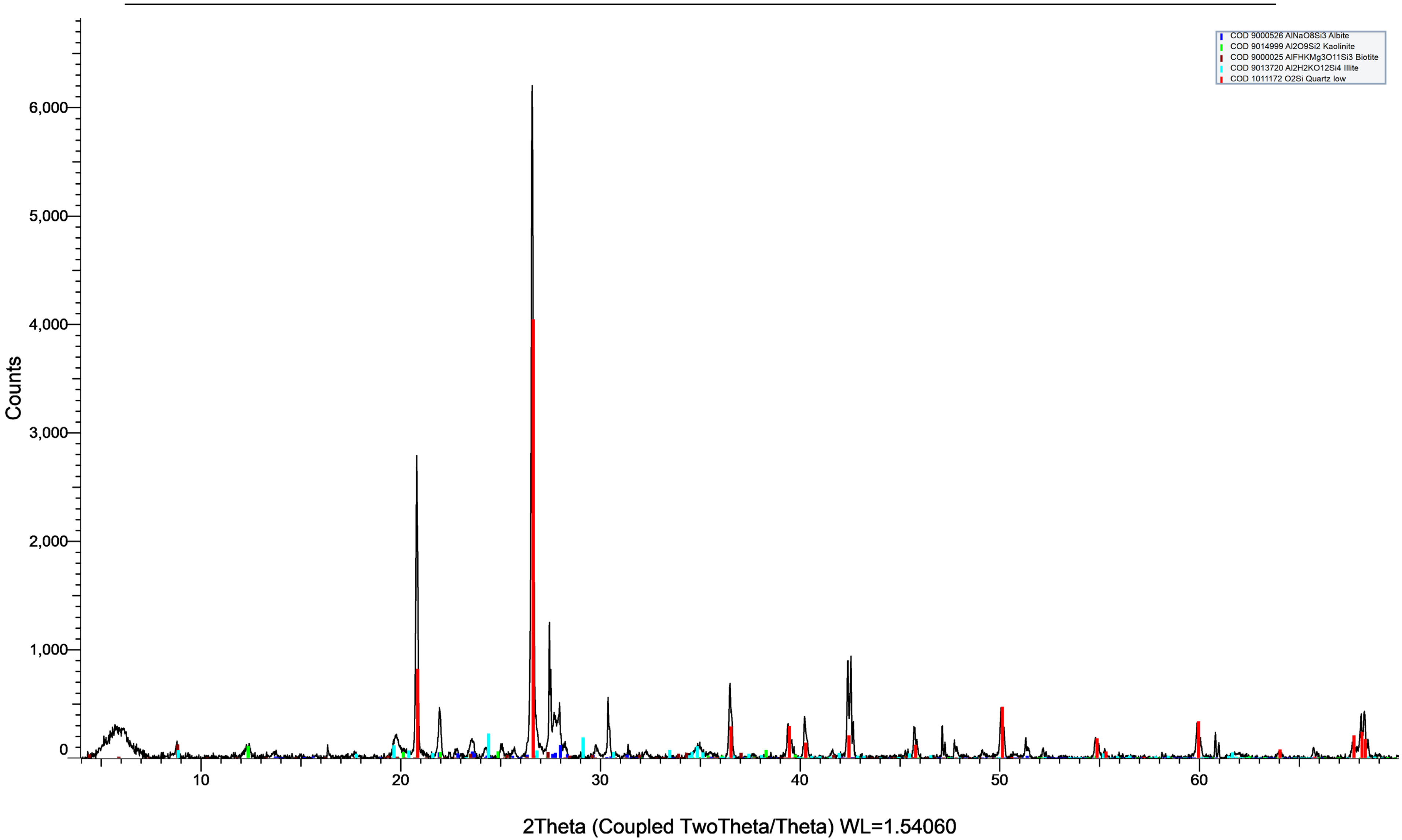


FIGURE A-II-10.
XRD phase diagram of sandstone sample no. 6 (bulk unwashed) from the Colgate Member of the Fox Hills Formation in Southeastern Morton Co., N.D.

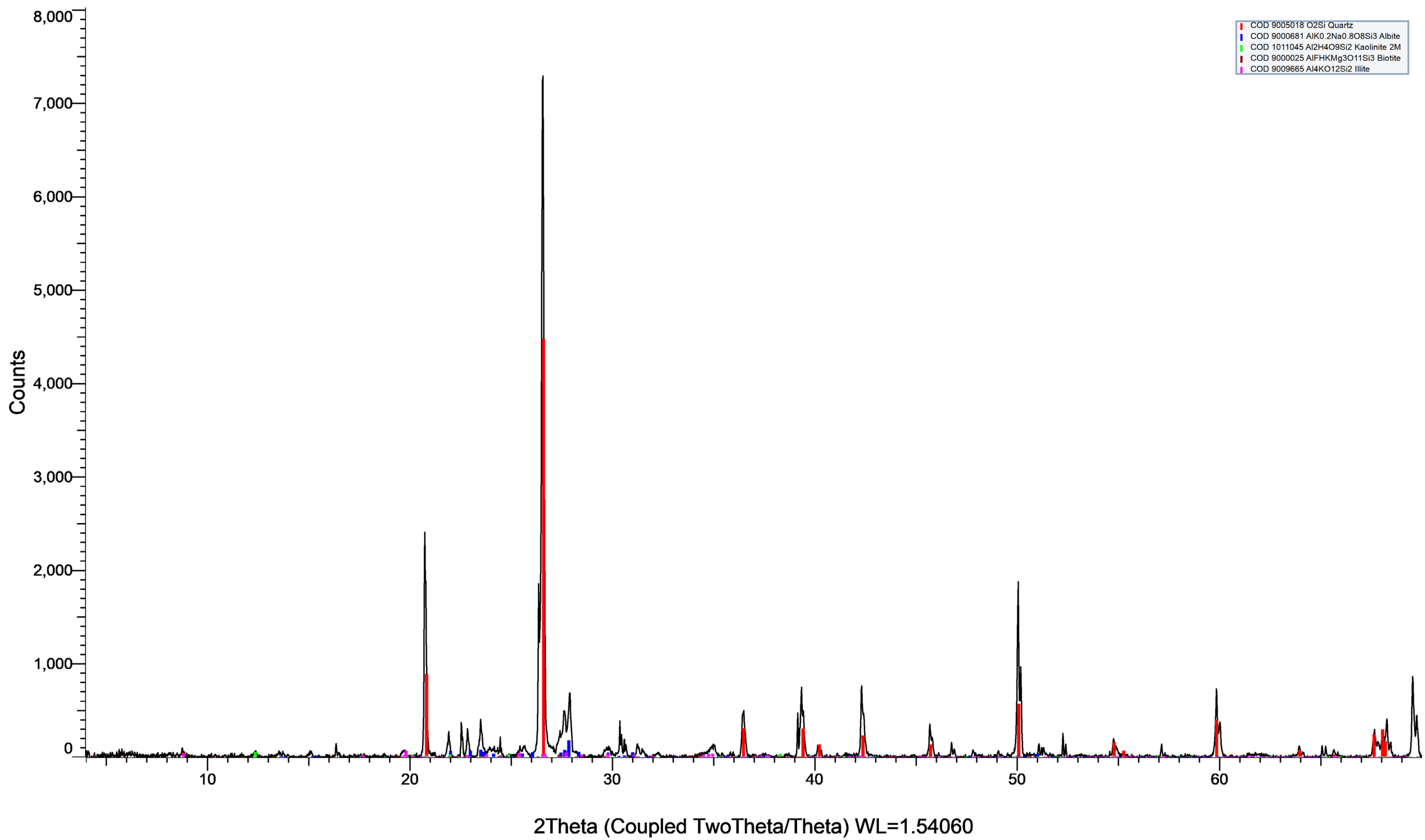


FIGURE A-II-11.
XRD phase diagram of sandstone sample no. 6 (washed 70/140) from the Colgate Member of the Fox Hills Formation in Southeastern Morton Co., N.D.

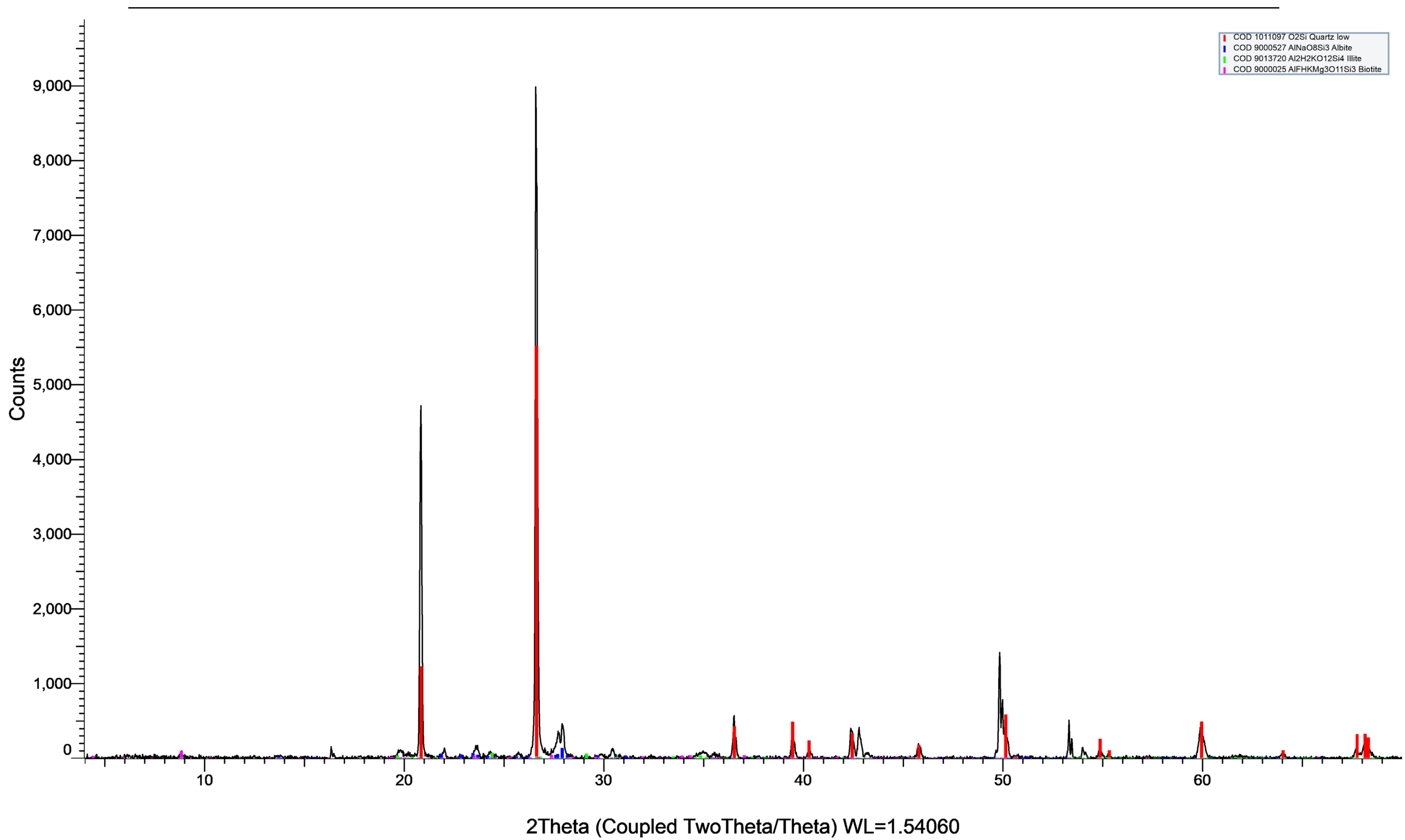


FIGURE A-II-12.
XRD phase diagram of sandstone sample no. 7 (bulk unwashed) from the Hell Creek Formation in Southwestern Slope Co., N.D.

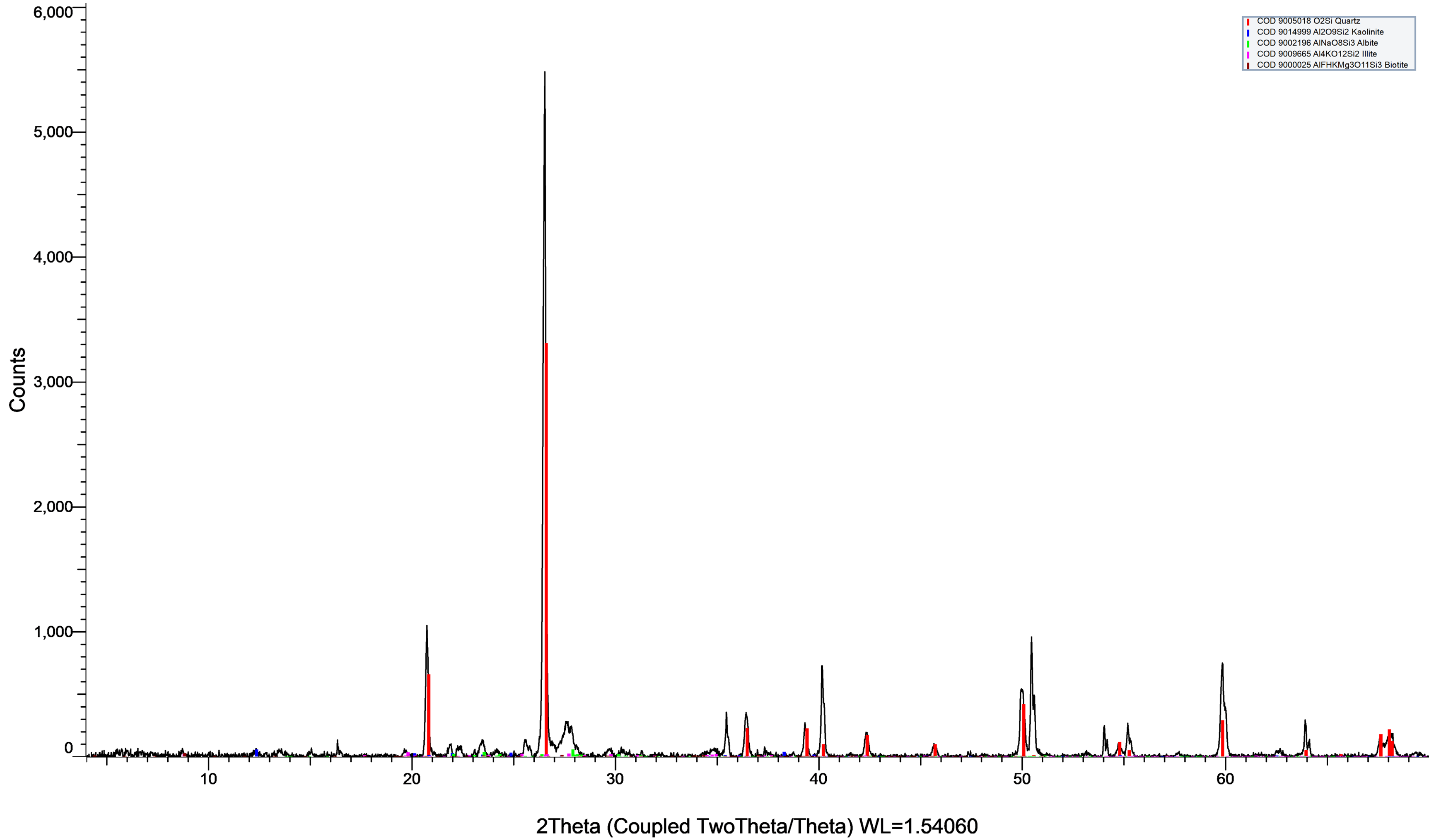


FIGURE A-II-13.
XRD phase diagram of sandstone sample no. 7 (washed 70/140) from the Hell Creek Formation in Southwestern Slope Co., N.D.

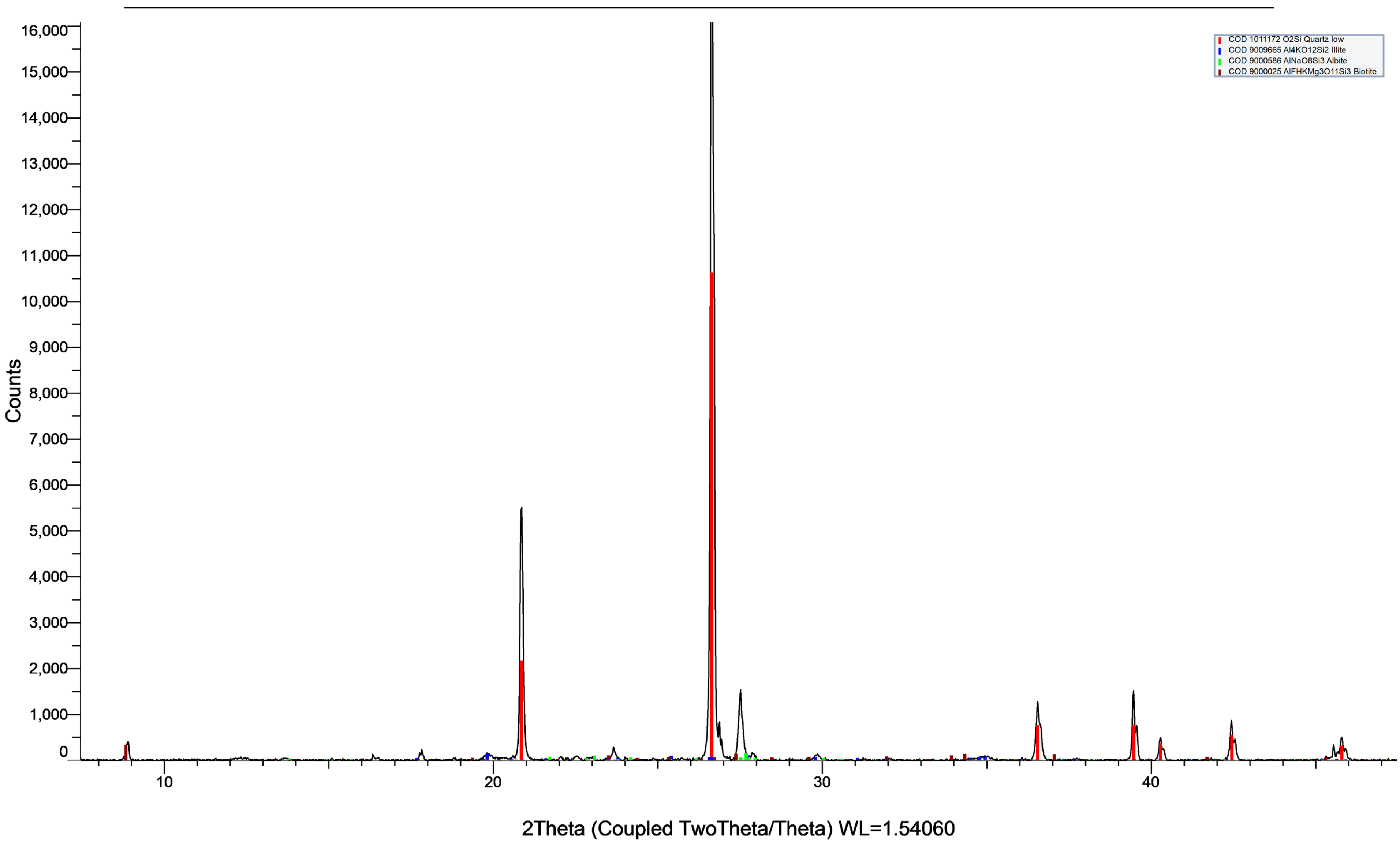


FIGURE A-II-14.
XRD phase diagram of sandstone sample no. 8 (bulk unwashed) from the Hell Creek Formation in Southwestern Slope Co., N.D.

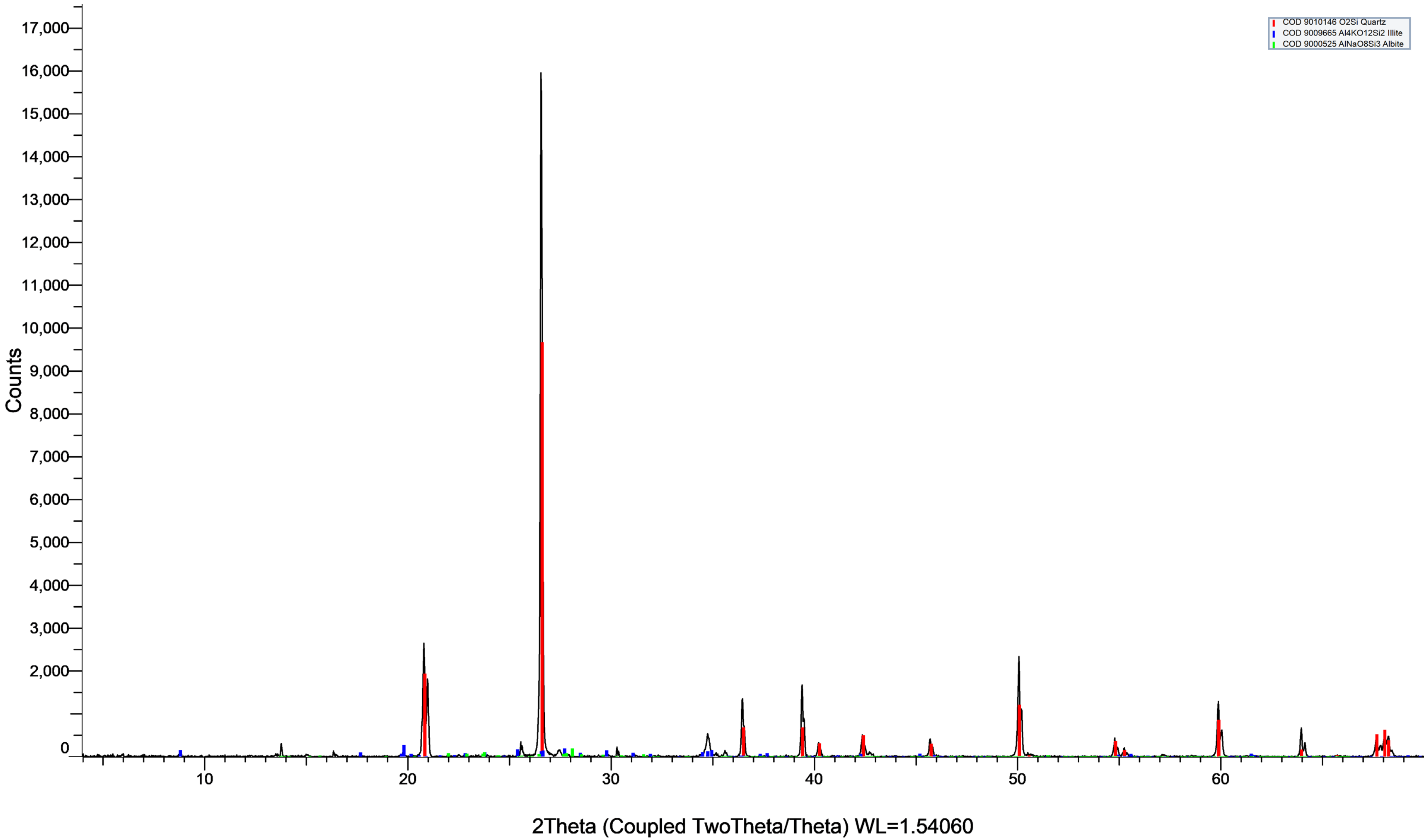


FIGURE A-II-15
XRD phase diagram of sandstone sample no. 8 (washed 70/140) from the Hell Creek Formation in Southwestern Slope Co., N.D.

APPENDIX III.

Testing Specifications and Recommendations for Natural Sand Proppants

Provided below is a summary of the current testing specifications and recommendations for natural sand proppants characterized for use in the hydraulic fracturing of oil and gas wells. These specifications and recommendations are summarized from current recommended specifications published by the International Organization for Standardization (ISO), the American National Standards Institute (ANSI), the American Petroleum Institute (API), and current industrial practice.

GRAIN-SIZE DISTRIBUTION (SIEVE ANALYSIS)

It is recommended that a minimum of 90% of the tested sand fall between the designated sieve sizes, meaning that for a 30/50 sized sand, 90% would pass the coarser primary sieve (i.e. the No. 30 sieve), and be retained on the finer secondary sieve selected (i.e. the No. 50 sieve).

SPHERICITY AND ROUNDNESS (PARTICLE SHAPE FACTORS)

Natural sands used in the hydraulic fracturing of oil and gas wells are recommended to have particle sphericity and roundness values of 0.6 or greater as determined by visual-manual comparison of sand grains under the microscope or through evaluation of suitable photomicrographs.

ACID SOLUBILITY

Evaluation of the solubility of sand in a 12-3 hydrochloric (HCL)-hydrofluoric (HF) acid gives a measure of the amount of undesirable and potentially deleterious “contaminants” such as carbonates, feldspars, iron oxides, and clays that are found in the sand. It is recommended that sands sized in the range from 6/12 to 30/50 contain no more than two percent (by weight) HCL-HF soluble constituents, and sands sized in the range from 40/70 to 70/140 contain no more than three percent (by weight) HCL-HF soluble constituents.

TURBIDITY

The amount of suspended clay, silt, or finely divided organic sediment in the water is a measure of a sand sample's turbidity. It is recommended that natural sands used as proppants have turbidity values no greater than 250 Formazin Turbidity Units (FTU).

CRUSH RESISTANCE

A sand sample’s resistance to crushing is an important characteristic in comparing different types of proppant sand and is performed by subjecting a particular sand sample to a predetermined level of stress and measuring (in percent by weight) the amount of crushed material (i.e. fines) generated in a two-inch diameter pistoncrushing cell. A crush resistance K-value is determined as the highest stress level at which no more than 10% crushed material is generated (rounded down to the nearest 1,000 psi). For a natural sand proppant sized at 6/12, it is recommended that no more than 20% of fines are generated when subjected to an applied stress of 2,000 pounds per square inch (psi). For a natural sand proppant sized at 8/16, it is recommended that no more than 18% of fines are generated when subjected to an applied stress of 2,000 psi. For a natural sand proppant sized at 12/20, it is recommended that no more than 16% of fines are generated when subjected to an applied stress of 3,000 psi. For a natural sand proppant sized at 16/30, it is recommended that no more than 14% of fines are generated when subjected to an applied stress of 3,000 psi. For a natural sand proppant sized at 20/40, it is recommended that no more than 14% of fines are generated when subjected to an applied stress of 4,000 psi. For a natural sand proppant sized at 30/50, it is recommended that no more than 10% fines are generated when subjected to an applied stress of 4,000 psi. For a natural sand proppant sized at 40/70, it is recommended that no more than 8% fines are generated when subjected to an applied stress of 5,000 psi. For a natural sand proppant sized at 70/140, it is recommended that no more than 6% fines be generated when subjected to applied stress of 5,000 psi.

MINERALOGY

In order to provide an understanding of overall mineralogical character, it is recommended that a qualitative mineralogical analysis be conducted, by Xray diffraction (XRD) methods, on a representative sample of sand that is either being used or being evaluated for use as a natural sand proppant. Evaluation of relative peak heights should be used to estimate the amount of clays present in addition to reporting any minerals found at levels above 1 percent. Sand with a high quartz content is desirable.