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Report of Investigations 115

X-RAY DIFFRACTION ANALYSIS OF POST-CRETACEOUS SAND AND GRAVEL UNITS IN SOUTHEASTERN SOUTH DAKOTA

by

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INTRODUCTION

Several quartz-rich sand and gravel bodies located in southeastern South Dakota have been described in outcrop, drill holes, and cores as being "western derived" in origin or having a Rocky Mountain or Black Hills source. The known "western derived" sands directly overlie bedrock. In southeastern South Dakota, suspected "western derived" sands are commonly preserved beneath glacial sediments where the Cretaceous bedrock forms a high. The deposits are typically clean (having a low clay content), quartz-rich, feldspar-bearing sand and gravel. They appear to be similar to deposits belonging to the Ogallala Group, Bon Homme gravels, and Herrick gravels that have a western source.

This report is the second part of a multi-phase investigation to classify the geologic age and provenance of "western derived" sediments. In this phase, 40 x-ray diffraction (XRD) analyses are presented. Another planned phase of this investigation includes modal mineralogical determinations of samples analyzed in this phase.

Sampling Methods

Sample locations are presented in figure 1. Map location numbers (fig. 1), geologic unit names (if known), collection dates, sample names and numbers, sample source, sample depths, legal locations, and counties are presented in table 1.

Samples are one of three types: 1) spot samples from outcrops, 2) composite samples from drill cuttings or core, or 3) spot samples from previously collected cores. For samples collected from outcrops, the area was cleared of recent debris until a fresh exposure could be seen and a representative sample could be collected. Samples obtained by drilling methods were collected as cuttings from mud rotary methods, auger flights, or a hand-auger bucket, and are composite samples over 5-foot intervals (10-foot intervals for mud rotary samples). Samples were also collected from cores available at the South Dakota Geological Survey. First, spot samples were taken from the cores and analyzed. Then, for each individual core, split samples of these spot samples were combined and analyzed as a composite of the entire sand unit sampled. For additional detailed descriptions of sample preparation, see Haggar and others (2002).

XRD Sample Preparation

Samples for XRD analysis were obtained from the grain-size analysis samples. Approximately 4 to 5 grams of the grain-size analysis samples were obtained by the quartering method of the spot/composite samples. The samples were ground by hand using a Diamonite® mortar and pestle and sieved, until all particles passed through a 100-mesh (150-micron) screen. After sieving, the dry, powdered sample was blended with a spatula to help reestablish a uniform distribution of the minerals throughout the powder.

LABORATORY METHODS

X-ray Diffraction

Powder x-ray diffraction analysis is seemingly the perfect technique for crystalline-mixture analysis, since each component of the mixture produces its characteristic pattern independently of the others, making it possible to identify the various components by unscrambling their superposed patterns (Klug and Alexander, 1974, p. 531). An x-ray beam of known wavelength is focused on a powdered sample and x-ray diffraction peaks are measured using a detector. The d-spacing of the observed diffraction peaks is calculated using Bragg's Law:

$$[n*\lambda = 2d\sin(\theta)]$$

where
n = the order of reflection
λ = wavelength (for copper, 1.5406 angstroms)
d = distance between planes in a crystal lattice, in angstroms
θ = theta, the angle of reflection which equals the angle of incidence

The South Dakota Geological Survey x-ray powder diffractometer is a Scintag Pad V which uses copper Kα radiation on a theta:2-theta, automated, goniometer equipped with a germanium solid-state detector as described in the appendix. For this project, acquisition conditions were 40 kV, 30 mA, and all scans were run at a speed of 2º 2-theta per minute, from 5º to 70º 2-theta, with a continuous step size of 0.02.

Scintag software packages were operated using a personal computer running Windows NT 4 and are available for routine powder diffraction data acquisition, background correction, and peak identification. Raw diffraction scans may be stripped of their Kα2 component, and the background may be corrected with a digital filter (or fourier filter). Peaks may be identified using a variety of algorithms. Observed peak positions were matched against the International Centre for Diffraction Data (ICDD) Joint Committee on Powder Diffraction System (JCPDS) card database (PDF2, release 2001).

Sample Mount Preparation

A representative portion of each sieved sample was placed in a small, round sample holder for XRD analysis. Sample powders were lightly tamped into the round, magnetic sample holders. The sample surface was gently scrapped flush with the top of the sample holder.

No oriented mounts were made or scanned. Thus, the clay mineral groups identified in table 2 are possible or tentative identifications.

Sample Mount Orientation

In an effort to help determine which varieties of feldspar were present in samples with a small amount of feldspar, a limited experiment was conducted. The feldspars were characterized and classified by their chemical composition and also by their structural state, which depends upon the temperature of crystallization and upon subsequent thermal history (Deer and others, 1978, p. 281). In practical terms, this means that XRD peak positions may shift slightly or even move because of chemical and structural variations within the feldspars.

The purpose of this experiment was to determine if the direction of scraping during sample mount preparation, relative to the orientation of the mount on the goniometer, could be used to help maximize weak feldspar XRD peak intensities. Two samples were selected for this analysis. These samples were loaded and the orientation of the scraping direction was labeled on the sample holder. Each mount was scanned four times, with only the scraping direction parameter being changed between runs. It was rotated 90º between each scan.

Semi-quantitative Analysis

In XRD, the intensity of every component's pattern is proportional to the amount of the component present (Klug and Alexander, 1974). For a true quantitative analysis, peak area should be used rather than peak height. However, in general terms and for the purposes of this project, the height of a mineral's primary 100-percent peak relative to the total counts-per-second value of the raw data scan was used to provide a semi-quantitative analysis of the mineral components in the samples. A mineral was categorized as being "abundant" in the sample, if the 100-percent peak was within 81-100 percent of the total counts-per-second value. It was categorized as being "moderate" if the 100-percent peak was within 21-80 percent of the total counts-per-second value, "minor" if the 100-percent peak was within 5-20 percent of the total counts-per-second value, or "trace" if the 100-percent peak was less than 5 percent of the total counts-per-second value.

A category called "possible" was created to list minerals that were likely to be present in the sample based on finding one characteristic peak in the 20º-35º 2-theta range. Usually these minerals had overlapping peaks with minerals present in greater abundance in that sample. For example, potassium feldspar (i.e., orthoclase and/or microcline) has overlapping peaks with quartz and many varieties of plagioclase feldspar. If a large quantity of quartz and a moderate amount of plagioclase were present in a sample, the diagnostic peak position for placing potassium feldspar in the "possible" category was a peak at 27.4º-27.5º 2-theta. The minerals listed in this category usually had intensities of less than 5 percent of the total counts-per-second value.

Another category called "possible low-angle minerals" was created to recognize the likely presence of minerals whose 100-percent peak falls in the "less than 20º 2-theta" category. Even though oriented mounts were not made, an educated guess may be made regarding the presence or absence of certain clay mineral groups. This category was also used to include minerals (like gypsum) that have their 100-percent peak in the low 2-theta range and that are not abundant in the samples.

Sample Chemical Reaction

After each XRD analysis, approximately 30 milligrams of the grain-size analysis sample 150-micron powder was separated for a chemical reaction test. These samples were visually inspected for chemical reaction with a drop of 10-percent hydrochloric acid to test for the presence or absence of calcite.

DATA ANALYSIS

Mineralogical XRD Results

Mineralogical results for the 40 samples are presented in table 2. Additional graphical and interpretive results are available in South Dakota Geological Survey files.

There were 39 of the 40 samples which contained an abundance of quartz with lesser amounts of feldspar. Only one sample (XRD sample number CO131) from Turkey Ridge probably had a larger component of feldspar than quartz present. Usually both plagioclase and potassium feldspar varieties were present in the samples. An illite/mica component was recognized in all the samples. The presence of calcite ranged from absent to moderate in amount. Kaolinite and/or smectite clay varieties appear to be present occasionally.

Sample Mount Orientation Results

For two different samples, it was found that there was a tendency for minor peak maxima in the 25º-30º 2-theta range to be larger if the sample mount was oriented with the scraping direction parallel to, and in the direction of, the diffraction beam orientation. In general, it appears that random orientation of the particles may be maximized with this orientation.

Sample Chemical Reaction Results

Results of chemical reaction with the powdered samples are presented in table 2. Only 11 out of 40 analyses indicated the presence of calcite using this method.

REFERENCES

Deer, W.A., Howie, R.A., and Zussman, J., 1978, *An introduction to the rock forming minerals:* Longman Group Limited, 518 p.

Haggar, T.N.; McCormick, K.A.; Chadima, S.A.; Schulz, L.D., 2002, *Grain-size analysis of post-Cretaceous sand and gravel units in southeastern South Dakota:* South Dakota Geological Survey Report of Investigation 114.

Klug, H.P., and Alexander, L.E., 1974, *X-ray diffraction procedures for polycrystalline and amorphous materials:* Wiley and Sons, 900 p.

Figure 1. Locations of collected samples.

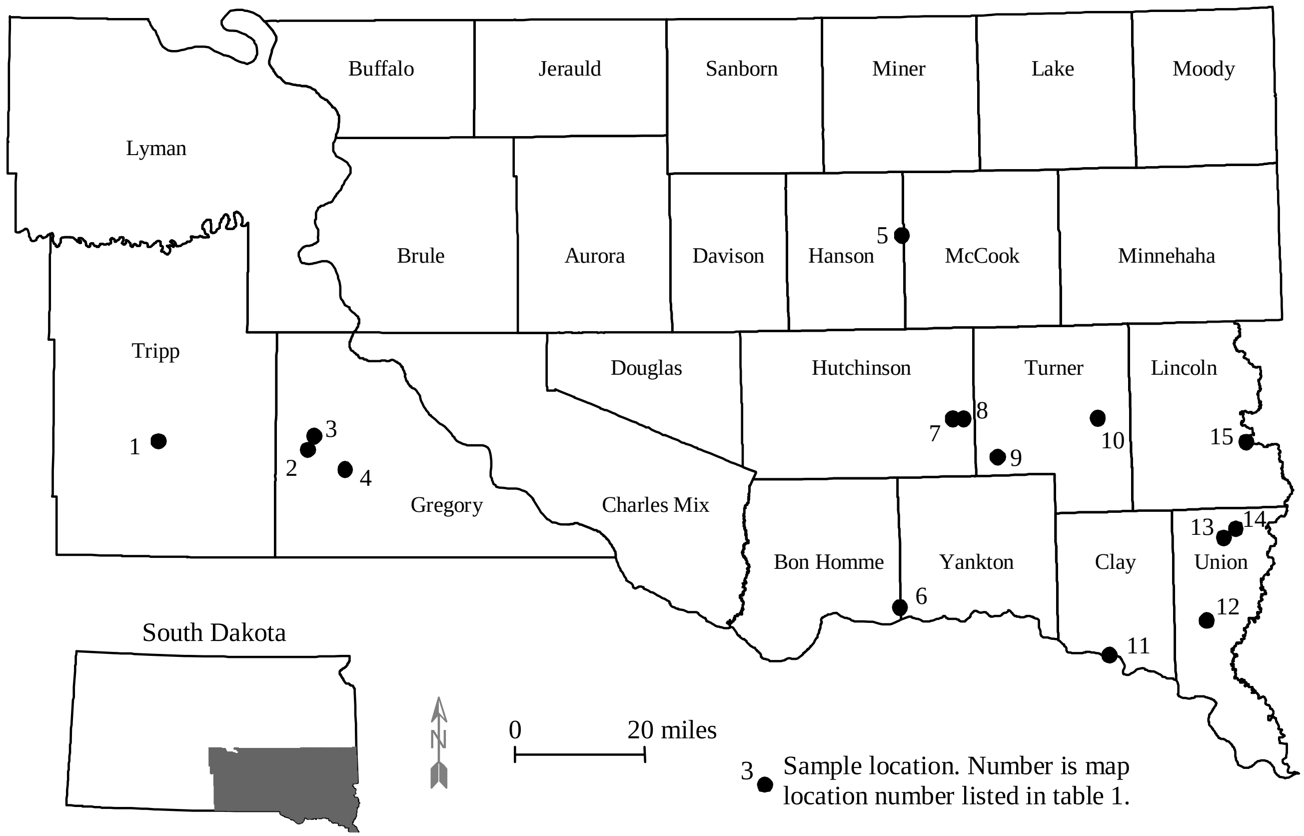

Table 1. Sample collection and location data

Map location number[1]	Geologic group or formation	Collection date	Sample name and number	Sample source	Sample depth (in feet)	Legal location	County
1	Ogallala	06-04-01	R20-01-5	core	27-28 40-41 58.5-59.5 67-68 75-76 88-89 composite[2]	SW SW SW SW sec. 31, T. 98 N., R. 76 W.	Tripp
2	Ogallala – Ash Hollow Member?	05-23-01	Gregory City 5-23-5	outcrop	surface	SW NW NW NW sec. 12, T. 97 N., R. 73 W.	Gregory
2	Ogallala – Ash Hollow or Valentine Member	05-23-01	Gregory City 5-23-6	outcrop	surface	SW NW NW NW sec. 12, T. 97 N., R. 73 W.	Gregory
3	Ogallala – Ash Hollow Member	05-23-01	Ash Hollow 5-23-4	Road cut/ outcrop	surface	NW NW NW NW sec. 31, T. 98 N., R. 72 W.	Gregory
4	“western derived” sand	05-23-01	Herrick gravel 5-23-3	sand pit	surface	NW NE NE NE sec. 26, T. 97 N., R. 72 W.	Gregory
5	Sioux Quartzite	05-10-01	Spencer quarry 5-10-1	quarry	surface	NE sec. 24, T. 103 N., R. 57 W.	Hanson
6	“western derived” sand	05-22-01	Bon Homme 5-22-2	sand pit	surface	SE SE SE SE sec. 12, T. 93 N., R. 58 W.	Bon Homme
7	Uncertain – Ogallala?	04-24-01	Turkey Ridge R20-01-1	Core	105-110 117 127 141-145 151 160-161 173-174 180-185 composite[2]	SW SW SW SW sec. 15, T. 98 N., R. 56 W.	Hutchinson
8	Uncertain – “western derived”? sand	05-01-01	Turkey Ridge R20-01-2	mud rotary cuttings	40-50	SW SW SW SW sec. 14, T. 98 N., R. 56 W.	Hutchinson

Table 1 – continued

Map location number[1]	Geologic group or formation	Collection date	Sample name and number	Sample source	Sample depth (in feet)	Legal location	County
9	Uncertain – Ogallala?	06-25-01	Turkey Ridge R20-87-14	core	200-205	NW NE NE NW sec. 22, T. 97 N., R. 55 W.	Turner
					205-210		
					210-215		
					225-230		
					230-235		
					composite[2]		
10	Glacial outwash	05-22-01	Hurley 5-22-5	small gravel pit	surface	NW NW NW SW sec. 20, T. 98 N., R. 52 W.	Turner
11	Recent sand and gravel	05-21-01	Missouri River 5-21-1	outcrop	surface	SW NE sec. 7, T. 32 N., R. 4 E.	Clay
12	"western derived" sand	06-25-01	Heeren core R20-88-30	core	27.5-28.25	SW SW SW SW sec. 25, T. 93 N., R. 50 W.	Union
					30-35		
					35-40		
					40-45		
					45-50		
					50-55		
					55-60		
					60-65		
					65-70		
					composite[2]		
13	uncertain – "western derived"? sand	06-01-01	Alcester 6-1-2	auger cuttings	depth not recorded	SE SE NE NE sec. 29, T. 95 N., R. 49 W.	Union
14	uncertain – "western derived"? sand	06-01-01	Alcester 6-1-7	auger cuttings	depth not recorded	NE NW NE NE sec. 22, T. 95 N., R. 49 W.	Union
15	uncertain – "western derived"? sand	05-24-01	Newton Hills 5-24-2	outcrop	surface	NE SW NE SE sec. 12, T. 97 N., R. 49 W.	Lincoln

[1] See figure 1.

[2] Composite samples were not analyzed using XRD.

Table 2. Abbreviated mineralogical and chemical reaction results

Map location number	Field sample name and number	XRD sample name	Powdered sample reacts vigorously with HCl	XRD mineralogy	Counts per second of the raw data
1	Core R20-01-5 27-28 feet	C0164	no	Abundant: quartz Moderate: --[1] Minor: plagioclase Trace: dolomite Possible: potassium feldspar? Possible low-angle minerals: palygorskite?, illite/mica group?	4,961
1	Core R20-01-5 40-41 feet	C0165	no	Abundant: quartz Moderate: --[1] Minor: plagioclase Trace: potassium feldspar Possible: --[1] Possible low-angle minerals: illite/mica group, kaolinite group?	2,665
1	Core R20-01-5 58.5-59.5 feet	C0166	no	Abundant: quartz Moderate: amphibole? or illite/mica group? or feldspar with unusual intensity? Minor: probably feldspars, potassium feldspar Trace: --[1] Possible: --[1] Possible low-angle minerals: illite/mica group, gypsum?	1,346
1	Core R20-01-5 67-68 feet	C0167	no	Abundant: quartz Moderate: feldspar (albite) with unusual intensity? or illite/mica group? Minor: probably feldspars Trace: --[1] Possible: --[1] Possible low-angle minerals: illite/mica group, smectite group?	1,883
1	Core R20-01-5 75-76 feet	C0168	no	Abundant: quartz Moderate: --[1] Minor: plagioclase Trace: --[1] Possible: potassium feldspar, calcite?, dolomite? Possible low-angle minerals: illite/mica group, smectite group?	2,320

Table 2 – continued

Map location number	Field sample name and number	XRD sample name	Powdered sample reacts vigorously with HCl	XRD mineralogy	Counts per second of the raw data
1	Core R20-01-5 88-89 feet	C0159	yes	Abundant: quartz Moderate: --[1] Minor: plagioclase, calcite, potassium feldspar? Trace: --[1] Possible: --[1] Possible low-angle minerals: smectite group, illite/mica group	2,986
2	Outcrop 5-23-5 Gregory City	C0120	yes	Abundant: quartz Moderate: calcite, plagioclase Minor: --[1] Trace: --[1] Possible: potassium feldspar Possible low-angle minerals: illite/mica group?	1,968
2	Outcrop 5-23-6 Gregory City	C0124	no	Abundant: quartz Moderate: plagioclase Minor: potassium feldspar Trace: --[1] Possible: --[1] Possible low-angle minerals: illite/mica group, gypsum?	3,338
3	Outcrop 5-23-4 Ash Hollow	C0112	no	Abundant: quartz Moderate: --[1] Minor: feldspars Trace: --[1] Possible: calcite?, dolomite? Possible low-angle minerals: illite/mica group, gypsum?	2,138
4	Sand pit 5-23-3 Herrick gravel	C0114	no	Abundant: quartz Moderate: --[1] Minor: plagioclase, potassium feldspar? Trace: --[1] Possible: calcite? Possible low-angle minerals: illite/mica group, gypsum?	4,146
5	Quarry 5-10-1 Spencer Quarry	C0169	no	Abundant: quartz Moderate: --[1] Minor: --[1] Trace: --[1] Possible: --[1] Possible low-angle minerals: kaolinite group, illite/mica group?; since the Sioux Quartzite frequently contains pyrophyllite, this mineral was evaluated – there no hint of a peak (based on peak shape) at 9.6º 2-theta	14,118

Table 2 – continued

Map location number	Field sample name and number	XRD sample name	Powdered sample reacts vigorously with HCl	XRD mineralogy	Counts per second of the raw data
6	Sand pit 5-22-2 Bon Homme	C0113	no	Abundant: quartz Moderate: --[1] Minor: potassium feldspar Trace: plagioclase Possible: --[1] Possible low-angle minerals: illite/mica group, gypsum?	5,286
7	Core R20-01-1 105-110 feet Turkey Ridge	C0135	yes	Abundant: quartz Moderate: plagioclase, dolomite Minor: potassium feldspar Trace: calcite Possible: gypsum? Possible low-angle minerals: illite/mica group, kaolinite group	2,738
7	Core R20-01-1 117 feet Turkey Ridge	C0136	no	Abundant: quartz Moderate: --[1] Minor: plagioclase , potassium feldspar Trace: --[1] Possible: --[1] Possible low-angle minerals: illite/mica group, gypsum?, smectite group?	3,368
7	Core R20-01-1 127 feet Turkey Ridge	C0137	no	Abundant: quartz Moderate: plagioclase, potassium feldspar Minor: --[1] Trace: --[1] Possible: --[1] Possible low-angle minerals: illite/mica group, kaolinite group	6,168
7	Core R20-01-1 141-145 feet Turkey Ridge	C0138	no	Abundant: quartz Moderate: plagioclase, potassium feldspar Minor: --[1] Trace: --[1] Possible: --[1] Possible low-angle minerals: illite/mica group, kaolinite group, gypsum	7,200
7	Core R20-01-1 151 feet Turkey Ridge	C0139	no	Abundant: quartz Moderate: plagioclase, potassium feldspar Minor: --[1] Trace: --[1] Possible: dolomite?, calcite? Possible low-angle minerals: illite/mica group, gypsum	5,005

Table 2 – continued

Map location number	Field sample name and number	XRD sample name	Powdered sample reacts vigorously with HCl	XRD mineralogy	Counts per second of the raw data
7	Core R20-01-1 160-161 feet Turkey Ridge	C0140	no	Abundant: quartz Moderate: --[1] Minor: plagioclase Trace: potassium feldspar Possible: --[1] Possible low-angle minerals: illite/mica group	4,908
7	Core R20-01-1 173-174 feet Turkey Ridge	C0141	no	Abundant: quartz Moderate: --[1] Minor: --[1] Trace: plagioclase Possible: potassium feldspar? Possible low-angle minerals: illite/mica group, smectite group?	4,806
7	Core R20-01-1 180-185 feet Turkey Ridge	C0142	no	Abundant: quartz Moderate: --[1] Minor: plagioclase Trace: potassium feldspar Possible: gypsum, calcite? Possible low-angle minerals: illite/mica group	7,893
8	Mud rotary cuttings R20-01-2 40-50 feet Turkey Ridge	C0119	yes	Abundant: quartz Moderate: potassium feldspar, plagioclase Minor: gypsum Trace: calcite Possible: dolomite Possible low-angle minerals: kaolinite group?	3,275
9	Core R20-87-14 200-205 feet Turkey Ridge	C0130	yes	Abundant: quartz Moderate: plagioclase, potassium feldspar? Minor: --[1] Trace: calcite Possible: --[1] Possible low-angle minerals: smectite group, illite/mica group, gypsum?	3,180
9	Core R20-87-14 205-210 feet Turkey Ridge	C0131	yes	Abundant: not sure, probably plagioclase Moderate: quartz Minor: potassium feldspar? Trace: calcite Possible: --[1] Possible low-angle minerals: smectite group, illite/mica group	12,275

Table 2 – continued

Map location number	Field sample name and number	XRD sample name	Powdered sample reacts vigorously with HCl	XRD mineralogy	Counts per second of the raw data
9	Core R20-87-14 210-215 feet Turkey Ridge	C0132	no	Abundant: quartz Moderate: --[1] Minor: plagioclase, potassium feldspar Trace: --[1] Possible: --[1] Possible low-angle minerals: illite/mica group, gypsum?, smectite group?	2,760
9	Core R20-87-14 225-230 feet Turkey Ridge	C0133	no	Abundant: quartz Moderate: plagioclase, potassium feldspar Minor: --[1] Trace: --[1] Possible: --[1] Possible low-angle minerals: illite/mica group, kaolinite group, smectite group	1,681
9	Core R20-87-14 230-235 feet Turkey Ridge	C0134	no	Abundant: quartz Moderate: --[1] Minor: plagioclase, potassium feldspar? Trace: --[1] Possible: --[1] Possible low-angle minerals: illite/mica group, gypsum?, smectite group?	1,958
10	Small gravel pit 5-22-5 Hurley	C0117	yes	Abundant: quartz Moderate: --[1] Minor: dolomite, calcite Trace: potassium feldspar, plagioclase Possible: --[1] Possible low-angle minerals: smectite group, illite/mica group, kaolinite group	4,233
11	Outcrop 5-21-1 Missouri River	C0110	yes	Abundant: quartz Moderate: --[1] Minor: plagioclase Trace: calcite Possible: potassium feldspar? Possible low-angle minerals: illite/mica group, gypsum?, smectite group?	3,756
12	Core R20-88-30 Heeren core 27.5-28.25 feet	C0143	no	Abundant: quartz Moderate: --[1] Minor: plagioclase, potassium feldspar Trace: --[1] Possible: calcite? Possible low-angle minerals: illite/mica group, gypsum?	5,633

Table 2 – continued

Map location number	Field sample name and number	XRD sample name	Powdered sample reacts vigorously with HCl	XRD mineralogy	Counts per second of the raw data
12	Core R20-88-30 Heeren core 30-35 feet	C0144	no	Abundant: quartz Moderate: --[1] Minor: plagioclase Trace: potassium feldspar Possible: calcite? Possible low-angle minerals: mica/illite group	5,243
12	Core R20-88-30 Heeren core 35-40 feet	C0145	no	Abundant: quartz Moderate: --[1] Minor: plagioclase Trace: --[1] Possible: potassium feldspar? Possible low-angle minerals: illite/mica group, kaolinite group	5,470
12	Core R20-88-30 Heeren core 40-45 feet	C0146	no	Abundant: quartz Moderate: --[1] Minor: --[1] Trace: plagioclase, potassium feldspar Possible: calcite? Possible low-angle minerals: mica/illite group	7,470
12	Core R20-88-30 Heeren core 45-50 feet	C0147	no	Abundant: quartz Moderate: --[1] Minor: plagioclase Trace: potassium feldspar Possible: calcite? Possible low-angle minerals: illite/mica group, gypsum?	4,620
12	Core R20-88-30 Heeren core 50-55 feet	C0148	no	Abundant: quartz Moderate: --[1] Minor: potassium feldspar Trace: plagioclase Possible: calcite? Possible low-angle minerals: --[1]	5,570
12	Core R20-88-30 Heeren core 55-60 feet	C0149	no	Abundant: quartz Moderate: plagioclase Minor: potassium feldspar Trace: --[1] Possible: dolomite? Possible low-angle minerals: illite/mica group, gypsum?	5,948
12	Core R20-88-30 Heeren core 60-65 feet	C0150	no	Abundant: quartz Moderate: --[1] Minor: --[1] Trace: potassium feldspar, plagioclase Possible: --[1] Possible low-angle minerals:	10,468

Table 2 – continued

Map location number	Field sample name and number	XRD sample name	Powdered sample reacts vigorously with HCl	XRD mineralogy	Counts per second of the raw data
12	Core R20-88-30 Heeren core 65-70 feet	C0151	no	Abundant: quartz Moderate: --[1] Minor: plagioclase, potassium feldspar Trace: --[1] Possible: gypsum?, dolomite? Possible low-angle minerals: illite/mica group	9,755
13	Auger cuttings 6-1-2 Alcester	C016	yes	Abundant: quartz Moderate: --[1] Minor: plagioclase Trace: calcite Possible: potassium feldspar Possible low-angle minerals: illite/mica group, smectite group?	3,963
14	Auger cuttings 6-1-7 Alcester	C014	slight	Abundant: quartz Moderate: --[1] Minor: plagioclase Trace: --[1] Possible: calcite, potassium feldspar, dolomite? Possible low-angle minerals: illite/mica group, gypsum	4,265
15	Outcrop 5-24-2 Newton Hills	C0123	yes	Abundant: quartz Moderate: --[1] Minor: --[1] Trace: calcite, plagioclase, potassium feldspar Possible: dolomite? Possible low-angle minerals: illite/mica group?	4,053

[1] -- = not applicable.

Appendix

XRD Instrument Summary

adapted from: Geological and Planetary Sciences Division
California Institute of Technology
1200 East California Boulevard
Pasadena, California 91125

The South Dakota Geological Survey Scintag Pad V X-ray Powder Diffractometer is a theta:2-theta goniometer instrument. It is outfitted with a copper x-ray tube with two beam slits, a four-sample automated sample changer with sample spinning capability, and a low-noise liquid-nitrogen cooled germanium detector outfitted with two detector slits. The x-ray tube has a filter wheel allowing one to manually select a single filter. The wheel has filters made of vanadium, manganese, iron, nickel (which is the default filter that is used to remove copper $K\beta$ radiation), or zirconium. There is also an option for no filter.

The goniometer can be scanned in normal mode (i.e., theta and 2-theta are scanned in sync), or independent theta and 2-theta scans can be performed. The sample is mounted in a small, round magnetic disc for a diffraction run. The sample can be spun in the plane of the disc in order to increase the random orientation of powder grains in the mount. The instrument is outfitted with a set of beam and detector slits that balance the needs of resolution and intensity for typical runs.

The pulse-height analyzer that processes the detected x-ray pulses from the germanium detector is set for narrow discrimination of copper $k\alpha$ pulses. All other energy ranges are rejected, and in this way, any x-ray fluorescence emanating from the sample is rejected via pulse-height analysis.

The instrument is capable of very rapid scans for identification of powders that are simple in composition and abundant on the mount. Much slower scans are usually required for the analysis of complex mixed phases or high resolution work. Two scan modes are possible: a continuous scan mode that integrates counts over a small angular range for rapid scans, and a step-scan mode that is used for slower scans and any precision work

The Scintag PAD V X-ray Powder Diffractometer has the ability to perform very rapid phase identifications of powdered specimens in a fully automated mode. The XRD has a low noise germanium detector that greatly improves sensitivity. Data collection and manipulation is under control of a Gateway E4200 computer which contains a database of the ICDD powder diffraction files. Diffraction spectra are plotted and can be compared, in whole or in selected portions, to specified ICDD cards or automatically matched to the most similar spectra in the database.

The Scintag Pad V X-ray Powder Diffractometer is automated with a personal computer running Windows NT 4 operating system. The software package used to perform the diffraction work is the Scintag program Diffraction Management System for NT (DMSNT). The DMSNT program allows one to set up a sequence of automated tasks related to either collection of data or processing of the data already collected. This portion of the software package also allows one to perform search match procedures using the International Centre for Diffraction Data (ICDD) Joint Committee on Powder Diffraction System cards from an ICDD CD-ROM.