

**TESTING OF  
KITTSOON COUNTY BLOATING CLAYS AS  
LIGHTWEIGHT AGGREGATE IN  
STRUCTURAL CONCRETE**

by

Julie A. Oreskovich and Steven A. Hauck

August 1993

Technical Report  
NRRI/TR-93/17A

Funded by the Minerals Coordinating Committee Through the Minerals Diversification Plan

Natural Resources Research Institute  
University of Minnesota, Duluth  
5013 Miller Trunk Highway  
Duluth, Minnesota 55811

## ABSTRACT

Pleistocene glacial lake clays found in west-central Kittson County, Minnesota, were identified as bloating clays in 1989. One of the primary uses of bloating clays is as lightweight aggregate in structural concrete. Such lightweight structural concrete is used in the construction of high-rise buildings, where the reduced weight allows building higher while reducing the amount of structural steel support. While Minnesota has several different bloating clay deposits, most lightweight aggregate currently used in Minnesota is shipped by barge up the Mississippi River from West Memphis, Arkansas.

Among Minnesota's bloating clays are the Cretaceous shales near Springfield in southwestern Minnesota, the Ordovician Decorah Shale of southeastern Minnesota, and the Pleistocene Brenna Formation clays of Kittson County in northwestern Minnesota. The Kittson clays bloat at a lower temperature than the other Minnesota bloating clays and have a unique spherical bloating habit. In addition, the deposit is homogeneous over a large areal extent and thickness and lies within 20 ft. of the surface. The clays would be easily accessible for open pit mining.

This project was undertaken to determine whether the Kittson clays, when fired, produced a suitable lightweight aggregate for use in structural concrete. Testing was done according to American Society of Testing Materials (ASTM) standards by an independent testing laboratory. Tests results showed that the Kittson clays meet all ASTM requirements for use as a lightweight aggregate in structural concrete.

## TABLE OF CONTENTS

LIST OF FIGURES .....	iii
LIST OF APPENDICES .....	iv
INTRODUCTION .....	1
PURPOSE AND SCOPE .....	1
HISTORY .....	3
ACKNOWLEDGEMENTS .....	5
TESTING OF THE KITTSOON CLAYS AS LIGHTWEIGHT AGGREGATE IN STRUCTURAL CONCRETE .....	6
SELECTION OF INDEPENDENT TESTING LABORATORY .....	6
NRRI SAMPLE SELECTION AND PREPARATION .....	7
Sample Selection .....	7
Sample Preparation .....	7
CTL ROTARY KILN PROCESSING .....	8
AGGREGATE AND CONCRETE TEST PROCEDURES, DATA, AND RESULTS .....	9
Summary of Results .....	9
DISCUSSION .....	11
CONCLUSIONS .....	13
REFERENCES .....	14

**LIST OF FIGURES**

Figure 1. Location map of Kittson County bloating clays. . . . . 4

**LIST OF APPENDICES**

Appendix A. CTL Report on Preparation and Testing of the Kittson Clays . . . . . 15

Appendix B. CTL Test Data . . . . . 27

Appendix C. NRRI Composite Sample Data . . . . . 39

Appendix D. Composite Sample Crushing Characteristics . . . . . 45

## **INTRODUCTION**

Minnesota has several bloating clay deposits with economic potential. Among these are the Ordovician Decorah Shale of southeastern Minnesota and the Cretaceous shales near Springfield in southwestern Minnesota, which have been producers of lightweight aggregate in the past. A third bloating clay deposit, the Pleistocene glacial lake clays (Brenna Formation) of west-central Kittson County in northwestern Minnesota, has recently been identified (Fig. 1). The Kittson clays bloat at a lower temperature, expand in a spherical manner, are homogeneous and widespread, and occur near the surface for ease of mining. These clays, therefore, are a likely source for production of lightweight aggregate.

## **PURPOSE AND SCOPE**

The University of Minnesota-Duluth's Natural Resources Research Institute (NRRI) has been investigating properties of the Kittson County bloating clays (Hauck, et al., 1990; Hauck, 1991; and Hauck, in press). In keeping with NRRI's mission "to foster economic development of Minnesota's natural resources in an environmentally sound manner to promote private sector employment," (also a goal of the Minerals Coordinating Committee [MCC], the funding source for this project) potential end uses for fired Kittson County bloating clays have been delineated. One end use, investigated here, is the use of Kittson County bloating clays as lightweight aggregate in structural concrete.

To be considered for use as a lightweight aggregate, a material must meet several criteria as compiled by Prokopovich and Schwartz (1957).

Raw material requirements:

1.     bloats at low temperature (conserves fuel expense)
2.     bloats over a wide firing range (requires fewer controls)

3. has bulk density less than 50 lbs./ft<sup>3</sup>
4. bloated particles have sufficient strength (insures strength of the aggregate)
5. bloated particles are rounded (for good workability)
6. bloated particles have low moisture absorption (to prevent cement dehydration)
7. provides a well-graded mixture of coarse to fine particles (for concrete workability)
8. must be chemically inert (insures concrete strength)

Economic requirements:

1. location close to market (reduces transportation costs)
2. transportation facilities available (highways, railroads)
3. availability of low-cost fuel source
4. sufficient deposit size
5. sufficient deposit thickness
6. little overburden (not to exceed 35-40 ft.)
7. location of deposit above ground water level

Historically, lightweight aggregate came into its own in the United States during the 1950s, a period which saw states rushing to identify sources of the material. Materials such as expanded clays and shales and slag proved useful to the construction industry. Taller buildings require lighter materials. Lighter materials reduce the amount of structural steel required for support, an economic bonus.

Currently, most lightweight aggregate used in Minnesota is barged up the Mississippi River to Minneapolis from the Arkalite plant in West Memphis, Arkansas. The largest market for lightweight aggregate use in Minnesota is the Twin Cities area. The purpose of this study is to evaluate the Kittson clays according to ASTM standards to determine their suitability for use as

lightweight aggregate in structural concrete. Given favorable transportation rates, Minnesota would have an in-state source of lightweight aggregate as well as additional jobs in the private sector (see Zanko, in prep., for discussion of economics).

## **HISTORY**

The distinct bloating nature of the Kittson County clays was recognized by the Natural Resources Research Institute (NRRI) in 1989 during a Legislative Commission of Mineral Resources (LCMR) study of Minnesota clays (Hauck et al., 1990). Test briquets made from Kittson County clay samples collected by the United States Bureau of Mines (USBM; see Haas et al., 1987) in 1985 yielded a mass of connected balls when kiln-fired across a wide range of temperatures (Hauck, in press). Further evaluation of the bloating characteristics of these clays was undertaken with funding by Minnesota Technology, Inc. (MTI), formerly the Greater Minnesota Corporation (GMC), in 1990-1991 (Hauck, in press).

The identified Kittson County bloating clays occur in an area along the central part of the western edge of the county near the Red River of the North (Fig. 1). This area lies along the axis of the deepest part of Pleistocene Glacial Lake Agassiz in a narrow basin in which lake sediments accumulated to thicknesses exceeding 75 ft. In 1991, eight drill holes were put down west of the city of Hallock (DH1-DH8, Fig. 1; Hauck, in press). Seven of the holes were augered and one, DH1, was split-core augered. Kiln firings were done on 1-ft. intervals of the cored hole and on grab samples of 5-ft. intervals from each of the remaining four locations. Material from as shallow as 13 ft. in depth (lower Sherack Formation) to 75 ft. in depth (Brenna Formation) expanded when fired to temperatures averaging 2,100EF. Detailed firing results as well as geological and geochemical information on the Kittson County bloating clays are found in Hauck (in press).

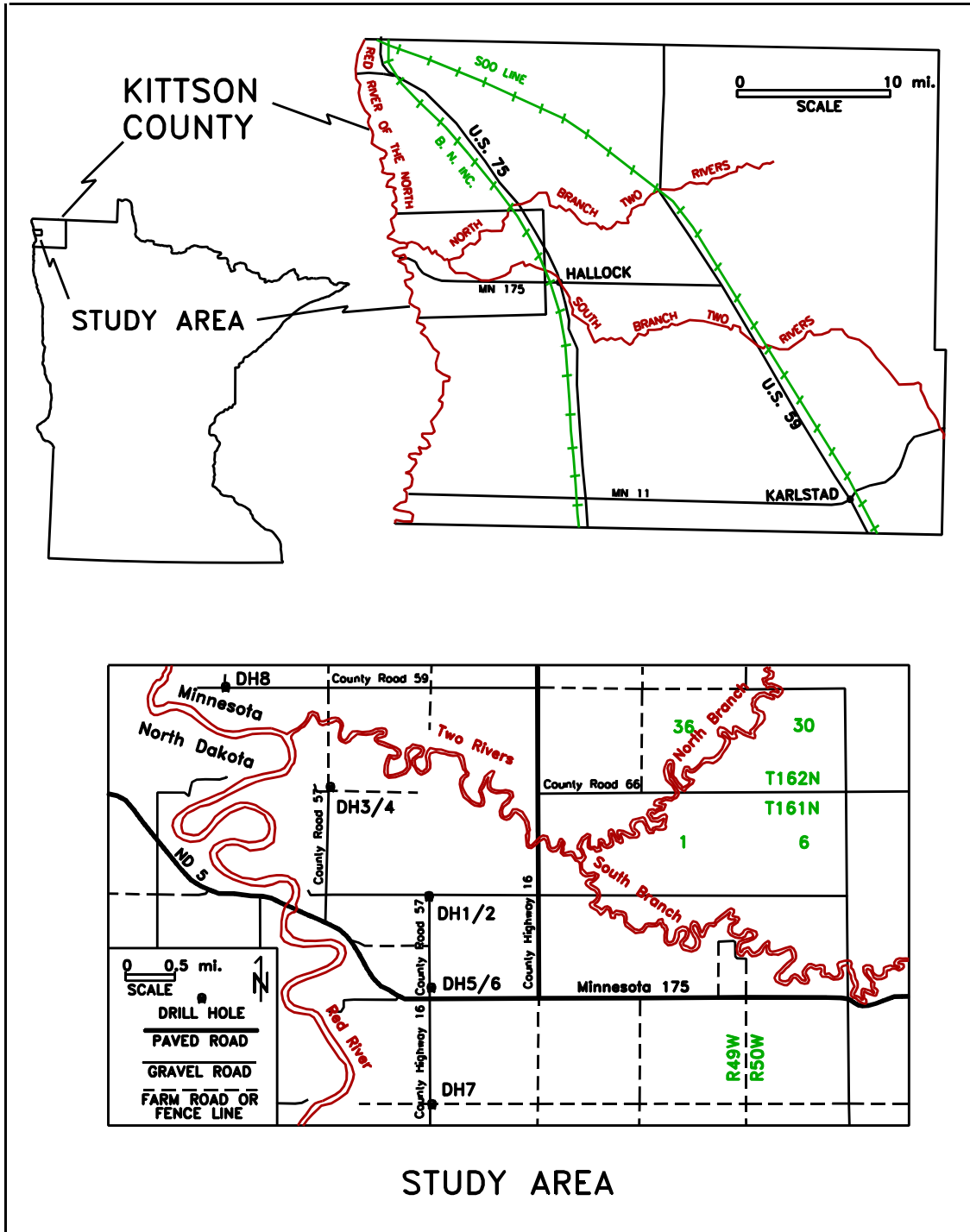


Figure 1. Location map of Kittson County bloating clays.

## **ACKNOWLEDGEMENTS**

Funds for this project were provided by a grant from the Minerals Coordinating Committee through the Minerals Diversification Plan. Testing was done by Construction Technology Laboratories, Inc. (CTL) of Skokie, Illinois. We thank Mr. Alexander Mishulovich, Principal Process Engineer for Chemical/Physical Research, and the CTL laboratory personnel for their assistance with this project.

## **TESTING OF THE KITTSON CLAYS AS LIGHTWEIGHT AGGREGATE IN STRUCTURAL CONCRETE**

### **SELECTION OF INDEPENDENT TESTING LABORATORY**

The services of an independent testing laboratory were required in order to rotary kiln process the Kittson County clays and test them in structural concrete. Construction Technology Laboratories, Inc. (CTL) of Skokie, Illinois, was selected because of their close ties to the Portland Cement Institute. The contract was written such that CTL would conduct rotary kiln testing of the Kittson County clays to determine optimum bloating temperature and residence time in the kiln and to produce sufficient aggregate to conduct the following ASTM C 330-87 standard tests for lightweight aggregate in structural concrete:

1. ASTM C 641 - staining materials in lightweight aggregate
2. ASTM C 114 - loss on ignition
3. ASTM C 127 - specific gravity and absorption
4. ASTM C 136 - sieve analysis of aggregate
5. ASTM C 142 - clay lumps and friable particles
6. ASTM C 29 - dry rodded unit weight and voids
7. ASTM C 192 - concrete mixes to cast specimens
8. ASTM C 192 - concrete trial mixes
9. ASTM C 39 - compressive strength of concrete specimens
10. ASTM C 496 - splitting tensile strength
11. ASTM C 567 - unit weight of lightweight concrete
12. ASTM C 157 - drying shrinkage of hardened concrete
13. ASTM C 151 - popout test by autoclave

14. ASTM C 666 - resistance of concrete to rapid freezing and thawing

A final report written by CTL includes sample preparation procedures and other relevant data as well as results from the testing laboratory on the rotary kiln and concrete tests. This report is included as Appendix A. Actual CTL test data is presented in Appendix B.

### **NRRI SAMPLE SELECTION AND PREPARATION**

Preparation of a representative sample of Kittson County bloating clays for shipment to CTL laboratories involved drill hole and interval selection, followed by air drying, crushing, screening, and blending the selected material.

#### **Sample Selection**

On average, the interval from 20 ft. to 40 ft. in depth provided the greatest expansion as well as a highly developed sphericity (Hauck, in press). Drill holes DH1, DH2, DH5, DH6, and DH7, spanning a linear distance of 2 miles (Fig.1), were chosen as the source material for the composite sample to be tested. Grab samples from DH3 and DH4, located - 1.4 miles northwest of DH1 and DH2, bloated rather poorly in comparison to the above listed drill holes. DH8, however, located - 2.8 miles northwest of the holes listed above and - 1.4 miles northwest of DH3 and DH4, displayed excellent bloating. Material from DH8 was not included in the composite sample because the poorer bloating material from DH3/DH4 broke the areal continuity of highly expanding clays.

#### **Sample Preparation**

Material from the selected holes and intervals was removed from the sample bags in which it had been stored and set out in pans to be air-dried. The individual intervals were combined into one large composite sample weighing just over 1,200 lbs. A breakdown of the composite sample

by drill hole, interval, and pounds of material is given in Appendix C. Drying generally took a period of three days, with the clays being overturned once or twice daily. When sufficiently dried, the clays were broken up by hand and/or hammer. Crushing characteristics, including general particle shape and size, were noted (Appendix D). The clays were screened to -3/4 in. size, as required by CTL. Oversize material was further broken down by hammer until passing the 3/4" screen. CTL did not require screening out the fines.

Blending of the composite sample was achieved by coning and quartering the material a minimum of five times. Three 55-gallon drums were then filled and shipped by truck to CTL for processing. Total weight of sample shipped was 1,210.7 lbs. A portion of the composite sample was retained by the NRRI laboratory for testing and control purposes.

### **CTL ROTARY KILN PROCESSING**

Expansion of the clays was accomplished at CTL by means of firing in a pilot rotary kiln 15 ft. long by 20 in. in diameter (12 in. inside brick diameter). The kiln's slope was set at 6.7%, with a rotation speed of 2.4 rpm. The resultant residence time for material in the kiln was 20-30 minutes.

Temperature in the burning zone of the kiln was held in the range of 1,950-2,040EF. Such temperatures proved to be too hot, in particular for the finer grain sizes, and led to melting and fusing of the material, which plugged up the kiln. This was indicative of a very short firing range and a need for tight temperature control. CTL defined the firing range as the temperature differential between initial softening and almost complete melting of the fired material (Appendix A).

Repeating the firing process at slightly reduced temperature and retention time yielded a satisfactory product. Twenty minutes at 1,900EF is reported by CTL to be the optimum residence time and bloating temperature for the Kittson County clay material tested. Fifteen cubic feet or 510 lbs. of usable material was produced by CTL (Appendix A).

## **AGGREGATE AND CONCRETE TEST PROCEDURES, DATA, AND RESULTS**

The aggregate and concrete test methods, data, and results can be found in CTL's report, "Preparation and Testing of Expanded Shale," by Alex Mishulovich (Appendix A). Two trial concrete mixes were initially produced to test compressive strength. Different proportions of coarse/fine aggregate and water/cement were used, with the following results\*:

Coarse/Sand Ratio	Unit Weight pcf	Compressive Strength, psi	
		7 days	28 days
60/40	100.5	2,810	3,500
50/50	105.9	2,820	3,520

\* A. Mishulovich, CTL, correspondence

Final testing proceeded with the second mixture: 50% lightweight coarse aggregate and 50% sand.

### **Summary of Results**

CTL test data is presented in Appendix B. The following listing is a summary of test results as taken from Mishulovich's report (Appendix A):

#### ASTM TEST

#### RESULTS

1. ASTM C 641 -  
staining materials in lightweight aggregate: None observed

2. ASTM C 114 -  
loss on ignition: 0.88 (5% - max allowed)

3. ASTM C 127 -  
absorption:

at 15 minutes: 8.3%  
at 30 minutes: 8.7%  
at 45 minutes: 9.1%  
at 60 minutes: 9.3%  
at 2 hours: 10.1%  
at 4 hours: 11.1%

specific gravity factor (ACI 211.2-81): 1.1

4. ASTM C 136 -  
sieve analysis of aggregate:

Sieve #	% Ret. Ind. Sieve	Cum. % Ret. Ind. Sieve	Cum. % Pass Ind. Sieve
3/4	15.0%	15.0%	85.0%
1/2	20.4%	35.4%	65.0%
3/8	54.7%	90.1%	10.0%
4	9.9%	100.0%	--

5. ASTM C 142 -  
clay lumps and friable particles: 0.2% (2% - max allowed)

6. ASTM C 29 -  
dry rodded unit weight and voids: 33.2 lbs/ft<sup>3</sup> (55 lbs/ft<sup>3</sup> - max allowed)

7. ASTM C 192 -  
concrete mix to cast specimens: (see Appendix A, Table 2)

8. ASTM C 192  
concrete trial mixes (see chart, p. 9)

9. ASTM C 39 -  
compressive strength of concrete specimens:  
(moist cured for 7 days, air dried for 21 days): 4,590 psi (avg. of 3 specimens)

(moist cured for 28 days): 4,110 psi (avg. of 3 specimens)

- |     |  |                               |
|-----|--|-------------------------------|
| 10. | ASTM C 496 -<br>splitting tensile strength:<br>(moist cured for 7 days, air dried for 21 days):          | 350 psi (avg. of 8 specimens) |
| 11. | ASTM C 567 -<br>unit weight of lightweight concrete:<br>(moist cured for 7 days, air dried for 21 days): | 107.1 lbs/ft <sup>3</sup>     |
| 12. | ASTM C 157 -<br>drying shrinkage of hardened concrete:<br>(32% relative humidity and 100EF for 28 days): | -0.02% (avg. of 3 specimens)  |
| 13. | ASTM C 151 -<br>popout test by autoclave:  | no surface popouts observed   |
| 14. | ASTM C 666 -<br>resistance of concrete to rapid freezing and<br>thawing:                                 | (see p. 26, Appendix A)       |

## **DISCUSSION**

The optimum residence time and bloating temperature for the Kittson clays as determined by CTL is 20 minutes at 1,900EF. The Kittson clays, as rotary kiln-processed by CTL, meet all ASTM standards for lightweight aggregate in structural concrete. The unit weight of 33.2 lbs/ft<sup>3</sup> is well below the maximum of 55 lbs/ft<sup>3</sup>. Compressive strength of 4,590 psi exceeds the required 4,000 psi.

The optimum firing temperature of the Kittson clays processed in the rotary kiln differ from those of the Kittson clays processed in an electric muffle oven. An explanation for the difference is that in order to increase strength, CTL chose not to fully expand the clays. Strength decreases as size increases. The end product will be the determinant factor as to degree of expansion required.

The "very short firing range" of the Kittson clays, as evidenced by the problems encountered with fusing of the material in the rotary kiln, can be overcome to some degree by screening out the fines. As the fines melt more quickly, they tend to act as binder for the remaining particles. To

maximize throughput and reduce the effect of temperature fluctuations in the kiln, the Kittson clay chips could be screened for size prior to firing. Firing runs could then be made on each size fraction, with firing temperature and retention time adjusted accordingly.

## CONCLUSIONS

The Kittson clays have been shown by CTL to be suitable for use as a lightweight aggregate in structural concrete when processed in a rotary kiln. The sample submitted for testing (CTL Blend), a composite sample covering an areal extent of 2 linear miles, is indicative of the vast bloating clay resource located in west-central Kittson County. The same 20 ft.-40 ft. interval used in the composite sample is known to bloat as well, if not better, in an area 2 miles northwest of the northern limit of the composite sample.

Use of the Kittson clay as lightweight aggregate in structural concrete will be determined by market proximity and transportation costs. The fact that the Kittson clays meet the ASTM standards opens up the possibility for its use in other lightweight concrete products. Further testing needs to be done to determine if the Kittson clays are suitable for lightweight aggregate in insulating concrete. Based on the amount of air space contained in the numerous sealed chambers within each expanded clay chip, there is a strong probability that it may have value to the insulating concrete industry. Because the Kittson clays are located at a distance from larger cities that make use of lightweight aggregate in structural concrete in high-rise buildings, a more likely market niche for the Kittson clays would be in insulating concrete.

## REFERENCES

- Haas, L. A., Aldinger, J. A., Blake, R. L., and Swan, S. A. 1987, Sampling, characterization, and evaluation of midwest clays for iron ore pellet bonding: U.S. Bur. Mines, Rept. Invest. 9116, 44 p.
- Hauck, S., Heine, J., Zanko, L., Power, B., Geerts, S., Oreskovich, J., Reichhoff, J., 1990, LCMR clay project: NRRI summary report: Natural Resources Research Institute, Univ. Minn., Duluth, Tech. Rept., NRRI/GMIN-TR-89-12A, 201 p.
- Hauck, S. A., ed., in press, Ceramic tile and lightweight aggregate product development using Minnesota clays: Natural Resources Research Institute, Univ. Minn., Duluth, Tech. Rept., NRRI/TR-91/23.
- Prokopovich, N., and Schwartz, G. M., 1957, Preliminary survey of bloating clays and shales in Minnesota: Minn. Geol. Survey, Sum. Rept. No. 10, 68 p.
- Zanko, L. M., in prep., The economic impact of transportation costs on the potential utilization of Kittson County bloating clays and Minnesota River Valley kaolins: Natural Resources Research Institute, Univ. Minn., Duluth, Tech. Rept.

**APPENDIX A**

CTL REPORT ON PREPARATION AND TESTING  
OF THE KITTSON CLAYS























**APPENDIX B**

**CTL TEST DATA**























**APPENDIX C**

**NRRI COMPOSITE SAMPLE DATA**



Hole #	Footage (ft.)	Initial Wt. (1-19-91) (lbs.)	Current Bag Wt. (lbs.)	% Wt. Loss	Crushed Wt. (lbs.)	Total % Wt. Loss	Add'l % Wt. Loss	Total Lbs. Per Hole	Retained Sample (lbs.)	Total Retained Lbs./Hole
	30-35	65	49.0	24.6%	47.4	27.1%	2.5%		4.3	
	30-35	54	39.0	27.8%	38.6	28.5%	0.7%		4.4	
	35-40	44	32.0	27.3%	31.4	28.6%	1.4%		4.0	
	35-40	71	51.0	28.2%	49.4	30.4%	2.3%		4.7	
								322.4		34.7
DH6	20-25	55	40.2	26.9%	39.6	28.0%	1.1%		4.1	
	20-25	66	47.8	27.6%	47.2	28.5%	0.9%		4.7	
	25-30	60	45.0	25.0%	44.4	26.0%	1.0%		3.8	
	25-30	65	47.2	27.4%	46.8	28.0%	0.6%		4.1	
	30-35	54	38.8	28.1%	38.4	28.9%	0.7%		3.8	
	30-35	57	42.8	24.9%	42.2	26.0%	1.1%		3.7	
	30-35	59	43.4	26.4%	42.6	27.8%	1.4%		4.2	
	30-35	37	26.4	28.6%	26.0	29.7%	1.1%		4.0	
	35-40	67	48.4	27.8%	47.8	28.7%	0.9%		3.9	
								375.0		36.3
DH7	20-25	71	54.4	23.4%	53.8	24.2%	0.8%		4.6	
	20-25	67	49.2	26.6%	49.0	26.9%	0.3%		4.4	
	25-30	49	37.8	22.9%	37.4	23.7%	0.8%		4.1	
	25-30	64	45.4	29.1%	45.0	29.7%	0.6%		4.0	
	30-35	57	43.6	23.5%	43.0	24.6%	1.1%		4.6	

Hole #	Footage (ft.)	Initial Wt. (1-19-91) (lbs.)	Current Bag Wt. (lbs.)	% Wt. Loss	Crushed Wt. (lbs.)	Total % Wt. Loss	Add'l % Wt. Loss	Total Lbs. Per Hole	Retained Sample (lbs.)	Total Retained Lbs./Hole
	30-35	74	52.0	29.7%	51.4	30.5%	0.8%		4.3	
	35-40	72	50.2	30.3%	49.6	31.1%	0.8%		4.3	
								329.2		30.3
						Total Lbs. All Holes:		1,514.0		152.7

BREAKDOWN BY DDH										
Hole #	20-25 ft. (lbs.)	% By Hole	25-30 ft. (lbs.)	% By Hole	30-35 ft. (lbs.)	% By Hole	35-40 ft. (lbs.)	% By Hole	Total (lbs.)	Total (%)
DH1	36.2	10.8%	47.6	13.8%	49.6	9.2%	27.2	9.2%	160.6	10.6%
DH2	40.0	11.9%	39.0	11.3%	157.4	29.3%	90.4	30.6%	326.8	21.6%
DH5	70.8	21.0%	84.8	24.6%	86.0	16.0%	80.8	27.3%	322.4	21.3%
DH6	86.8	25.8%	91.2	26.4%	149.2	27.8%	47.8	16.2%	375.0	24.8%
DH7	102.8	30.5%	82.4	23.9%	94.4	17.6%	49.6	16.8%	329.2	21.7%
Total	336.6	100.0%	345.0	100.0%	536.6	100.0%	295.8	100.0%	1,514.0	100.0%
BREAKDOWN BY DRILL SITE										
Site #	20-25 ft. (lbs.)	% By Site	25-30 ft. (lbs.)	% By Site	30-35 ft. (lbs.)	% By Site	35-40 ft. (lbs.)	% By Site	Total (lbs.)	Total (%)
Site 1 (DH1,DH2)	76.2	22.6%	86.6	25.1%	207.0	38.6%	117.6	39.8%	487.4	32.2%
Site 3 (DH5,DH6)	157.6	46.8%	176.0	51.0%	235.2	43.8%	128.6	43.5%	697.4	46.1%
Site 4 (DH7)	102.8	30.5%	82.4	23.9%	94.4	17.6%	49.6	16.8%	329.2	21.7%
Total	336.6	100.0%	345.0	100.0%	536.6	100.0%	295.8	100.0%	1,514.0	100.0%

CTL SHIPMENT			
Drum #	Sample Weight (lbs.)	Drum Weight (lbs.)	Shipping Weight (lbs.)
BBL-1	433.6	49.0	482.6
BBL-2	432.5	46.0	478.5
BBL-3	344.6	44.8	389.4
Total	1210.7	139.8	1350.5

**APPENDIX D**

**COMPOSITE SAMPLE CRUSHING CHARACTERISTICS**

## 1991 MATERIAL SENT TO CTL

### **DH1 19'-29' (50# bag weight) 1 of 2 bags**

Chunks to chips; brownish gray in color; easily broken to hard where compacted against bottom and sides of bag; plenty of fines; some very small to small carbonate dropstones.

### **19'-29' (65#) 2 of 2 bags**

Graded from chunks to chips to curved flakes to fines; black organic chunks present; hard to break; small carbonate dropstones; sample had been sealed in plastic bag, was not exposed for drying; sample still slightly damp when broken up.

### **29'-34' (69#)**

Hard chunks to chips; non-flaking; brownish gray in color; small carbonate dropstones.

### **34'-39' (40#)**

Chunks to chips to fines; somewhat flaky in middle part of bag; brownish gray in color; hard to break; small to medium carbonate dropstones in top part of bag grading to none in bottom part; sample had been enclosed in plastic bag, not exposed for drying.

### **DH2 20'-25' (57#)**

Chunks; few fines; hard to break.

### **25'-30' (64#)**

Gray flakes; some brownish chunks; easy to break.

### **30'-35' (52#)**

Large flakes, came off the auger in relatively thin curls; brownish gray in color; minimal fines; very easy to break; hard where compacted against bag-produced powder when broken.

### **30'-35' (50#)**

Large flakes to chips; light gray to brown in color; some darker gray crumbly-looking pieces; top half of bag hard to break-produced many fines; lower half of bag easy to break-produced fewer fines.

### **30'-35' (69#)**

Large flakes; light gray to brown in color; easy to break; minimal fines when originally broken up; plenty of fines after handling.

### **30'-35' (65#)**

Flaky; easy to break; many fines and powder in top half of bag.

### **35'-40' (61#)**

Curved flakes, decreasing in size in bottom half of bag along with an increase in fines; easy to break; brownish gray in color with some light brown.

### **35'-40' (77#)**

Curved flakes; easy to break except where compacted against bag-breaks into chunks and powder, found mainly in middle portion of bag; plenty of fines in bottom portion of bag; a few fairly large (1.5" x 1.0"-) calcareous pieces found in middle to bottom of bag, full of solution cavities and covered with milky-white glaze in places.

**DH5 20'-25' (62#)**

Brownish to grayish flakes; easily broken; some hard compacted material in the lower half of the bag, brown with hard black inclusion-crumbles rather than flakes; increased fines in lower half of bag; some carbonate dropstones.

**20'-25' (39#)**

Similar to previous sample; plenty of fines.

**25'-30' (54#)**

Some hard, compacted clumps in the top half of the bag; lower half easier to break.

**25'-30' (64#)**

Hard brownish clumps in the top half of bag; difficult to break up; lower half easier to break up; harder pieces were gray in color.

**30'-35' (65#)**

Non-flaking; relatively hard to break; crumbling, with plenty of fines and powder, in the top half of the bag; crumbly-hackly-in the lower half, much already broken up; some very hard gray clumps; some dropstones.

**30'-35' (54#)**

Flakes to chips in top half of bag; easily broken; lower half of bag already broken up in bag; remaining clumps very hard-break into smaller hard chunks and powder; some dropstones in the lower half.

**35'-40' (44#)**

Flakes; fairly easily broken; dropstones up to 1/2"+.

**35'-40' (71#)**

Flakes, easily broken, with some hard compacted clumps and small dropstones in the top half of the bag; somewhat flaking in lower half, with most easily broken; a few hard clumps; some small dropstones.

**DH6 20'-25' (55#)**

Large, medium-gray curved flakes, easily broken, with harder, brownish chunks and plenty of fines in the top half of the bag; easily breaking flakes with some interlayered brown muds containing black organic material and carbonate dropstones in the lower half; muds crumbly, hard where compacted.

**20'-25' (66#)**

Large, brownish gray chunks and chips, easily broken-crumbly-with plenty of fines in the top half; black organic material present; brownish gray to gray chips with flakes in the lower half; easy to break; fewer fines.

**25'-30' (65#)**

Large brownish to light gray curved flakes throughout; easily broken except where compacted; plenty of fines-some medium to dark gray in color.

**25'-30' (60#)**

Rounded, unconsolidated, friable clumps in the top half of the bag with some flakes; easily broken-disintegrating; plenty of fines; large medium to dark gray curved flakes and chips in the lower half of the bag, with some friable clumps; plenty of fines.

**30'-35' (59#)**

Chips; easily broken-crumbly rather than flaking in the top half of the bag; large flakes to chips, medium gray to brownish, easily broken but less crumbly in the lower half.

**30'-35' (57#)**

Round, unconsolidated, easily broken clumps containing plenty of small (1/8"-1/4") carbonate dropstones in the top half of the bag; some flakes; plenty of fines; distinct change in the lower half of the bag: easily broken, large, medium gray to brownish flakes from curved strands off the auger with only a few rounded clumps; some carbonate dropstones present, larger than those of the top half (1/4"-1/2"), as well as white to light carbonate inclusions.

**30'-35' (54#)**

Flakes, easily broken, in top half of bag; some dropstones, larger than previous ones (1/2"+); larger flakes in the lower half, easily broken; few, if any, dropstones in the lower half; some white to light carbonate inclusions; flakes and fines are medium to dark gray in color.

**30'-35' (37#)**

Large, medium gray flakes; easily broken; plenty of fines.

**35'-40' (67#)**

Large flakes, light gray to brownish in color; some chips; easily broken; plenty of fines; a few hard clumps of compacted material in the lower half.

**DH7 20'-25' (71#)**

Brown chunks with some gray to brown flakes in the top half of the bag; easily broken-clumps chip or powder on breaking; many black organic inclusions; crystalline needles catching light; plenty of fines; some small to large carbonate dropstones; brownish gray flaking clumps and brown chunks containing many organic inclusions in the lower half of the bag; easily broken; plenty of fines; some dropstones.

**20'-25' (67#)**

Chips to large flakes in top half of bag; breaks easily; fair amount of fines; mixture of brown muds and gray flaking shale layers in lower half; muds are hard-they chip and powder; flakes easily broken; brown clumps are easily broken-fairly unconsolidated; one large (1" x 1/4") carbonate dropstone in the gray flaking material.

**25'-30' (49#)**

Disintegrating clumps of brown mud throughout; darker, non-flaking chips; small (1/8") to large (1" x 3/4") carbonate dropstones; plenty of fines.

**25'-30' (64#)**

Curved flakes to chips; easily broken; hard where compacted; few fines.

**30'-35' (57#)**

Disintegrating clumps-chips to powder-with gray flakes, more flakes found in the top half of the bag; plenty of fines; small to medium carbonate dropstones throughout, larger in the top half; crystalline needles and black organic inclusions in the lower half of the bag.

**30'-35' (74#)**

Flakes to chips; easily broken; small to medium carbonate dropstones in top half of bag, two large well-rounded ones also found; non-flaking chips in lower half; fairly easy to break; small to medium carbonate dropstones.

**35'-40' (72#)**

Chips and flakes in top half of bag; very crumbly; plenty of fines; carbonate dropstones; very hard non-flaking chips in lower half of bag; carbonate dropstones.