EXAMINATION OF CLAY SAMPLES SUPPLIED BY DIVISION OF MINERAL RESOURCES

January 1970 Progress Report

Lab. No. 3518; A, B, C, D, & E - Book No. 261

by
Edwin H. Bentzen III

Introduction

In February 1969, five samples of white residual clay from the volcanic slate belt in North Carolina, were collected by the State Division of Mineral Resources, Raleigh. Samples from these locations had previously been submitted to the U. S. Bureau of Mines for testing. The Bureau gave potential uses for each sample; however, no uses were named which could be the basis for a large industry. (See Tables 1 through 10 for collection and testing data.) Later this year, portions of the resampled material were submitted to the Minerals Research Laboratory for preliminary beneficiation studies. The object of this work was to produce, by minimal processing, a product on which a larger scale industry might be based. The type products under consideration included ball clay, kaolinite clay, brick clay, sericite, and ceramic products.

Although three of the five materials sampled had been used as additives to brick clay, only one is in use as such today. Also, in each case they were minor constituents of a red brick, and not major constituents of a white brick.

Sample Identification

The five samples submitted were assigned Lab No. 3518, A through E. Cross identification with D.M.R. sample numbers is shown below:

Lab No.	D.M.R. Sample No.	County	Location
3518-A	N.C. 43H-9	Harnett	Angier
3518-B	N.C. 63M-14	Moore	Hancock clay pit
3518-C	N.C. 63M-16	Moore	Williams clay pit
3518-D	N.C. 63M-21	Moore	Glendon
3518-E	N.C. 76R-1	Randolph	Jay Williams site

Also see Tables 1, 3, 5, 7, and 9 for additional information.

Procedure

The samples were crushed through a roll crusher, set at ½ inch, mixed and sampled for chemical analysis. The complete chemical analyses are shown in Table 11. On the basis of the chemical analyses, and equations derived by Koenig* and Coffeen,** the following mineralogic compositions were projected:

Minerals	Sample No. A	B	C	D	E
Albite	10.3	1.0	0.9	4.1	18.6
Microcline	-	6.6	13.7	11.5	8.3
Anorthite	-	0.3	1.0	11.9	4.0
Muscovite	28.8	15.4	2.0	-	-
Quartz	33.7	57.7	40.3	51.3	41.3
Kaolinite	27.1	17.2	38.7	17.3	23.3
Total	99.9	98.2	96.6	96.1	95.5

*E.W. Koenig, "Calculation of Mineralogical Composition of Feldspar by Chemical Analysis," Journal of American Ceramic Society, 25 (14), 420.

**William W. Coffeen, "Simple Procedure for Correction of Mineral Compositions Calculated from Feldspar Analysis," Ceramic Age , 60 (6),29, Dec. 1952.

The exact method used to determine the rational analysis is given in Table 12.

The test procedure used to determine "grit" is an adaptation of one explained in J. M. Huber Corporation publication, Kaolin Clays and Their Industrial Uses. In place of a Hamilton-Beach mixer No. 18, a Wemco Mineral Master with a small propeller and a 500-gram capacity octagonal pot were used. The speed was adjusted to 2,000 R.P.M.

Tetrasodium pyrophosphate solution was made by dissolving 20 grams of anhydrous tetrasodium pyrophosphate ($Na_4 P_2O_7$) in 1,000 cubic centimeters of distilled water. One hundred grams of clay, 375 cubic centimeters of water and 25 cubic centimeters of tetrasodium pyrophosphate solution were placed in the pot and mixed at high speed for ten minutes. The mixture was poured slowly into an eight-inch diameter, 325 mesh screen which had previously been wetted with water. The pot was washed throughly, and the washings were poured onto the screen. Generally, the chemical treatment indicated above was sufficient to produce free screening. However, in cases where the screen became clogged because of a high-viscosity clay or excessive residue, the screening was facilitated by directing a stream of water against the screen from a rubber hose. The force of the water can be regulated by squeezing the end of the rubber hose. The clay was washed through the screen with a stream of water until the wash water was clear and free from clay particles. This operation can be completed in one minute or less.

The residue on the screen was washed into a small pan and allowed to stand for two to three minutes. The clean water was decanted by using a glass stirring rod pressed against the lip of the pan. The residue

was dried in the pan at 105°C, cooled and weighed.

The minus 325 mesh slurry was collected in a 2½-gallon bucket which was filled to a depth of ten inches. The slurry was well mixed to place all solids in suspension, and was allowed to settle. After a specified amount of time, the suspended material was poured off. The sinks were collected, dried at 105°C, cooled and weighed. The suspended solids were either further settled for a specified time, or flocculated. Assay samples were collected and analysed. Figure No. 1 shows the weight percent settled out of the suspension as a cumulative percent (including the plus 325 mesh grit), versus time in minutes.

Using the information in Figure 1, in connection with other data, such as percent oversize, percent clay, percent mica, and percent wasted slimes (unsettled after 24 hours), a settling time was chosen for each sample. It was chosen in such a manner as to remove the maximum amount of sand and silt, and to segregate the maximum amount of kaolinite. Trial runs were undertaken using these settling times. The results of these runs can be seen in Table 13.

It had been agreed upon, with Mr. Conrad, that one pound of clay product would be produced for U.S.B.M. testing. From the test data, it was seen that 2,000 grams of head feed would have to be treated in order to produce the necessary weight of product.

Only the three higher-grade samples were treated to produce clay product. Each was treated as before. One hundred-gram samples were mixed with a pyrophosphate solution, screened, and settled to remove grit and silt. The slimes and clay were poured into a cut-off 55-gallon drum to a depth of ten inches. After mixing they were allowed to stand for 24 hours. The slimes were then siphoned off and discarded. The

clay product was dried at 105°C, cooled and weighed. The dried clay was screened on a 200 mesh screen, sampled for assay, and checked for color reflectance and bulk weight. The results can be seen in Table 14.

Results

The clay content of the head feed, as determined by a rational analysis, was rather disappointing, especially when compared to some deposits in South Carolina and Georgia. Some of the deposits worked for ball clay or kaolin contain 85 to 95 percent kaolinite. These deposits lend themselves to simple beneficiation with high yield. With the minimal processing used here, aimed at the best balance between maximum weight recovery and high grade clay, the yield was low. More rigorous processing could probably produce a better yield, however this would prove costly in production.

The color readings on the products were not as high as those necessary for paper or paint filler markets. The bulk weight, however, was in the right range.

It is hoped that more light can be shed on market possibilities when the results of U.S.B.M. test work is received.

Table 1

NORTH CAROLINA CLAY AND SHALE INVESTIGATIONS #3518-A

Sample No: N.C. 43H-9 County: Harnett

Locality:

Angier Site - In N.E. edge of town of Angier, on N. side of S.R. 1502, 0.3 mile N.E. of intersection with S.R. 1500.

Geology:

Formation:

Felsic Volicanics

Type of Material:

Clay X Shale

Other

Description of Outcrop or Exposure:

Ditch and bank exposing 2 foot high face on N. side of road. Extends beneath thin veneer of sandy Coastal Plain soil.

Attitude of Bedding:

N.E. trending

Sampled Interval:

Every foot over three foot section along middle elevation of bank, near center.

Type of Sample:

Channel Composite X Grab

Other:

Present Use:

None

Remarks: Sampled by E.P.A. 11/19/68; E.P.A. + W.F.W. 2/13/69

White clay is reported to have been encountered in ditching for waterline for approx. 0.5 mi. ~~form~~ from point of sample N.E. along S.R. 1502 (extends beyond town limits about 0.3 mi.) 1st. reported by: Fred D. Boege, 501 Williamson Street, Whiteville, North Carolina.

RESEARCH ENGINEERING ANALYTICAL

Table 2

REPORT OF CLAY TESTS

#3518-A

Laboratory Number 72258-A Sender's Identification N. C. 43H-9

Name U. S. Bureau of Mines Address Knoxville, Tennessee

Laboratory Job No. 72258 Sender's Purchase Order No.

Type Material Clay Color: Dry WH-TAN Wet Y-WH Grind: Mesh 20

Water of Plasticity % 28 Plasticity Poor Workability Very short

% Drying Shrinkage Plastic Basis 2.9 Dry Strength Poor Drying Properties Good

Slow Firing Test:

Heating Rate: 525 °F./hr. to 1800 °F., then 100 °F./hr. L.O.I. 7.3 % pH 5.1

Temp °F	Color	Moh's Hardness	Total Shrinkage: % of Plastic Length	Firing Shrinkage: % of Plastic Length	Absorption %	Apparent Sp. Gr.
1800	YR 5/4	1	3.5	0.6	40.6	1.26
1900	YR 5/4	1	3.5	0.6	32.8	1.36
2000	YR 8/2	1	3.5	0.6	35.5	1.29
2100	YR 8/2	1	3.5	0.6	32.2	1.42
2200	WH	1	5.9	3.0	33.0	1.41
2300						

Quick-firing Test:

Retention time, Min: 15		Temperature °F					
Particle Size Thru On		1800	1900	2000	2100	2200	2300
	Weight lb/cu ft.						
	Absorption Percent						

Retention time, Min:		Temperature °F					
Particle Size Thru On		1800	1900	2000	2100	2200	2300
	Weight lb/cu ft.						
	Absorption, Percent						

Other Tests & Comments: Very short and fine, powders when dry. $CaCO_3$ (qual.): slight pos. Very weak at all temperatures.

Potential Uses: Non-plastic filler to modify shrinkage and color in brick and other S.C.P. If a D.T.A. indicates high kaolin, it might be used for whitewares. A high P.C.E. would indicate potential use in refractories.

Table 3

#3518-B

NORTH CAROLINA CLAY AND SHALE INVESTIGATIONS

Sample No: N.C. 63 M-14 ~~X-14~~

County: Moore

Locality:

Hancock Clay Pit. Pit is located on the west side of county road 1413. 0.5 mile north of intersection with county road 1412, in NW corner of county.

Geology

Formation:

Carolina Slate belt
Lower volcanic-rocks
Felsic tuff

Type of Material:

Clay_____ Shale_____

Other weathered felsic tuff

Description of Outcrop or Exposure:

Pit is developed in weathered felsic tuff. Material crumbles easily and has a silty feel. East face of quarry is about 15' high and 100' long. Material is fairly uniform.

Attitude of Bedding:

N 45° E. 65° NW

Sampled Interval:

Grab sample collected from east face of pit.

Type of Sample:

Channel_____ Composite_____ Grab x

Other:

Present Use:

Pit is now inactive but has been mined and used as an addition to triassic brick clay.

Remarks:

Collected by S.G.C. and ~~U.S.G.S.~~ E.P.A. 6/25/63; E.P.A. & W.F.W. 2/13/69

Table 4

#3518-B

NORRIS METALLURGY RESEARCH LABORATORY

Laboratory Number 1562-I

Date Received _____ Date to Lab. _____ Date Reported _____

Name N. C. Geo. Survey Address _____

Sender's Number and Description 63 M-14, Moore Co.

Type Material Silt-clay Color Off white pH 4.89

Raw Properties

Working Characteristics Short working, fine grit

Water of Plasticity % 32.0 % Drying Shrinkage 1.0 Dry Strength Fair

Drying Characteristics Fair - uneven

Slow Firing Test

Temp. °F	Color	Hardness	% Lin. Shk.	% Abs.	App. Sp. Gr.
1800	Buff	Very soft	Crumbled in water		
* 1900	Buff	Very soft	3.5	33.8	2.43
2000	Gray	Very soft	5.0	32.5	2.42
2100	Gray	Soft	5.0	28.2	2.45
2200	Gray	Soft	6.0	25.5	2.46
2300	Gray	Fair hard	9.0	21.6	2.52
2400	Gray	Hard	10.5	18.7	2.54

Pyrometric Cone Equivalent ---- Bloating Test Negative

Remarks: 85 - 90% silica (silt) rest mostly clay

Potential Use: Additive for ceramic use or possible abrasive (scouring) powder.

Other Tests: _____

*1900 specimen finally broke to pieces in handling.

Table 5

#3518-C

NORTH CAROLINA CLAY AND SHALE INVESTIGATIONS

Sample No: N.C. ~~11-16~~ 63M-16

County: Moore

Locality: Williams Clay Pit. Pit is located 0.6 mi. north of County Road 1269. Mine road turns north off 1269, 0.8 mi NW of Highway 705. County road 1269 is located 4.8 mi NE of Elberta.

Geology

Formation:

Carolina Slate Belt
Volcanic-Sedimentary Rocks
Varved Slate

Type of Material:

Clay_____ Shale_____

Other Weathered varved slate

Description of Outcrop or Exposure:

Interbedded sequence of weathered varved slates that vary from white to yellowish buff to light pink in color.

Attitude of Bedding:

Sampled Interval:

Composite sample from stockpile.

Type of Sample: Channel_____ Composite X Grab_____

Other:

Present Use:

Inactive - used as additive to brick clay.

Remarks:

Collected by S.G.C. and ~~U.S.G.S.~~ E.P.A. 6/25/63; E.P.A. / W.F.W. 2/13/69

Table 6

#3518-C

NORRIS METALLURGY RESEARCH LABORATORY

Laboratory Number 1562-K

Date Received _____ Date to Lab. _____ Date Reported _____

Name N. C. Geo. Survey Address _____

Sender's Number and Description 63 M-16 Moore Co.

Type Material Clay Color White pH 4.80

Raw Properties

Working Characteristics Short working, smooth, fine grit

Water of Plasticity % 32.0 % Drying Shrinkage 2.5 Dry Strength Fair

Drying Characteristics Fair, wavey surface

Slow Firing Test

Temp. °F	Color	Hardness	% Lin. Shk.	% Abs.	App. Sp. Gr.
1800	--------	Crumbled			
1900	--------				
2000	Buff	Very soft	1.0	27.1	2.46
2100	Buff	Soft	1.0	24.9	2.57
2200	Buff	Fair hard	5.5	19.3	2.46
2300	Grey	Hard	10.0	12.4	2.45
2400	Grey	Very hard	11.5	5.8	2.43

Pyrometric Cone Equivalent ---- Bloating Test Negative

Remarks: About half silt and half clay. Good additive to a clay body needing silica

Potential Use: None (see note above)

Other Tests: _____

#3518-D

Table 7

NORTH CAROLINA CLAY AND SHALE INVESTIGATIONS

Sample No: N.C. 63 M-21

County: Moore

Locality: Pit located 200 ft SE of the intersection of county road 1006 and 1618, 2.2 mi. north of Glendon.

Geology

Formation:

Carolina Slate belt
Lower Volcanic rocks
Felsic tuff

Type of Material:

Clay_____ Shale_____

Other Weathered felsic tuff

Description of Outcrop or Exposure:

Pit is located in zone of deeply weathered felsic tuff. Materials has a well developed cleavage and splits in thin sheets on weathering. Tuff in S. face of pit is chalk white.

Attitude of Bedding:

N 70° E 50 NW

Sampled Interval:

Grab sample from south face of pit. Chalk white.

Type of Sample:

Channel_____ Composite_____ Grab_____

Other:

Present Use:

Additive to triassic brick clay.

Remarks:

Pit is presently being operated by Broden Brick and Tile Company. Clay is hauled, to Sanford and Durham by truck.

Collected by S.G.C. and ~~I.F.C.~~ E.P.A. 6/26/63; E.P.A. & WFW. 2/13/69

Table 8

#3518-D

NORRIS METALLURGY RESEARCH LABORATORY

Laboratory Number 1562-Z

Date Received _____ Date to Lab. _____ Date Reported _____

Name N. C. Geo. Survey Address _____

Sender's Number and Description 63M-21 ~~Hoke~~, Moore Co.

Type Material Clay Color Off white pH 7.10

Raw Properties

Working Characteristics Long working, slightly plastic, smooth, smitty

Water of Plasticity % 22.0 % Drying Shrinkage 2.5 Dry Strength Good

Drying Characteristics Good

Slow Firing Test

Temp. °F	Color	Hardness	% Lin. Shk.	% Abs.	App. Sp. Gr.
1800	Cream	Soft	1.5	22.0	2.57
1900	-------				
2000	Cream	Fair hard	6.0	12.8	2.52
2100	Pale brown	Hard	10.0	6.1	2.48
2200	Grey	Steel hard	13.5	0.3	2.38
2300	Grey	Steel hard	14.0	0.3	2.20
2400	Grey	Steel hard	7.5	0.9	2.31

Pyrometric Cone Equivalent ------ Bloating Test Negative

Remarks: Shrinkage a little high, poor color, wavey surface.

Potential Use: None

Other Tests: _____

#3518-E

Table 9

NORTH CAROLINA CLAY AND SHALE INVESTIGATIONS

Sample No: N.C. 76R - 1 County: Randolph

Locality: Jay Williams Site A - approximate 4.5 miles SW of Ulah in SW Randolph County, on SE Side of S.R. 1143 and 2.65 miles SW of intersection with S.R. 1142 on SW side of Jay Williams brick residence.

Geology:

Formation:

Felsic Volcanics

Type of Material:

Clay X Shale

Other

Description of Outcrop or Exposure:

100 yd. long strip of abandoned road bed parallel to new road, two 7 foot deep test pits 100 ft. apart along middle of road bed; white and buff sandy clay outcrops along S.R. 1143 for 0.2 miles.

Attitude of Bedding:

NE strike, steeply dipping

Sampled Interval:

Composite blend from two test pits

Type of Sample: Channel Composite X Grab

Other:

Present Use:

None

Remarks: Sampled by E.P.A. & W.R.H. 6/16/83 ; E.P.A. + W.F.W. 2/13/69

Table 10

II 7518-E

TUSCALOOSA METALLURGY RESEARCH LABORATORY

Laboratory Number NC-5-1

Date Received 6-28-68 Date Reported 7-29-68

Sender's Identification 76-R-1 Clay

Name North Carolina Geol. Survey Address Raleigh, N. C.

Type Material - Color White

Raw Properties:

Water of Plasticity, Percent 26.4 Working Properties Moderate plasticity

Drying Defects None Drying Shrinkage, Percent 0.0 Dry Strength Low

Slow Firing Test:

Temp. °F	Color	Moh's Hardness	Percent Total shk.	Percent Abs.	Percent App. Por.	Bulk Dens. gm/cc
1800	Tan	2	0.0	22.6	35.7	1.58
1900	Tan	2	2.5	21.8	34.8	1.60
2000	Tan	2	2.5	17.9	30.4	1.70
2100	Brown	3	7.5	9.1	17.9	1.97
2200	Gray-brown	4	10.0	1.6	3.5	2.19
2300	-	-	Expanded	-	-	-

Bloating Test Negative

Other Tests pH - 7.5 Not effervescent with Hcl

Potential Use Face brick

Remarks: Should fire to "SW" face brick specifications at about 2100°F Poor color.

The data presented in this report are based on laboratory tests that are preliminary in nature and will not suffice for plant or process design.

Table 11

ORE DRESSING TEST DATA

Lab. No. 3518 Test No. 1

Operator Edwin H. Bentzen III Date September 1969

Object of Test Chemical analysis, head feed

Sample #		Analysis Percent									
		LOI	Na_2O	K_2O	Al_2O_3	SiO_2	Fe_2O_3	CaO	MgO		Total
A		5.9	1.22	1.31	23.8	66.4	1.2	-	0.06		99.89
B	-	3.1	0.12	2.92	14.2	77.7	1.6	0.06	0.34		97.12
C		5.5	0.11	2.54	19.1	69.1	2.4	0.20	0.51		99.46
D		3.4	0.49	1.95	14.1	74.8	2.4	2.4	0.72		100.26
E		4.1	2.20	1.40	15.8	72.0	2.6	0.80	0.58		99.48

Conditions				Reagents (lbs per ton)							
Process	Time	% Solids	pH								

Remarks:

Table 12

Mineral Composition from Chemical Analysis*

A = % LOI

B = % SiO_2

C = % Al_2O_3

D = % CaO

E = % Na_2O

F = % K_2O

U = % albite, $Na_2O \cdot Al_2O_3 \cdot 6SiO_2$

V = % microcline, $K_2O \cdot Al_2O_3 \cdot 6SiO_2$

W = % anorthite, $CaO \cdot Al_2O_3 \cdot 2SiO_2$

X = % muscovite, $K_2O \cdot 3Al_2O_3 \cdot 6SiO_2 \cdot 2H_2O$

Y = % quartz, SiO_2

Z = % Kaolinite, $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$

$X = 7.813C - 22.097A - 14.201D - 12.847E - 8.455F$

$U = 8.458E$

$V = (F - 0.118X) \div 0.169$

$W = 4.960D$

$Z = (C - 0.195U - 0.183V - 0.365W - 0.385X) \div 0.395$

$Y = B - 0.687U - 0.648V - 0.430W - 0.452X - 0.465Z$

*Procedure described in Industrial Ceramics, Felix Singer and Sonja Singer, Chemical Publishing Co., Inc.; N.Y., N.Y., 1963, pp.301-302.

Table 13

Results of Trial Production Settling Time

Sample #	Settling Time (Min.)	Projected Clay Product Weight %	Actual Clay Product Weight %	% Albite	% Microcline	% Muscovite	% Qtz.	% Kaolinite
A	20	27.1	26.0	16.1	(None)	51.3	1.9	31.7
B	17	17.2	14.0	2.5	3.4	40.9	6.2	43.9
C	32	38.8	40.0	1.7	13.3	14.9	9.8	56.0
D	27	17.3	18.5	5.1	14.1	17.9	15.5	42.1
E	8	23.3	23.5	13.5	18.3	(None)	15.2	45.2

Table 14

ORE DRESSING TEST DATA

Lab. No. 3518 Test No. 6

Operator Edwin H. Bentzen/Johnny Lawrence Date

Object of Test Production of one pound of clay product.

Chemical Analy.	Wt. %		LOI	Na_2O	K_2O	Al_2O_3	SiO_2	Fe_2O_3	CaO	MgO	Tota
A	22.8		8.1	1.82	1.82	34.7	51.2	1.57	0.26	-	99.97
C	37.6		8.2	0.18	4.18	29.3	53.6	3.60	0.10	0.48	99.64
E	23.1		6.6	1.34	2.73	26.5	58.4	3.83	0.64	0.73	100.77
	-										
Rational Analy.			Albite	Micro	Anor	Musco	Qtz	Kaolin			
A			15.39	-	1.29	49.66	3.37	30.66			100.37
C			1.52	15.27	0.50	13.55	5.68	65.89			102.41
E			11.33	7.90	3.17	11.82	18.62	43.39			96.23
Color & Bulk wt.			B	G	A		Blk.wt.				
A			75	85	88		11.28				
C			63	75	79		10.30				
E			58	73	74		21.42				

Conditions Reagents (lbs per ton)

Process	Time	% Solids	pH								

Remarks:

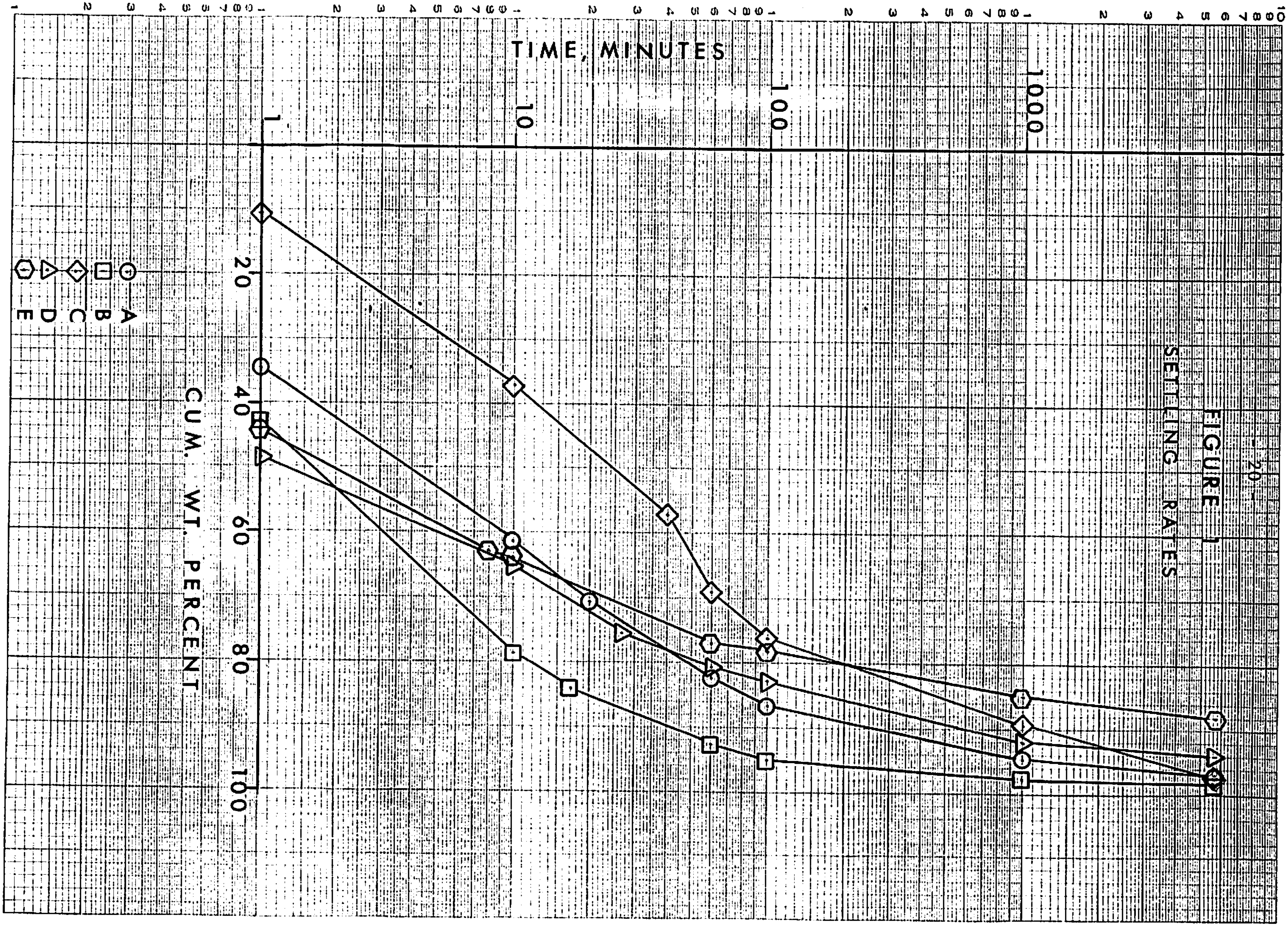

FIGURE 1
SETTLING RATES