

**SUITABILITY OF SYNTHETIC AGGREGATES MADE
FROM CLAY-TYPE SOILS FOR USE IN FLEXIBLE BASE**

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PREFACE

The primary objective of the synthetic aggregate research being conducted by the Texas Transportation Institute is to develop a recommended acceptance criterion for synthetic aggregates for use in all phases of highway construction. A tentative classification system was submitted in October, 1966, to the Texas Highway Department for review and comment. All synthetic aggregate research efforts are being conducted to further develop and improve this classification system in order that it will serve as a basis for a recommended acceptance criterion.

This is the fifth report issued under Research Study 2-8-65-81, one of the synthetic aggregate research studies being conducted at the Texas Transportation Institute in the cooperative research program with the Texas Highway Department and U. S. Bureau of Public Roads. The first four reports are:

“Correlation Studies of Fundamental Aggregate Properties with Freeze-Thaw Durability of Structural Lightweight Concrete” by W. B. Ledbetter, Research Report 81-1, Texas Transportation Institute, August 1965.

“Effect of Degree of Synthetic Lightweight Aggregate Pre-Wetting on the Freeze-Thaw Durability of Lightweight Concrete” by C. N. Kanabar and W. B. Ledbetter, Research Report 81-2, Texas Transportation Institute, December 1966.

“Aggregate Absorption Factor as an Indicator of the Freeze-Thaw Durability of Structural Lightweight Concrete” by W. B. Ledbetter and Eugene Buth, Research Report 81-3, Texas Transportation Institute, February 1967.

“Flexural Fatigue Durability of Selected Unreinforced Structural Lightweight Concretes” by J. C. Chakrabarti and W. B. Ledbetter, Research Report 81-4, Texas Transportation Institute, July 1967.

The authors wish to thank all members of the Institute who assisted in this research. They would like to express special appreciation to Mr. Lionel J. Milberger for suggestions and assistance in the study. His help in the development of equipment was particularly valuable. Special gratitude is also expressed to Dr. Edward A. Meyers for advice during the chemical investigation, to Mr. Charles E. Schlieker for suggestions during the development of several of the laboratory tests, and to Mr. Charles D. Carson for assistance in the interpretation of the X-ray diffraction and differential thermal patterns.

The authors wish to acknowledge the guidance and assistance given by the advisory committee for this study. The members are as follows: (a) Texas Highway Department Personnel—Mr. Kenneth D. Hankins, Study Contact Representative, Mr. Ivan K. Mays, Research Area Representative, Mr. H. A. Sandberg, Jr., Materials and Tests Division Representative, and Mr. H. D. Butler, Bridge Division Representative; (b) Bureau of Public Roads Personnel—Mr. Edward V. Kristaponis, Division Representative, and Mr. W. J. Lindsay, Regional Representative.

Appreciation is expressed to the Cordell Brick Company, Houston, Texas, for a valuable tour through its facilities and the American Colloid Company, Skokie, Illinois, which gratuitously furnished two of the clay materials used in the investigation.

The opinions, findings, and conclusions expressed in this publication are those of the authors and not necessarily those of the Bureau of Public Roads.

ABSTRACT

The 1966-67 plan of research for Phase 2 of Research Study 2-8-65-81 outlined an investigation to determine the limiting ranges of natural soils which can be fired to produce aggregates suitable for use in flexible base materials. This is a progress report of that investigation. Its purpose is to report the more significant findings to date, so that early utilization of the research may be possible. The findings are listed briefly below. They are based on limited study and should be considered preliminary and subject to change with future research.

1. The clay minerals, montmorillonite, illite, and kaolinite, will not rehydrate under atmospheric conditions once they have been completely dehydrated (dehydroxylated); therefore, once they have been completely dehydrated, they become chemically stabilized for use as highway construction materials. Complete dehydration is accomplished by heating the clay and holding it at the elevated temperature for sufficient time to allow the dehydration to occur. A period of 15 minutes at 1400°F. was sufficient to completely dehydrate the clay present in the small, oven dry laboratory specimens made from the Texas soils investigated.

2. Incomplete dehydration of aggregates made by dehydrating clay-type soils can be detected by a relatively simple laboratory test. The test procedure is given in Appendix 8.1.1.

3. Most (if not all) clay-type soils having a relatively high strength when air dried can be fired to produce hard, durable aggregates suitable for use in flexible base and asphaltic concrete.

The authors feel that synthetic aggregates produced from soils that are suitable for flexible base and asphaltic concrete will also, in many cases, be suitable for use as portland cement concrete aggregates. However, it should be noted that there are many chemical compounds present in synthetic aggregates produced from naturally occurring soils. Some of these compounds, though not detrimental in flexible base or asphaltic concrete applications, may adversely affect the hydration of portland cement concrete. Application of synthetic aggregates to portland cement concrete is the subject of Phase 1 of this research study and is not considered in this report.

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1. Introduction

While other reports on Research Study 2-8-65-81 have been published, this report is the first in the series which deals specifically with synthetic aggregates for flexible base. Consideration of synthetic aggregates for flexible base was added as Phase 2 of the Study in September, 1966. This is a progress report of the first year's efforts in this phase and the findings reported are based on limited study and should be considered preliminary and subject to change as future research indicates.

The primary objective of Phase 2 of this research is to develop a recommended acceptance criterion for synthetic aggregate base materials that are produced from naturally occurring soils. In the initial stages of this phase, the researchers recognized that aggregates could not be made in a rotary kiln from any soils other than the plastic, clay-type soils. Soils that have low plasticity also have low strength when air dried and therefore would be powdered by the tumbling action associated with such a kiln. The investigation reported here deals only with clay-type soils.

Aggregates produced by burning clay-type soils are necessarily the product of thermo-chemical reactions that take place during the firing operation. Because clay-type soils found in nature are heterogeneous mixtures of a wide variety of materials, it seemed of paramount importance to investigate the chemical as well as the physical stability of aggregates produced from them. To be more specific, the research was oriented toward answers to the following three questions:

1. Will synthetic aggregates made from soils that have been fired deteriorate when they are placed as flexible base in roadway?
2. Can laboratory tests indicate whether or not synthetic aggregates produced from clay-type soils will remain stable in a roadway environment?
3. How do the properties of synthetic aggregates produced from soils compare with aggregates that are currently being used in highway construction?

Preliminary results indicate answers to these three questions that are favorable to the use of synthetic aggregates in flexible bases.

2. Materials

For the investigation reported here, four aggregates produced in commercial rotary kilns for use as synthetic aggregate flexible base (these aggregates are nonbloomed and are too heavy to be classified as lightweight aggregates) and their respective raw soils were gathered from Hopkins County, Wharton County, and Madison County. Two of the four aggregates have been used in flexible base by the Texas Highway Department. The aggregates made from the Hopkins County soil have been used by the Paris district, and the aggregate made from the Wharton County soil has been used by both the Houston and the Yoakum districts. The two aggregates made from Madison County soils were investigated by the Bryan district for possible future use in flexible base. The results of laboratory tests conducted by the Bryan district indicated that either of the Madison County aggregates would be suitable for use as flexible base (1).¹ In order to compare test results, five other synthetic aggregates produced commercially in Texas are included in the investigation. They are the lightweight aggregates that are currently under investigation for portland cement concrete application in Phase 1 of this study (2).

Samples of ten other natural clay-type soils not previously used for synthetic aggregate flexible base were obtained from the following areas: two from Nueces County, two from Harris County, one from Liberty County, and five from Brazos County. Four additional soils, each having primarily a single clay mineral in its clay-size fraction (Na-montmorillonite, Ca-montmorillonite, illite, or kaolinite) were obtained commercially from sources outside Texas. Thus, this study is an investigation of (a) 18 clay-type soils of various compositions,

four of which at the outset appeared, on the basis of previous tests, to be suitable for use in the production of synthetic aggregate flexible base, and (b) 9 synthetic aggregates, four of which were produced commercially for flexible base and five of which are lightweight aggregates that are commercially available in Texas. Table 2-1 lists the material designations used in the text and shows generally how the materials were investigated.

TABLE 2-1. LIST OF MATERIALS INVESTIGATED

No.	Designation Used in Text	Commercially Produced Aggregates Investigated?	Natural Soils Investigated?
1	Hopkins	Yes	Yes
2	Wharton	Yes	Yes
3	Madison 1	Yes	Yes
4	Madison 2	Yes	Yes
5	Nueces 1	No	Yes
6	Nueces 2	No	Yes
7	Liberty	No	Yes
8	Harris 1	No	Yes
9	Harris 2	No	Yes
10	Brazos 1	No	Yes
11	Brazos 2	No	Yes
12	Brazos 3	No	Yes
13	Brazos 4	No	Yes
14	Brazos 5	No	Yes
15	Na-Mont.	No	Yes
16	Ca-Mont.	No	Yes
17	Illite	No	Yes
18	Kaolin	No	Yes
19	R	Yes	No
20	S	Yes	No
21	C	Yes	No
22	E	Yes	No
23	D	Yes	No

¹Numbers in parentheses refer to references listed in section 7.

TABLE 2-2. PROPERTIES OF SOILS USED IN STUDY

Sample	Liquid Limit	Plastic Limit	Gradation*		Minerals in Clay Fraction**		
			Percent Silt	Percent Clay	Percent Mont.	Percent Kaolin	Percent Illite
Hopkins	49	21	44	48	62	13	20
Wharton	67	23	15	83	25	20	50
Madison 1	61	28	19	62	25	45	25
Madison 2	83	27	35	48	90		9
Nueces 1	66	22	54	33	50	20	25
Nueces 2	70	23	16	58	27	15	48
Liberty	48	16	35	43	50	20	25
Harris 1	37	16	41	38	70	18	7
Harris 2	66	24	29	66	20	20	50
Brazos 1	63	27	26	46	50	15	30
Brazos 2	62	28	29	52	70	13	15
Brazos 3	71	36	31	65	30	40	25
Brazos 4	51	27	37	35	20	60	15
Brazos 5	93	33	25	68	65	15	18
Na-Mont.	600	47	5	92	99		
Ca-Mont.	70	40	49	44	100		
Illite	55	26	47	52		18	70
Kaolin	45	29	1	99		100	

*Determined by hydrometer analysis (MIT Classification: clay—smaller than 2 microns and silt—2 to 60 microns). Because of inability to disperse sample, gradation for Na-Mont. taken from suppliers report (5).

**Amount of clay minerals in clay fraction estimated from X-ray diffraction peak intensities (4).

The clay fraction (the minus two micron fraction) found in the Texas soils ranged from 30 to 80 percent by weight, and consisted primarily of three clay minerals: montmorillonite, illite, and kaolinite.

Table 2-2 shows the Atterburg limits, gradation, and the approximate quantities of the clay minerals for the 18 soils used in this investigation. It is believed that these 18 soils are representative of the clay-type soils found in Texas. The Atterburg limits were determined

by standard Texas Highway Department test procedures (3). The gradations shown were determined by hydrometer analysis, also in accordance with standard Texas Highway Department procedure. The approximate quantities of the clay minerals were estimated from the peak intensities of X-ray diffraction patterns of the clay fractions (4). Typical differential thermal patterns and X-ray diffraction patterns are given in Appendices 8.2 and 8.3.

3. Sample Preparation and Firing

In order to develop a standard laboratory aggregate for firing and testing, aggregates were made by extruding the soils (at a moisture content equal to the plastic limit) through a $\frac{1}{2}$ " diameter die. The extruded soil was then cut in lengths of $1\frac{1}{2}$ ". The extrusion apparatus in operation is shown in Figure 3-1. Soil in the various stages of processing is shown in Figure 3-2. The extruded samples were air-dried for 24 hours, then dried for 24 hours in an oven set at 140°F. and finally dried for 24 hours in an oven set at 212°F. This long drying procedure was used in order to eliminate shrinkage and maintain the cylindrical shape of the aggregates. After the drying operation, about ten samples of each soil were fired in a preheated muffle furnace for 15 minutes at each of the following temperatures: 600, 1000, 1200, 1400, 1600, 1800, and 2000°F. After 15 minutes in the muffle furnace the samples were removed and allowed to cool to room temperature. Photographs of the firing operation are shown in Figure 3-3. Prior to testing, the samples were sawed to a length of one inch with parallel saws.

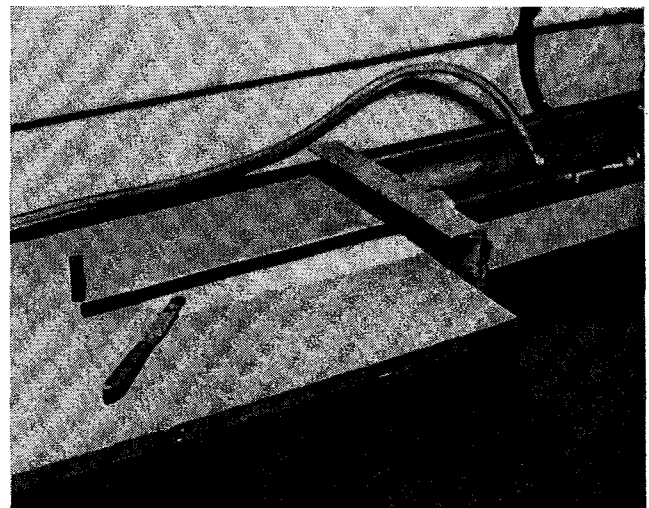


Figure 3-1. Extrusion of plastic soil.

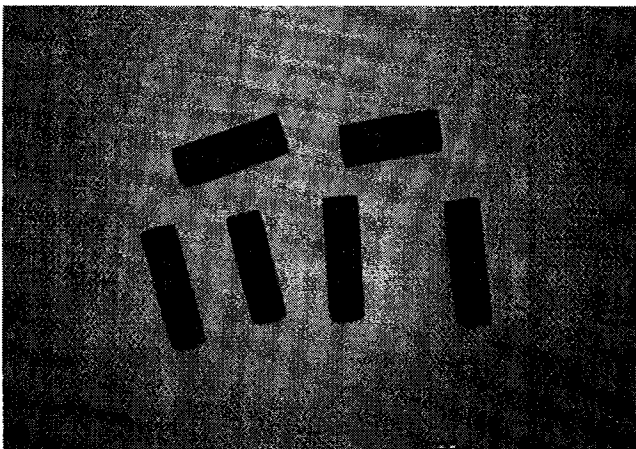
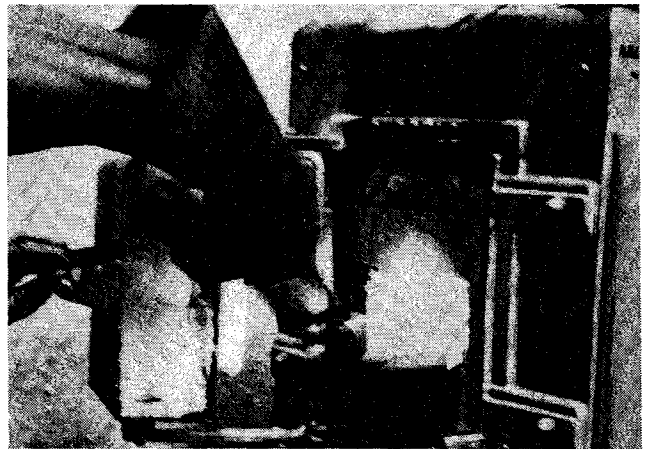
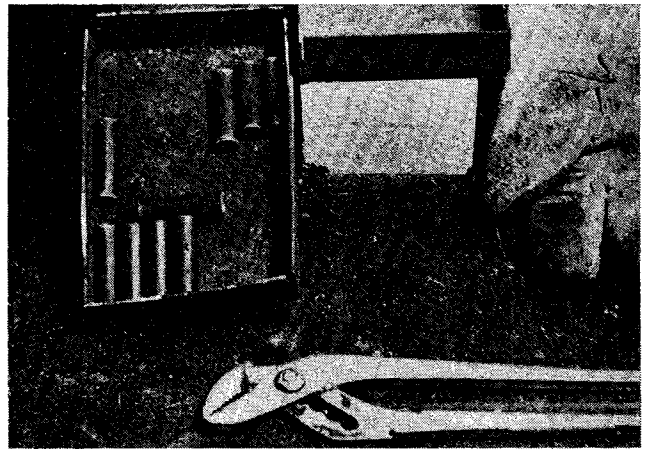
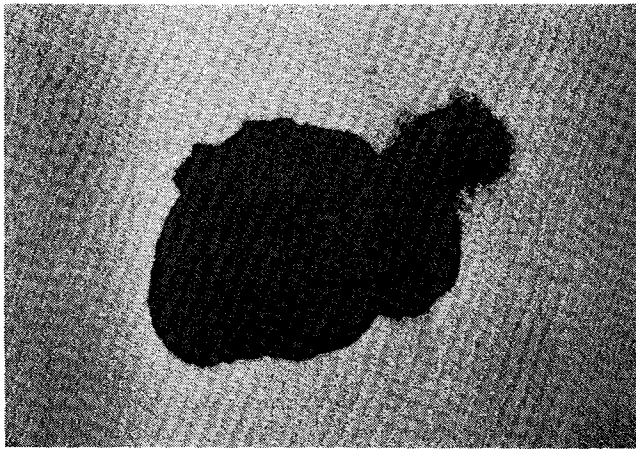


Figure 3-2. Soil in various stages of processing. The upper photograph shows pulverized air dried soil, the middle shows soil at the plastic limit ready for extrusion, and the lower shows the 1½ in. by ½ in. diameter extruded samples.

Figure 3-3. Firing operation of the samples. The upper photograph shows the oven dried specimens ready for firing, the middle shows the samples being placed in the preheated muffle furnace, and the lower shows the samples being removed.

4. Chemical Stability

In order to determine the chemical stability of the fired aggregates, several different laboratory tests were investigated. An obvious requirement for any synthetic aggregate used in flexible base is that it remain stable indefinitely after being placed in a roadway. Therefore, laboratory tests were designed to subject the aggregates to a more severe treatment than would ever be experienced in a roadway environment based on the supposition that aggregates withstanding the severe laboratory tests would also remain stable in a roadway.

The two laboratory tests that were probably the most severe were the treatment of the aggregates with boiling, 5-N solutions of hydrochloric acid (HCl) and sodium hydroxide (NaOH). It should be noted that about 80% of the aggregates were sawed with parallel saws (exposing the interior of the aggregate) prior to chemical test-

ing. No significant difference was noted between the pre-sawed samples and those samples which were not or could not be sawed due to fracturing during firing, etc. The acid treatment seemed to be much less severe, because it did not affect many of the aggregates that had not been dehydrated; whereas the alkaline solution broke down all of these aggregates. Typical results of these treatments are shown in Figure 4-1. The acid also had no visible effect on any of the aggregates fired at 1400°F. and higher, while the NaOH produced a slight deterioration in some of these aggregates.

Because of the visible distress associated with the boiling NaOH, a test was designed to subject aggregates to a severe treatment with it. This test will be referred to as the NaOH Test. The procedure used for conducting it is given in Appendix 8.1.2. It is estimated that the treatment given the aggregates in the NaOH Test is equivalent to more than a year's exposure to an extremely adverse environmental condition—that is, submersion in an aqueous solution of NaOH having a pH of 12 and a temperature of 130°F.² The equipment used in the test, and typical aggregates after testing, are shown in Figure 4-2. Results of testing the laboratory aggregates are given in Table 4-1. Although some of the aggregates fired at 1400°F. and higher appear to have been partially destroyed by the treatment, results of the physical tests on the aggregates after treatment (both compressive strength and abrasive resistance) indicated that the partly destroyed aggregates were not significantly affected. In fact, in many instances they appeared stronger after treatment. Compressive strengths of aggregates made from two of the Texas soils which appear in the table to have been somewhat affected by the NaOH Test are given in Table 4-2.

Samples of nine synthetic aggregates produced in commercial rotary kilns were also subjected to the NaOH Test. The results are given in Table 4-3. Considerable degradation in some of the larger size aggregates can be noted in Table 4-3, but the test did not degrade the material enough to produce any significant quantity of particles finer than number 40 mesh sieve. The degradation in the larger aggregates does indicate detrimental alkaline chemical reactions which may adversely affect their use in portland cement concrete. However, if some degradation in the larger aggregates should occur in a flexible pavement environment, the authors do not believe it would cause any significant detrimental effect in flexible base or asphaltic concrete application.

Probably the most significant thermo-chemical reaction that occurred in the laboratory fired aggregates in the 1000-1400°F. temperature range was the expulsion of the hydroxyl water in the clay minerals. This expulsion of hydroxyl water is normally termed complete dehydration (6). Like most thermal reactions the time required for it to occur will decrease as the reaction temperature is increased (7). In fact, if the reaction time is long enough, the authors believe that most clay-type soils would completely dehydrate at 1000°F., and also that

²The assumptions used to make this estimate are believed to be conservative. They were that the reaction rate would (a) double for each 10°C. temperature increase and (b) be directly proportional to the solution concentration.

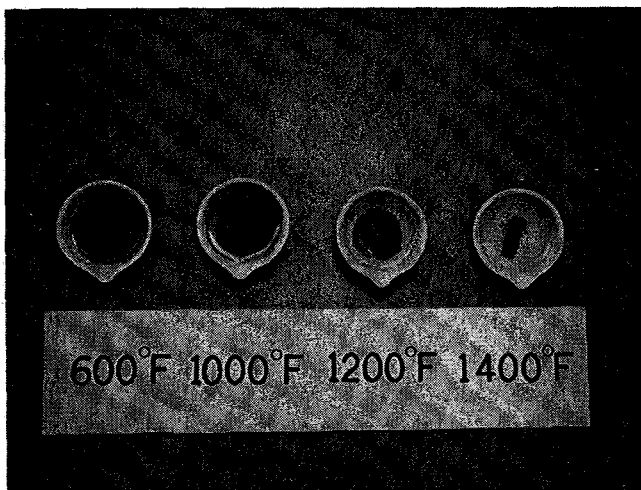
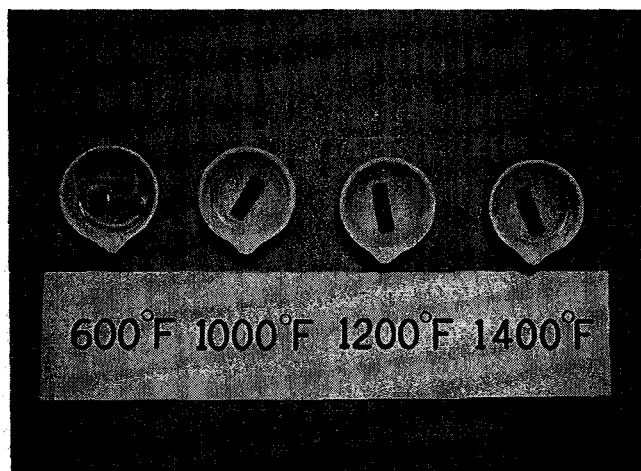


Figure 4-1. Laboratory aggregates after severe chemical treatments. The upper photograph shows aggregates after severe treatment with HCl and the lower shows aggregates after severe treatment with NaOH. Samples were fired for 15 minutes at the temperatures indicated in the photographs.

TABLE 4-1. RESULTS OF NaOH TEST ON EXTRUDED CLAY AGGREGATES FIRED AT VARIOUS TEMPERATURES

Sample	600°F	1000°F	1200°F	1400°F	1600°F	1800°F	2000°F
Hopkins	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF	NO EFF
Wharton	DST	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF
Madison 1	DST	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF
Madison 2	DST	PT DST	PT DST	PT DST	PT DST	PT DST	PT DST
Nueces 1	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF	NO EFF
Nueces 2	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF	NO EFF
Liberty	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF	NO EFF
Harris 1	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF	NO EFF
Harris 2	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF	NO EFF
Brazos 1	DST	PT DST	PT DST	PT DST	NO EFF	PT DST	PT DST
Brazos 2	DST	PT DST	NO EFF	NO EFF	NO EFF	PT DST	PT DST
Brazos 3	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF	NO EFF
Brazos 4	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF	NO EFF
Brazos 5	DST	DST	PT DST	PT DST	PT DST	PT DST	PT DST
Na-Mont.	DST	DST	DST	DST	PT DST	PT DST	PT DST
Ca-Mont.	DST	DST	DST	PT DST	NO EFF	NO EFF	NO EFF
Illite	DST	DST	DST	NO EFF	NO EFF	NO EFF	NO EFF
Kaolin	DST	DST	DST	NO EFF	NO EFF	NO EFF	NO EFF

DST—Sample was completely destroyed.

PT DST—Sample was partly destroyed.

NO EFF—No visible effect on sample.

they will not completely dehydrate at 2000°F. if the reaction time is too short.

A relatively simple test was found to be a good indicator of the tendency of partially dehydrated aggregates to rehydrate. It is called the Slaking Test and involves cooking aggregates under water in a common, kitchen-type, pressure cooker. The procedure used for conducting the Slaking Test is given in Appendix 8.1.1. Results for the laboratory aggregates subjected to it are given in Table 4-4. When the aggregates slaked or decomposed even slightly as a result of the test, the slaked-down material had evidently rehydrated because it was plastic (that is, it could be molded with one's fingers). All of the aggregates fired at 1000°F. were found to slake somewhat. Half of them fired at 1200°F., and all of them, except the Na-montmorillonite aggregate, fired at 1400°F. and higher were found to be unaffected.

Additional Na-montmorillonite aggregates were prepared and fired at 1200 and 1400°F. as described in Section 3, Sample Preparation and Firing, except that

TABLE 4-2. COMPRESSIVE STRENGTH—BEFORE AND AFTER NaOH TEST*

Firing Temp. °F	Madison 2		Brazos 1	
	Comp. Str. After Firing (psi)	Comp. Str. After Test (psi)	Comp. Str. After Firing (psi)	Comp. Str. After Test (psi)
600	4400	Destroyed	3400	Destroyed
1000	4200	5500	4400	1700
1200	4900	5000	3400	2000
1400	6300	6400	5000	3600
1600	8000	13,700	7500	5300
1800	1800	1800	6200	13,700
2000	2700	2000	7600	5300

*The values in the table are comparisons between the compressive strength of one aggregate after firing to one that was subjected to the NaOH Test, dried, and then tested in compression.

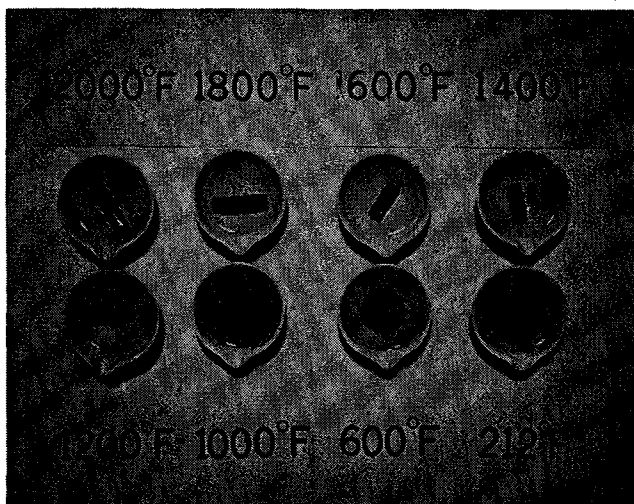
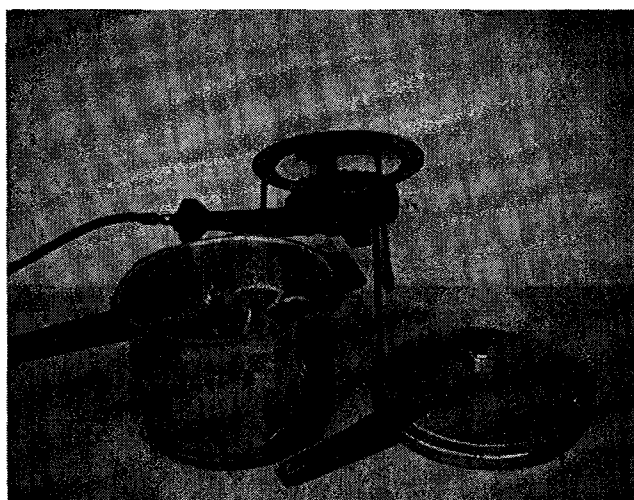


Figure 4-2. Sodium hydroxide Test. The upper photograph shows the testing equipment and the lower shows typical aggregates after testing.

TABLE 4-3. RESULTS OF NaOH TEST ON SYNTHETIC AGGREGATES PRODUCED IN COMMERCIAL KILNS*

Aggregate**	Percent Retained on Sieves Before Test			Percent Retained on Sieves After Test		
	%"	No. 10	No. 40	%"	No. 10	No. 40
Hopkins	13.7	85.0	100.0	12.1	83.6	99.1
Wharton	49.5	91.5	100.0	46.5	91.0	99.0
Madison 1	9.0	84.0	100.0	7.9	80.2	96.4
Madison 2	10.0	68.0	100.0	7.4	67.1	99.1
R	56.6	100.0	100.0	56.7	99.7	100.0
S	37.1	99.7	100.0	27.5	99.4	99.8
C	66.9	98.1	100.0	61.3	97.6	99.7
E	82.2	100.0	100.0	71.8	99.8	99.9
D	57.8	100.0	100.0	41.4	99.2	99.7

*Values in the table are based on a NaOH Test (Appendix 8.1.2) made on one sample (approximately 600 grams) for each aggregate.

**The Hopkins and the Wharton aggregates have been used in flexible base on Texas highways, the two Madison aggregates have been investigated for possible similar use in the future, and the remaining five aggregates are samples of lightweight aggregates that are available commercially in Texas.

the firing time was doubled (increased from 15 to 30 minutes). The aggregates fired at 1200°F. were partially destroyed by the Slaking Test, and the ones fired at 1400°F. were not affected. Similarly, the aggregates fired at 1200°F. were destroyed by the NaOH Test, and those fired at 1400°F. were only slightly affected. Comparison of the chemical tests made on the aggregates fired for 15 minutes with those made on the aggregates fired for 30 minutes indicated the time dependency of the thermochemical reactions. In other words, 15 minutes at 1400°F. was not long enough to completely dehydrate the Na-montmorillonite soil; whereas 30 minutes was sufficient.

TABLE 4-5. RESULTS OF SLAKING TEST ON SYNTHETIC AGGREGATES PRODUCED IN COMMERCIAL KILNS*

Aggregate**	Percent Retained on Sieves Before Test			Percent Retained on Sieves After Test		
	%"	No. 10	No. 40	%"	No. 10	No. 40
Hopkins	11.0	92.0	100.0	11.1	92.0	99.8
Wharton	42.3	94.7	100.0	45.6	93.8	99.2
Madison 1	6.6	75.7	100.0	6.6	74.6	99.7
Madison 2	4.4	59.8	100.0	4.3	59.4	98.7
R	59.2	100.0	100.0	58.2	99.8	99.8
S	18.8	100.0	100.0	18.4	99.9	99.9
C	27.5	98.3	100.0	28.6	98.1	99.7
E	64.8	100.0	100.0	62.0	99.9	100.0
D	42.6	100.0	100.0	41.8	99.9	100.0

*Values in the table are based on a Slaking Test (Appendix 8.1.1) made on one sample (approximately 600 grams) for each aggregate.

**The Hopkins and the Wharton aggregates have been used in flexible base on Texas highways, the two Madison aggregates have been investigated for possible similar use in the future, and the remaining five aggregates are samples of lightweight aggregates that are available commercially in Texas.

Results of the Slaking Test made on synthetic aggregates produced in commercial kilns are given in Table 4-5. None of these aggregates were significantly affected.

From the results of the chemical tests made on synthetic aggregates, it is concluded that all of the clay-type soils studied can be completely dehydrated at some temperature between 1000 and 1400°F., provided that sufficient time is allowed for the reaction, and that once they have been completely dehydrated, they are chemically stabilized sufficiently for use in flexible base and asphaltic concrete. It is also concluded that the Slaking Test is a good indicator as to whether or not aggregates have been completely dehydrated.

TABLE 4-4. RESULTS OF SLAKING TEST ON EXTRUDED CLAY AGGREGATES FIRED AT VARIOUS TEMPERATURES

Sample	600°F	1000°F	1200°F	1400°F	1600°F	1800°F	2000°F
Hopkins	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF	NO EFF
Wharton	DST	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF
Madison 1	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF	NO EFF
Madison 2	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF	NO EFF
Nueces 1	DST	PT DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF
Nueces 2	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF	NO EFF
Liberty	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF	NO EFF
Harris 1	DST	PT DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF
Harris 2	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF	NO EFF
Brazos 1	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF	NO EFF
Brazos 2	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF	NO EFF
Brazos 3	DST	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF
Brazos 4	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF	NO EFF
Brazos 5	DST	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF
Na-Mont.	DST	DST	DST	PT DST	NO EFF	NO EFF	NO EFF
Ca-Mont.	DST	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF
Illite	DST	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF
Kaolin	DST	DST	PT DST	NO EFF	NO EFF	NO EFF	NO EFF

DST—Sample was destroyed (it returned to plastic state).

PT DST—Part of sample returned to plastic state.

NO EFF—No visible effect on sample.

5. Physical Stability

Three tests were used to evaluate the physical properties of the laboratory aggregates. They were compressive strength, hardness, and abrasion resistance, which are illustrated on Figure 5-1. The compressive strength was measured in a universal testing machine operated at a loading rate of 0.05 inches per minute. Hardness was measured using the procedure for the 15W Rockwell hardness test scale outlined in ASTM Designation E 18 (8). The loss due to abrasion was taken as the decrease in length of a cylindrically shaped aggregate abraded by a grinding wheel in a fixed time; a small length loss indicates high abrasive resistance. Details of the abrasion test are given in Appendix 8.1.3.

The results of compressive strength, hardness, and abrasion tests made on laboratory aggregates are given in Tables 5-1, 5-2, and 5-3, respectively. It should be noted that the values given in these tables were obtained by testing one specimen made at each firing temperature. The results of a special experiment conducted to determine the reliability of testing a single laboratory prepared aggregate are given in Appendix 8.4. The experiment indicated that the chances are about 2 out of 3 that the result of a single compression test will be within 23% of the average obtained from a large number of tests. Similar percentages for the Rockwell hardness and the abrasion loss tests were 15% and 30%, respectively. The chances are about 95 out of 100 that the result of a single test will be within twice these percentages. One-half inch diameter cores of a few conventional materials were similarly tested for compression, hardness, and abrasion. The results are included in Table 5-4 for comparison with Tables 5-1 through 5-3. Aggregates were selected for coring to represent the variability of conventional materials. Variations in this table beyond 15% to 30% are taken to represent real variations in materials rather than the method of testing. The results obtained on most of the laboratory aggregates compare favorably with those made on the cores of conventional materials. It can be noted from Table 5-1 that generally the soils having a compressive strength greater than 1000 psi when dried in an oven set at 212°F. made the strongest aggregate when fired. However, the most important single fact which one can observe from the data given in Tables 5-1 through 5-4 is that aggregates comparing favorably with conventional aggregates can be made in the laboratory from *all 14 clay-type Texas soils investigated*.

Aggregates obviously cannot be made in rotary kiln from the kaolinite soil; it has almost no strength when it is dry and would be powdered in such a kiln. It is questionable whether the Harris 1 or the Brazos 5 soil would be suitable for the same reason. No *definite* correlation was found between the plasticity of the raw soil and the physical characteristics of the laboratory aggregates; however, the authors do not believe that aggregates can be made from soils having a plasticity index less than about 20. It is also questionable whether any aggregates larger than a $\frac{3}{8}$ " sieve could be made from Na-montmorillonite or the Ca-montmorillonite soils because of the fracturing resulting from the tremendous shrinkage occurring during the dehydration of these soils. However, the small aggregates that were produced in the

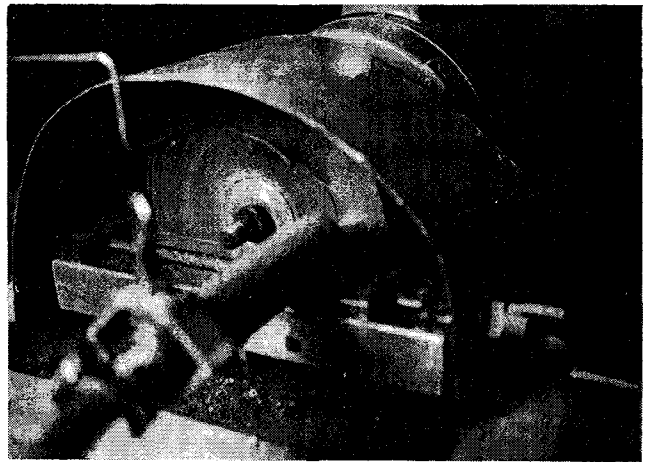
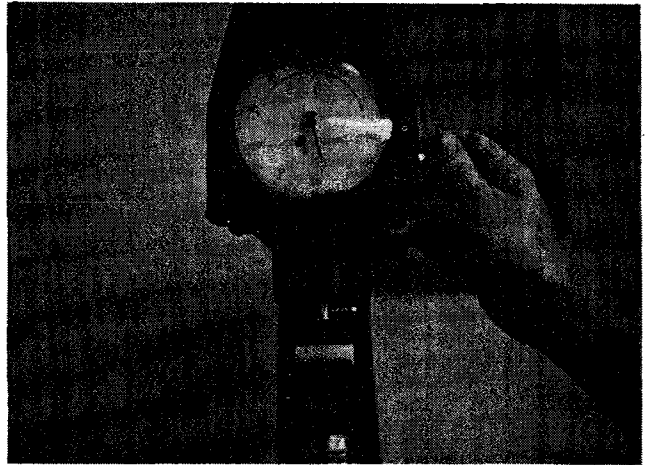
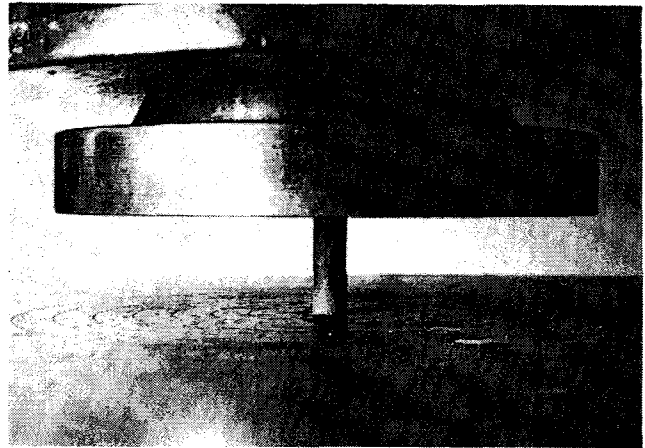


Figure 5-1. Physical tests made on laboratory aggregates. The upper photograph shows aggregates being tested in compression, the middle shows a hardness test and the lower shows an abrasion test.

laboratory appeared extremely hard and durable, though the pieces remaining after firing were too small to permit the performance of strength tests.

In about half of the soils tested an optimum firing temperature seemed to exist similar to that reported by Henson K. Stephenson (9). According to Stephenson,

TABLE 5-1. COMPRESSIVE STRENGTHS OF EXTRUDED CLAY AGGREGATES FIRED AT VARIOUS TEMPERATURES*

Sample	212°F	1000°F	1200°F	1400°F	1600°F	1800°F	2000°F
Hopkins	2100	3200	4400	4100	5200	7800	10,400
Wharton	1500	6300	6700	10,400	9900	6200	BRK
Madison 1	NM	1600	3900	5500	5400	8600	9000
Madison 2	2400	4200	4900	6300	8000	1800	2700
Nueces 1	1500	2900	4000	6900	6300	7800	4900
Nueces 2	1200	4900	6600	6100	6500	5900	6700
Liberty	1300	2900	3800	4900	6000	6600	5800
Harris 1	770	2700	3700	3500	2900	3600	6700
Harris 2	NM	2700	4300	6400	11,500	9800	3800
Brazos 1	2800	4400	3400	5000	7500	6200	7600
Brazos 2	2100	6200	7100	7500	6000	6100	6000
Brazos 3	1800	BRK	4700	6000	BRK	8300	5300
Brazos 4	2700	3800	4200	4800	5300	5600	5000
Brazos 5	600	1400	1300	1600	2500	5000	6500
Na-Mont.	BRK	BRK	BRK	BRK	BRK	BRK	BRK
Ca-Mont.	BRK	BRK	BRK	BRK	BRK	BRK	BRK
Illite	1100	300	600	2300	BRK	BRK	BRK
Kaolin	50	80	130		140	200	80

NM—No test made.

BRK—Samples fractured during firing and could not be tested.

*Compressive strengths are expressed in psi. The values given in table were obtained by testing one specimen at each firing temperature. The coefficient of variation is about 23 percent (see Appendix 8.4).

TABLE 5-2. HARDNESS OF EXTRUDED CLAY AGGREGATES FIRED AT VARIOUS TEMPERATURES*

Sample	1000°F	1200°F	1400°F	1600°F	1800°F	2000°F
Hopkins	5	25	33	46	70	70
Wharton	11	22	36	55	17	42
Madison 1	VS	1	7	31	40	60
Madison 2	13	29	42	72	9	1
Nueces 1	VS	VS	3	21	41	31
Nueces 2	VS	VS	7	16	21	25
Liberty	VS	VS	VS	4	20	22
Harris 1	VS	VS	VS	VS	VS	27
Harris 2	VS	16	16	46	34	24
Brazos 1	3	5	19	44	61	62
Brazos 2	21	22	52	72	54	47
Brazos 3	BRK	6	26	BRK	VS	VS
Brazos 4	VS	8	25	42	45	21
Brazos 5	VS	VS	VS	VS	BRK	51
Na-Mont.	BRK	BRK	BRK	BRK	BRK	BRK
Ca-Mont.	BRK	BRK	BRK	BRK	BRK	BRK
Illite	VS	VS	BRK	BRK	BRK	BRK
Kaolin	VS	VS	VS	VS	VS	VS

VS—Too soft to obtain hardness reading.

BRK—Samples fractured during firing and could not be tested.

*The hardness values given in the table are the average of three (Rockwell 15-W) readings (ASTM E 18) obtained on one specimen made at each firing temperature. The coefficient of variation is about 15 percent (see Appendix 8.4).

TABLE 5-3. ABRASION LOSS OF EXTRUDED CLAY AGGREGATES FIRED AT VARIOUS TEMPERATURES*

Sample	1000°F	1200°F	1400°F	1600°F	1800°F	2000°F
Hopkins	0.465	0.213	0.079	0.054	0.026	0.039
Wharton	0.239	0.100	0.132	0.033	0.006	0.023
Madison 1	0.330	0.116	0.027	0.021	0.011	0.003
Madison 2	0.336	0.178	0.026	0.011	0.140	0.189
Nueces 1	>1.0	0.350	0.128	0.020	0.010	0.012
Nueces 2	>1.0	0.458	0.035	0.057	0.034	0.030
Liberty	>1.0	>1.0	0.881	0.030	0.259	0.620
Harris 1	>1.0	>1.0	>1.0	>1.0	0.197	0.102
Harris 2	0.432	0.196	0.124	0.027	0.020	0.018
Brazos 1	0.642	0.119	0.037	0.018	0.023	0.023
Brazos 2	0.273	0.024	0.011	0.007	0.006	0.079
Brazos 3	BRK	0.103	0.183	BRK	BRK	0.313
Brazos 4	>1.0	0.131	0.094	0.239	0.198	0.207
Brazos 5	>1.0	>1.0	>1.0	>1.0	BRK	0.002
Na-Mont.	BRK	BRK	BRK	BRK	BRK	BRK
Ca-Mont.	BRK	BRK	BRK	BRK	BRK	BRK
Illite	BRK	0.498	BRK	BRK	BRK	BRK
Kaolin	>1.0	>1.0	>1.0	>1.0	>1.0	>1.0

BRK—Samples fractured during firing and could not be tested.

*Abrasion loss is expressed in inches. The values given in the table were obtained by testing one specimen made at each firing temperature. The coefficient of variation is about 30 percent (see Appendix 8.4). See Appendix 8.1.3 for procedure.

TABLE 5-4. RESULTS OF PHYSICAL TESTS MADE ON CORES OF CONVENTIONAL AGGREGATES*

Description	Compressive Strength (psi)	Rockwell Hardness	Abrasion Loss (In.)
Brazos Co., Iron Ore 1	1200- 4200 (3)	VS- 1 (3)	0.007-0.175 (3)
Brazos Co., Iron Ore 2	1900- 3800 (3)	VS (3)	0.017 (1)
Brazos River Gravel	6700-24500 (2)	47-64 (3)	0.012 (1)
Walker Co., Cr. Ss.	4900-13900 (4)	VS-56 (3)	0.006-0.025 (3)
Grimes Co., Cr. Ss.	290- 630 (3)	VS (3)	>1.0 (1)
East Texas Cr. Ss.	13,800 (1)	31-50 (3)	0.005 (1)
Bexar Co., Ls. Gravel	2700- 3500 (3)	6-42 (4)	0.108-0.132 (2)
Waco Cr. Ls.	6100- 8900 (3)	61-77 (2)	0.015-0.061 (2)
AASHO Rd. Test Ls.	6000- 9000 (3)	67-78 (2)	0.013-0.017 (2)
Serv-Tex Cr. Ls.	3200- 9400 (3)	32-69 (4)	0.010 (2)

VS—Too soft to obtain hardness reading on 15-W scale.

*The numbers in parentheses represent the number of cores of each aggregate type that were subjected to the test indicated by the column headings. The actual test values are given in the table for the low-high results obtained from the cores tested. The cores were tested in accordance with the same procedures used for the laboratory aggregates made from soils and the results can be compared with those given in Tables 5-1, 5-2, and 5-3.

the optimum temperature is the particular temperature at which the strongest, most desirable aggregates are produced. Because of the many and complex thermochemical reactions that occur in soils with their temperature, atmosphere, and reaction rate interrelationships, the authors do not place much physical significance on an optimum temperature determined in a muffle furnace. Nevertheless, the fact that an optimum temperature may exist for a given set of conditions does suggest the possibility of improving the properties of commercially produced aggregates by simply changing the feeding rate, the burner temperature, etc.

Results of the Los Angeles Abrasion Test made on synthetic aggregates produced in commercial kilns are given in Table 5-5. All of these aggregates appear suitable for use in flexible base in accordance with the criteria set out in "A Recommended Synthetic Coarse Aggregate Classification System," a special report submitted to the Texas Highway Department, for review purposes only (10).

TABLE 5.5. RESULTS OF LOS ANGELES ABRASION TEST ON SYNTHETIC AGGREGATES PRODUCED IN COMMERCIAL KILNS*

Aggregate**	Grading	Percent Wear
Hopkins	c	35.0
Wharton	c	43.6
Madison 1	c	33.3
Madison 2	c	38.3
R	b	27.5
S	c	22.5
C	b	40.4
E	b	25.2
D	b	23.1

*Values in table are based on a standard Los Angeles Abrasion test (ASTM C-131) made on one sample (5000 gms) of each aggregate.

**The Hopkins and Wharton aggregates have been used in flexible base on Texas highways, the two Madison aggregates have been investigated for possible similar use in the future, and the remaining five aggregates are samples of lightweight aggregates that are available commercially in Texas.

6. Conclusions

Listed below are the more significant findings which were reached as a result of the investigation described in this report. Because it was a rather limited investigation, they should be considered preliminary and subject to change as future research indicates.

1. When clay minerals are completely dehydrated, they become chemically stabilized for use as highway construction materials. This is accomplished by heating the clay to a temperature somewhere between 1000 and 1400°F. depending on the clay, and holding it at the elevated temperature for sufficient time to allow the

dehydration to occur. A period of 15 minutes at 1400°F. was sufficient to completely dehydrate the clay present in the small laboratory specimens made from the Texas soils investigated.

2. Incomplete dehydration of clay aggregates can be detected by a relatively simple laboratory test. The test procedure is given in Appendix 8.1.1.

3. Most (if not all) clay-type soils having a relatively high strength when air dried can be burned to produce hard, durable aggregates which are suitable for use in flexible base and asphaltic concrete.

7. References

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8. Appendix

8.1. LABORATORY PROCEDURES

8.1.1. Slaking Test

The procedure for the Slaking Test, a test utilized in this study to evaluate aggregates that have been produced by a synthetic burning process, is divided into two parts. The first is designed to evaluate laboratory produced aggregates (aggregates fired in a muffle furnace) and the second is designed to evaluate irregular-shaped aggregates of various sizes (aggregates produced in rotary kilns).

PART I—THE TESTING OF LABORATORY PRODUCED AGGREGATES

1. Select laboratory aggregates fired at temperatures of 600, 1000, 1200, 1400, 1600, 1800, and 2000°F.
2. Place the fired aggregates in separate, clean, 200 ml. beakers. Add 50 ml. distilled water to each beaker.
3. Clean and rinse the pressure cooker³ with distilled water before testing to insure removal of all foreign matter.
4. Place approximately 1/2" distilled water in the bottom of the pressure cooker, place the beakers containing the aggregates in it, and seal the lid tightly.
5. Heat the pressure cooker with a large Bunsen burner until full pressure is indicated by the pressure regulator.
6. Adjust flame to allow only a slight escape of steam and maintain pressure for 15 minutes. Remove the Bunsen burner, release the pressure, and remove the beakers.

³The pressure cooker consisted of a common kitchen-type, stainless steel, 6 qt. pressure cooker with a 15 psi pressure regulator (pressure relief valve).

7. The results of the test are determined by the visual appearance of the aggregates when they have cooled sufficiently to be removed (for example, see Table 4-4).

PART II—TESTING KILN PRODUCED AGGREGATES

1. Select a representative 600 gm. sample of the material to be tested. Wash the sample on a No. 40 mesh sieve to remove any fines and dry sample at 212-230°F. to a constant weight.
2. Determine the gradation after oven drying (Mechanical Analysis of Aggregates, Tex 110-E) (7).
3. Recombine the sample, place it in a 1200 ml. beaker, and cover the sample with distilled water.
4. Follow the cooking procedure outlined in Part I, Items 4 through 6.
5. Upon removing the sample from the pressure cooker, wash it over a No. 40 sieve, taking care not to lose any material.
6. Dry both fractions to a constant weight in a 212-230°F. oven, first decanting excess water in the fine fraction, provided this can be done without losing any material.
7. Determine gradation of material retained on the No. 40 sieve using same procedure followed in Item 2 except that the weight of the -40 materials is included in the total weight of the sample.⁴
8. Results are expressed as changes in gradation (for example, see Table 4-5).

⁴Due to rehydration the total weight of the sample may be greater than the initial weight.

8.1.2 Sodium Hydroxide Test

The sodium hydroxide test was utilized in this study as an indicator of the chemical stability of both laboratory and rotary kiln produced synthetic aggregates. The test is divided into two parts: the first to evaluate laboratory aggregates, the second to evaluate rotary kiln produced aggregates.

PART I—THE TESTING OF LABORATORY PRODUCED AGGREGATES

1. Select laboratory aggregates fired at temperatures of 600, 1000, 1200, 1400, 1600, 1800, and 2000°F.
2. Place the fired aggregates in separate, clean, 200 ml. beakers. Add 50 ml. of 5-N NaOH solution to each beaker.
3. Clean and rinse the pressure cooker⁵ with distilled water before testing to insure removal of all foreign matter.
4. Place approximately ½" distilled water in the bottom of the pressure cooker, place the beakers containing the aggregates in it, and seal the lid tightly.
5. Heat the pressure cooker with a large Bunsen burner until full pressure is indicated by the pressure regulator.
6. Adjust flame to allow only a slight escape of steam and maintain pressure for 15 minutes. Remove the Bunsen burner, release the pressure, and remove the beakers.
7. The results of the test are determined by the visual appearance of the aggregates when they have cooled sufficiently to be removed (for example, see Table 4-1).

PART II—TESTING KILN PRODUCED AGGREGATES

1. Select a representative 600 gm. sample of the material to be tested. Wash the sample on a No. 40 mesh sieve to remove any fines and dry sample at 212-230°F. to a constant weight.
2. Determine the gradation after oven drying (Mechanical Analysis of Aggregate, Tex 110-E).
3. Recombine the sample, place it in a 1200 ml. beaker, and cover the sample with 550-600 ml. of 5-N NaOH solution.
4. Follow the cooking procedure outlined in Part I, Items 4 through 6.
5. Upon removing the sample from the pressure cooker, decant the solution through filter paper. Wash the sample twice with distilled water. (The wash water is also decanted through the filter paper.)
6. Recombine any material retained on the filter paper with the contents of the beaker.
7. Dry the sample at 212-230°F. to a constant weight.
8. Determine the gradation of the sample using the procedure followed in Item 2.
9. Results are expressed as changes in gradation (for example, see Table 4-3).

⁵The pressure cooker consisted of a common kitchen-type, stainless steel, 6 qt. pressure cooker with a 15 psi pressure regulator (pressure relief valve).

8.1.3 Abrasion Test

This test was designed to indicate the ability of aggregates to resist abrasive wear. It was designed for use on laboratory produced aggregates (cylindrical specimens having a diameter of approximately ½") and ½" diameter cores of any other aggregates.

The procedure for performing the test is as follows:

I. Preparation of the test specimen:

1. Saw off both ends of the specimen to be tested with parallel diamond blades so that the specimen is a right circular cylinder approximately 1" long.
2. Measure and record its diameter and length to the nearest 0.001." (Use the average of three measurements.)

II. Preparation of the abrasive device (Figure 8.1-1)

1. Dress the face of the abrasive wheel using a stone dresser placed in "V" trough A⁶ and held in place by clamp B. Set the abrasive wheel in motion and move the stone dresser across the face of the wheel by turning thumb screw F. Several passes across the wheel may be required to obtain a flat surface.
2. Adjust the "V" trough so that its bottom is 2¼" out from the center of the abrasive wheel.
3. Adjust the flexible water tubing so that a stream of water strikes the stone approximately 1" above the bottom of the "V" trough.
4. Install a standard laboratory timer in the electrical circuit so that it turns off the motor 15 seconds after it is started. Place it on stand-by.
5. Place the air cylinder assembly C into the "V" trough and clamp it in place with clamp B. Install an external regulated air pressure supply (0-30 psi) onto nipple D and leave set at 0 psi.

III. Performance of the test:

1. Place the prepared specimen in the "V" trough between the abrasive wheel and the air cylinder assembly.
2. Adjust the pressure regulator so that the specimen is held between the abrasive wheel and the air cylinder piston G. The pressure is to be adjusted so that the average stress on the face of the specimen is 50 psi. (Take into account the diameter of the specimen, diameter of the piston, and the pressure acting on it.)
3. Turn on water supply valve E and make any final adjustments in the position of the water stream.
4. Turn the timer on. It will cut the motor off after 15 seconds.
5. After the motor stops, turn off the water supply and the air pressure. Remove the specimen.
6. Measure and record the length of the specimen to the nearest 0.001." (Use average of three measurements.)
7. Results are expressed as the length loss due to abrasion (initial length minus final length); a small length loss indicates high abrasion resistance.

⁶Letters designate components of abrasion device shown in Figure 8.1-1.

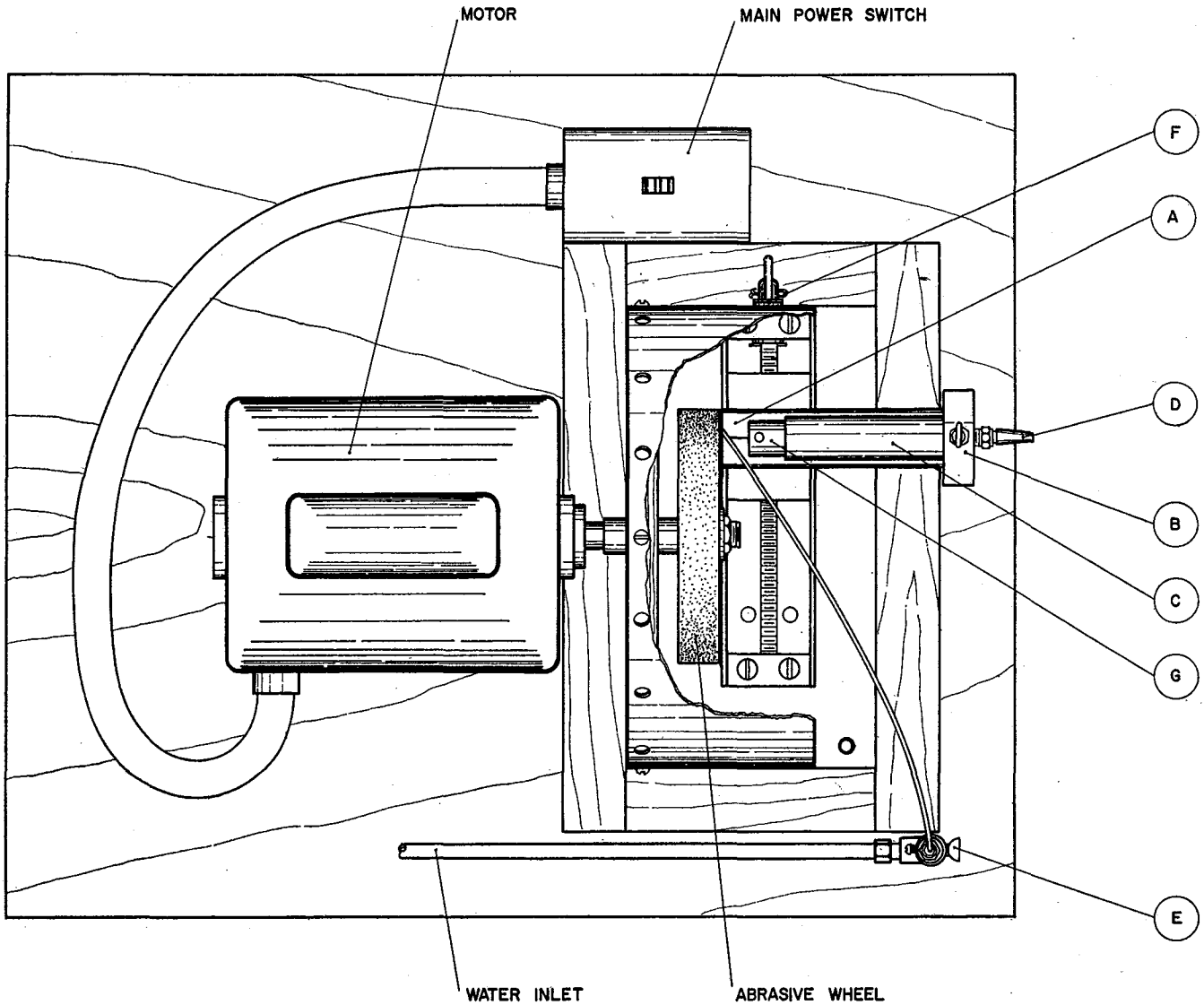


Figure 8.1-1. Top view of abrasion device.

8.2. DIFFERENTIAL THERMAL PATTERNS FOR SOILS USED IN THIS STUDY

The differential thermal patterns of 18 soils were investigated in this study. Patterns for five of them (the four used in commercial kilns to produce flexible base aggregates and the Na-montmorillonite) are included in this appendix. These patterns are typical of the results. There was no particular thermal property except dehydroxylation that characterized the samples that produced acceptable aggregates.

The heating rate used was 12°C. per minute. The material tested was a representative powdered sample (-260 micron) mixed with Al_2O_3 in the ratio of one part soil to three parts Al_2O_3 . The reference sample was Al_2O_3 .

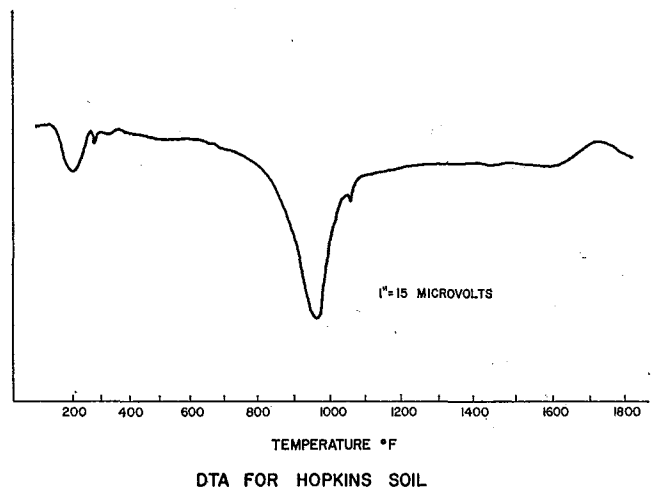
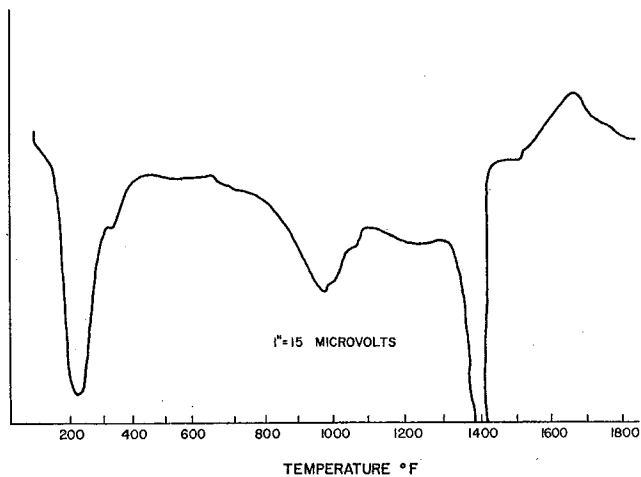
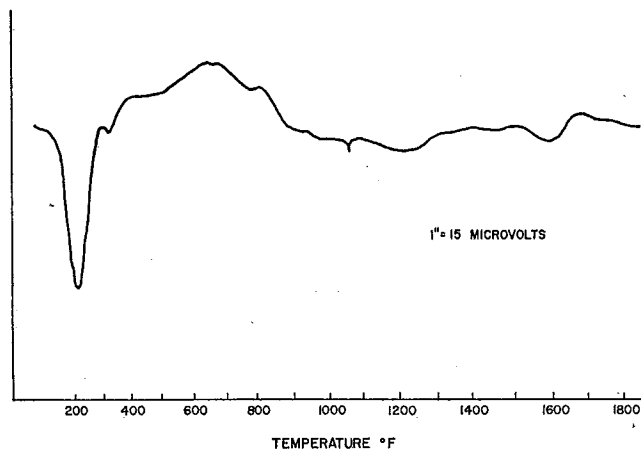


Figure 8.2-1.



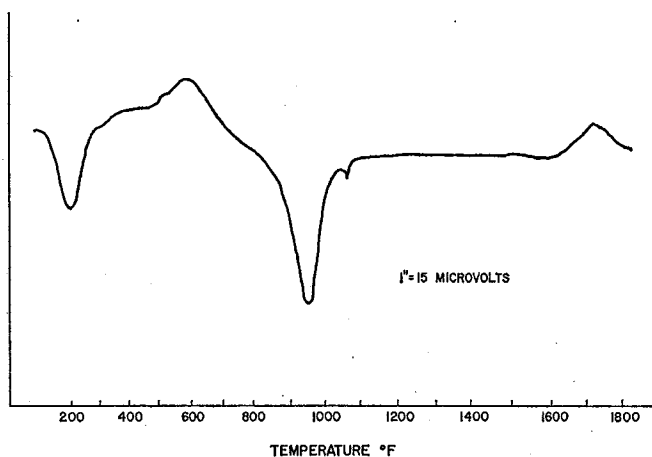
DTA FOR WHARTON SOIL

Figure 8.2-2.



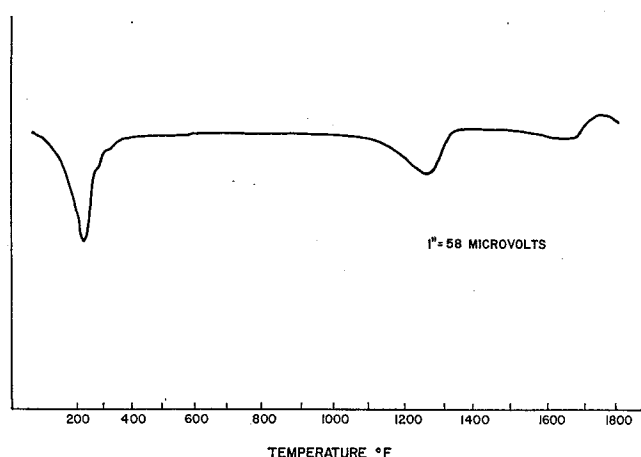
DTA FOR MADISON 2 SOIL

Figure 8.2-4.



DTA FOR MADISON 1 SOIL

Figure 8.2-3.

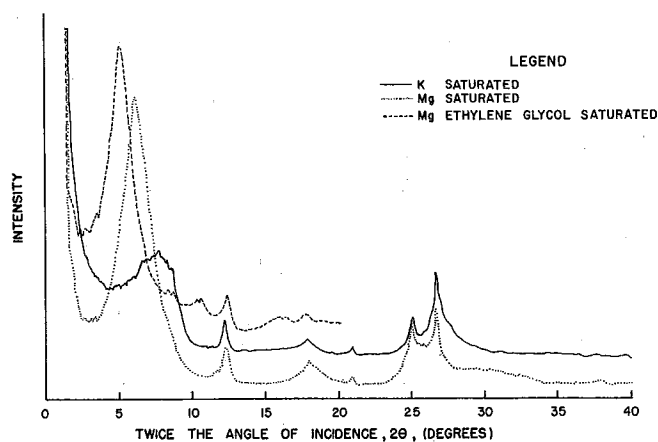


DTA FOR Na-MONT

Figure 8.2-5.

8.3. X-RAY DIFFRACTION PATTERNS FOR SOILS USED IN THIS STUDY

In order to more accurately define the soils investigated, X-ray diffraction patterns for their clay fractions were obtained.⁷ The patterns showed that the clay fractions of these soils were composed of about 90 to 100 percent of three clay minerals (montmorillonite, illite, kaolinite) in various combinations. Patterns for the four soils used in commercial kilns to produce flexible base aggregates are included in this appendix. These patterns are typical of the results.



X-RAY DIFFRACTION FOR HOPKINS SOIL

Figure 8.3-1.

⁷Due to the fineness and purity of the Na-montmorillonite, the X-ray diffraction data were obtained from the raw, untreated sample.

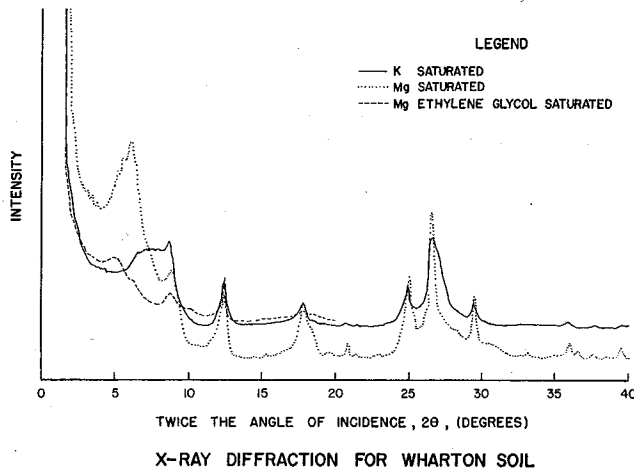


Figure 8.3-2.

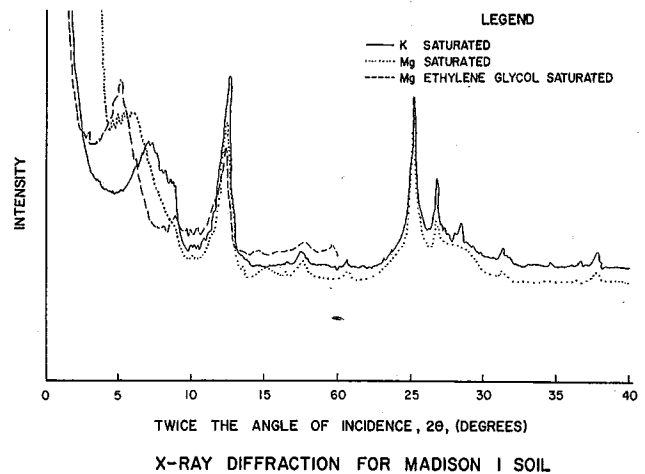


Figure 8.3-3.

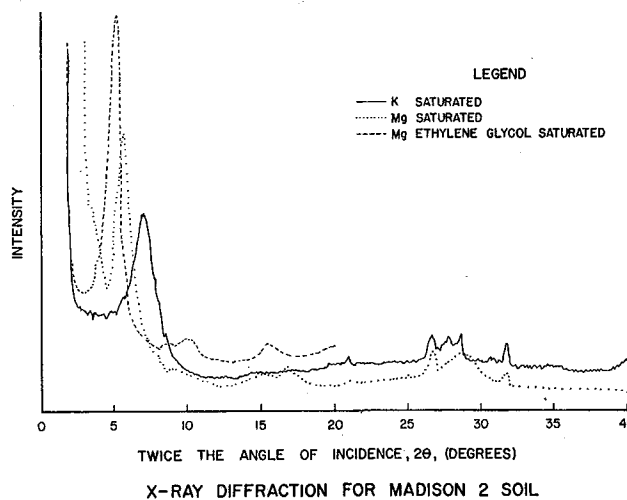


Figure 8.3-4.

8.4. RELIABILITY OF PHYSICAL TESTS MADE ON LABORATORY SPECIMENS

Because of the large number of specimens required for the chemical and the physical tests performed on the laboratory produced specimens, it was desirable to test single laboratory specimens. Reported here are the results of a special experiment conducted to determine the reliability of the single test value used in physical stability evaluation.

Groups of three replicate specimens of laboratory fired aggregates were considered as a treatment and were subjected to physical testing. The results of these tests are shown in Tables 8.4-1 through 8.4-3. Graphs of the standard deviation of each treatment versus the treatment mean are shown for the three physical tests in Figure 8.4-1. The "within treatment" standard deviation and the "within treatment" coefficient of variation, determined from a standard analysis of variance, are also plotted on these graphs. As evidenced from the graphs, the standard deviation of a treatment appears to be gen-

erally proportional to the treatment mean. Thus the standard deviation should not be considered as a constant value, but rather as a percentage of the treatment mean. In this case an estimate (which is believed to be conservative) for the standard deviation "within treatments" is the coefficient of variation. With this assumption, the result of a single test will be within the coefficient of variation from the true treatment mean (or the average value obtained from a large number of tests) approximately 67% of the time. In the treatments subjected to the compression tests, 20 out of 24 (or 70%) had standard deviations of less than 23% of the treatment mean. In the treatments subjected to the Rockwell hardness test, 11 out of 15 (or 73%) had standard deviations of less than 15% of the treatment mean. And, in the treatments subjected to the abrasion tests, 10 out of 14 (or 71%) had standard deviations of less than 30% of the treatment mean.

TABLE 8.4-1. SPECIAL EXPERIMENT TO DETERMINE THE RELIABILITY OF THE COMPRESSIVE STRENGTH MEASURED BY SINGLE TEST

Treat. No.	Comp. Str. psi	Mean Value	Std. Dev.	Treat. No.	Comp. Str. psi	Mean Value	Std. Dev.
1	2000	2100	110	11	6890	7920	1710
	2080				9890		
	2220				6980		
2	6140	5390	660	12	9800	9590	1340
	5120				10810		
	4910				8150		
3	9500	8710	960	13	1290	1950	660
	7650				2610		
	8990				1960		
4	7680	7480	710	14	3460	3340	270
	8060				3520		
	6690				3030		
5	9840	9240	2890	15	3000	3380	400
	6010				3800		
	11880				3340		
6	5500	6180	3060	16	3440	3910	930
	3520				3300		
	9530				4980		
7	2100	2250	180	17	8160	5600	2230
	2200				4550		
	2440				4100		
8	3640	3860	320	18	6320	6540	600
	3710				6080		
	4230				7220		
9	7550	7590	430	19	4800	5580	720
	7180				6220		
	8030				5710		
10	9080	8760	310	20	6270	6800	1780
	8740				8780		
	8460				5350		

Analysis of Variance:
 Within Treatment Standard Deviation—1340.
 Within Treatment Coefficient of Variation—23.0%.

TABLE 8.4-2. SPECIAL EXPERIMENT TO DETERMINE THE RELIABILITY OF THE ROCKWELL HARDNESS MEASURED BY SINGLE TEST

Treat. No.	Rockwell Hardness	Mean Value	Std. Dev.	Treat. No.	Rockwell Hardness	Mean Value	Std. Dev.
21	30	29.7	2.5	28	72	66.3	5.5
	27				66		
	32				61		
22	13	28.0	13.4	29	53	50.3	3.1
	39				47		
	32				51		
23	61	68.7	6.8	30	72	66.3	7.4
	74				58		
	71				69		
24	82	82.0	1.0	31	23	20.7	3.2
	83				22		
	81				17		
25	83	67.0	16.5	32	28	26.7	2.3
	50				28		
	68				24		
26	28	25.7	4.9	33	56	61.3	4.7
	20				63		
	29				65		
27	62	61.3	7.0	34	70	71.7	10.6
	54				83		
	68				62		
				35	0	0.0	0.0
					0		

Analysis of Variance:
 Within Treatment Standard Deviation—7.4.
 Within Treatment Coefficient of Variation—15.3%.

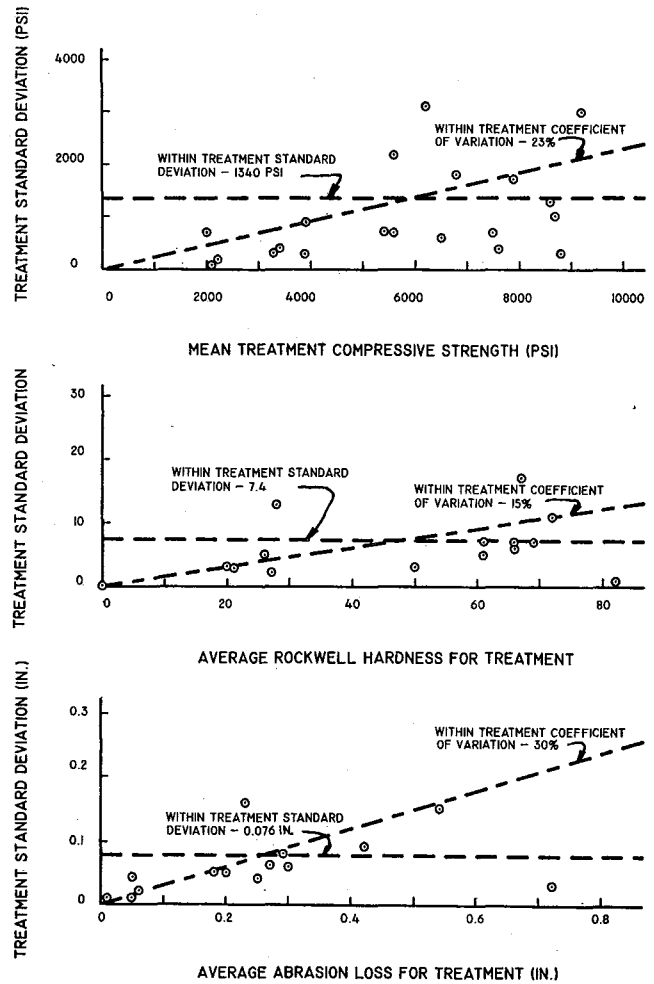


Figure 8.4-1. Mean of three replicate test measurements versus standard deviation from the mean.

TABLE 8.4-3. SPECIAL EXPERIMENT TO DETERMINE THE RELIABILITY OF THE ABRASION LOSS MEASURED BY SINGLE TEST

Treat. No.	Abrasion Loss (in.)	Mean Value	Std. Dev.	Treat. No.	Abrasion Loss (in.)	Mean Value	Std. Dev.
36	0.424	0.416	0.093	43	0.351	0.300	0.057
	0.505				0.309		
	0.319				0.239		
37	0.197	0.178	0.053	44	0.023	0.013	0.009
	0.219				0.008		
	0.118				0.007		
38	0.055	0.046	0.009	45	0.746	0.716	0.031
	0.046				0.718		
	0.038				0.685		
39	0.083	0.058	0.022	46	0.374	0.540	0.150
	0.041				0.665		
	0.050				0.581		
40	0.080	0.051	0.041	47	0.202	0.286	0.085
	0.004				0.371		
	0.070				0.286		
41	0.121	0.229	0.159	48	0.252	0.197	0.048
	0.155				0.164		
	0.411				0.176		
42	0.216	0.247	0.040	49	0.340	0.268	0.063
	0.292				0.226		
	0.232				0.237		

Analysis of Variance:
 Within Treatment Standard Deviation—0.076.
 Within Treatment Coefficient of Variation—30.0%.