Bituminous Stabilization of Wyoming Heat-Altered Shale

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Wyoming has future highway projects that will pass through areas lacking first-class aggregates, but containing a quantity of unproven heat-altered shale aggregate. Considering the possible transportation savings if the shale were to be used, research was undertaken to explore the possibility of improving the utility of asphalt as a stabilizer for heat-altered shale by the use of trace quantities of chemical additives known to react with asphalt, or with soil-mineral surfaces. Resulting Marshall design control methods (ASTM D 1559-58T) and effective weathering tests were used to evaluate the materials used.

Various asphalts and/or additives were well above the minimum design criteria for roads receiving heavy traffic. Thus, constructing roads of heat-altered shale would be justified.

● KNOWN SOURCES of local quality aggregates for road building purposes are being rapidly depleted throughout the nation. Areas in the northeastern section of Wyoming are deficient in aggregate materials of proven quality. Future highway construction projects in these areas may require transporting sizable quantities of gravel aggregates for distances as great as 70 miles. However, large quantities of a heat-altered shale are available in the vicinity near highway locations.

Field problem investigation indicated these shales may provide satisfactory road building materials. Heat-altered shale has been used as a patching material, but has not been incorporated as a pavement aggregate. The performance of shale as a patching material shows that it compares favorably with gravel aggregates as a surface course.

Laboratory research, supported by a grant from the Wyoming Highway Department, was conducted to determine the possibilities of improving the utility of asphalt as a stabilizer for Wyoming heat-altered shale. Trace quantities of chemical additives known to react with asphalt and/or with soil mineral surfaces were incorporated into the investigation.

If these shales could be shown to produce satisfactory stability for the subbase, base, and pavement, future construction with this material would result in a tremendous savings.

DESCRIPTION OF MATERIALS

To determine the stability of heat-altered shale, laboratory work was conducted in the bituminous-soils laboratory at the University of Wyoming. The materials used in the testing procedure included shale aggregate; asphalt cements of various penetrations; non-organic and organic additives.

Aggregate

Mineralogical Classification and Properties. — The baked shale originally was a sedimentary bed. Stratigraphically below these sea beds was a large vein of coal. At one time, portions of this coal bed burned; the heat and escaping gases affected the sedimentary beds above to produce heat-altered shale. Evidence of the coal seam, about 100 ft thick, is seen at the WyoDak coal mine (Fig. 1).

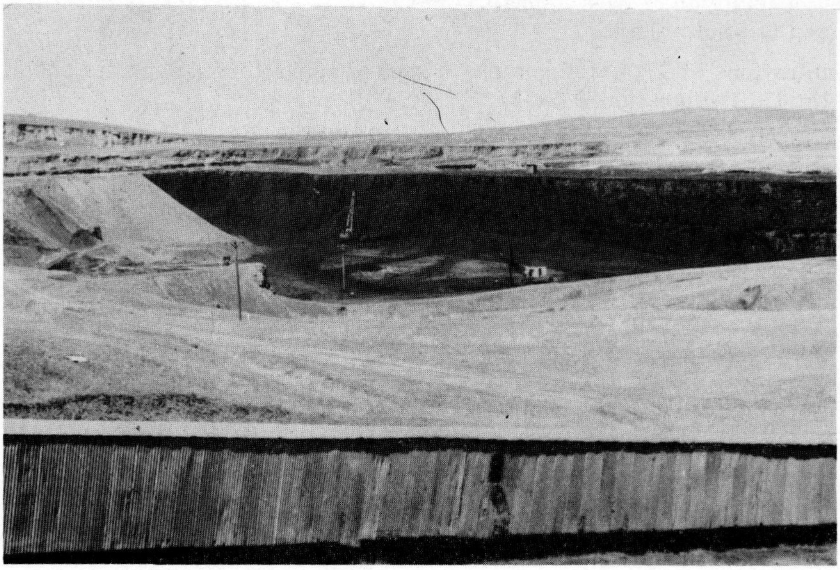

Figure 1. Coal seam at Wyo Dak.

Baked shale is variable in quality because of the manner in which it was formed. Some of the formation received extremely high temperatures, especially the rock centered near an escaping gas vent. Harder clinkers in the formation were formed by the higher temperatures. Heat-altered shale has a natural affinity for water and, as a result, has poor resistance to prolonged atmospheric exposure. Figure 2 shows the variability in the weathering of the heat-altered shale. The center of the picture illustrates slaking of a soft baked shale after prolonged exposure to the elements, but the shales shown on the sides of the picture which have been in the area for the same length of time have not weathered very much.

Gradation. — The aggregate used in this investigation was representative heat-altered shale. Samples were obtained from a pit located 1 mi south of WyoDak coal mine and 6 mi east of Gillette, Wyoming. The shale, taken from the pit deposit in irregular sized slab-like clinkers, was laminated in texture. After crushing, the aggregate particles were flat, elongated chips.

Wyoming Highway Specifications (1956) and the Asphalt Institute Specifications (1956) were followed as gradation analysis standards (Fig. 3). The percentage passing the No. 4 sieve somewhat exceeds the specifications. Wyoming specifications allow a 6 percent deviation on the gradation uniformity passing the No. 4 sieve during a day of crushing.

Engineering Classification. — Figure 4 is a micro-photograph of a thin slice of baked shale. The white or light-textured material is quartz or SiO_2, showing the high percentage of SiO_2 as was found by a chemical analysis of shale (Table 1). Table 2 contains the engineering classification of the aggregate.

Asphalts and Additives

The asphalt cements used in the testing program were Husky AC 150 pen., and Husky Rubberized AC 160 pen., and Socony Mobil 85-100 pen. Laboratory tests on the asphalt cements are compiled in Table 3.

Non-organic additives used in the combination of the asphalt-aggregate system were anhydrous lime and concentrated phosphoric acid. The organic additives incorporated in the testing of heat-altered shale for bituminous stabilization (added to Husky AC 150 or to the aggregate) were diamine No. 21 (N-coco trimethylene diamine) and Alamine No. 26 (primary tallow amine). Characteristics of the amines are given in Table 4.

Further description of base asphalt cements and asphalt cements containing additives (summarized in Table 5) are:

1. Penetrations at 77 F (100 grams, 5 sec) of asphalts and asphalts containing additives (ASTM Designation D 5-52).

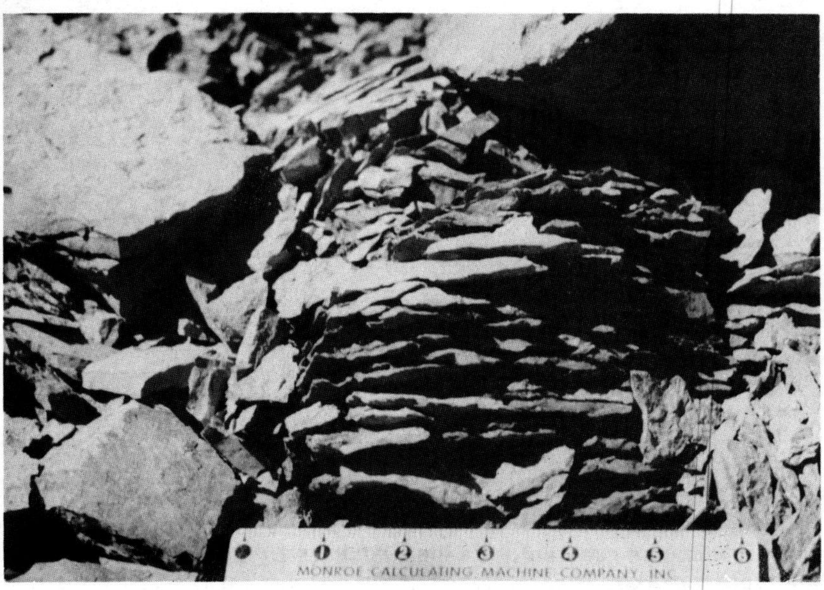

Figure 2. Slaked shale after prolonged exposure to the atmosphere.

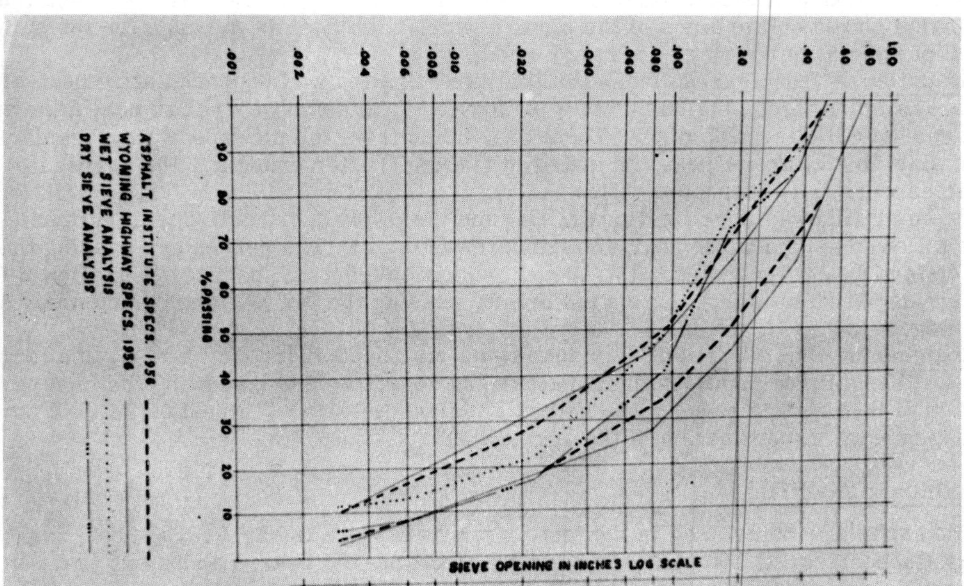

Figure 3. Gradation analysis.

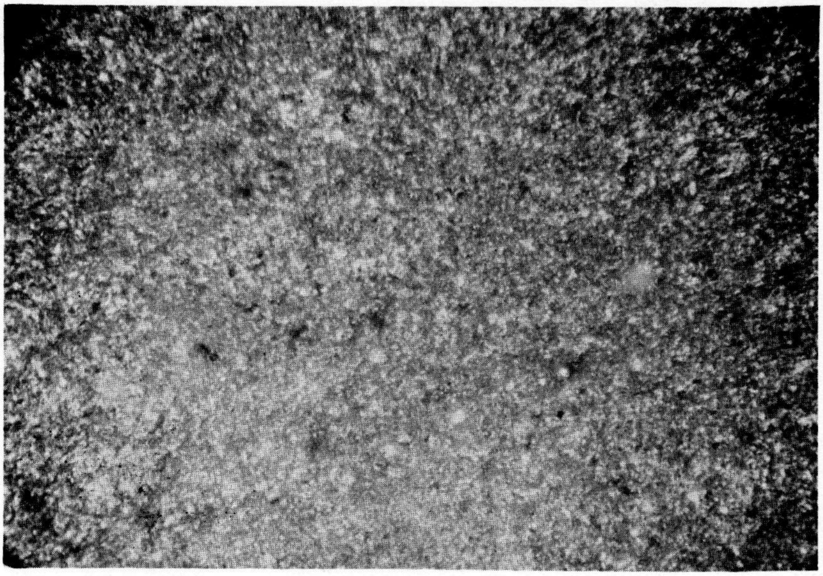

Figure 4. Photograph of a thin slice of heat-altered shale taken through a microscope. The light textured material is quartz.

2. Specific gravity at 77/77 F of asphalts and asphalts containing additives (ASTM Designation D 70-52).

3. Contact angles formed between drops of various mediums and a glass plate surface.

The penetration and specific gravity are standard qualitative tests; the measurement of contact angles has been used by others "....as a measure of the suitability of materials for bituminous stabilization" and as an evaluation of the effect of pretreatment of soil and (or) asphalt.

TABLE 1

CHEMICAL ANALYSIS OF WYOMING HEAT-ALTERED SHALE

Constituents	Percent by Weight
L.O.I.	0.78
SiO_2	68.0
Al_2O_3	15.3
Fe_2O_3	5.88
TiO_2	0.62
P_2O_5	0.20
MnO	0.06
CaO	3.23
MgO	2.09
Na_2O	0.26
K_2O	3.01
Li_2O	none
Summation	99.40%

The contact angles were determined from dimensions obtained from an enlarged profile photograph of small uniform liquid drops resting upon a horizontal glass plate (Fig. 5). These figures, applied to calculations and tables by Mack and Lee, gave the resulting contact angles summarized in Table 5.

The smaller contact angles indicate increased spreading or dispersion ability of the liquid. Thus better dispersion of asphalt should result in improved waterproofness and consequent retention of stability when wet. However, the ease of coating is but one factor that need be considered in evaluating the effect of admixes.

The admixes have more effect on the physical properties and stability of compression specimens after immersion than is demonstrated by their effect on the contact angle.

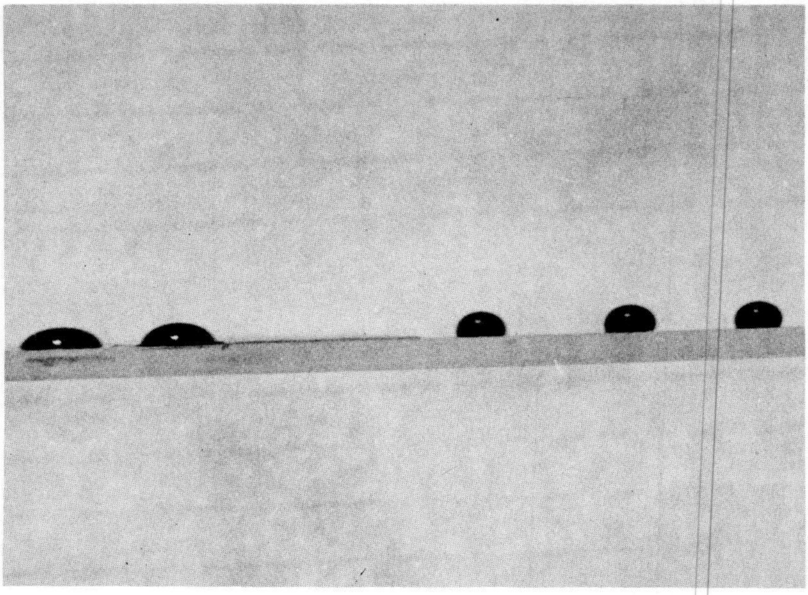

Figure 5. Silhouette photograph showing shape of asphaltic droplet for contact angle measurements (left: rubber AC 160, angle of contact 52.5°; right: AC containing phosphoric acid, angle of contact 98°).

TABLE 2

ENGINEERING CLASSIFICATIONS OF HEAT-ALTERED SHALE

Determination	Value	ASTM Desig.
Liquid limit, -40 (%)	30.3	D423-54T
Plastic limit, -80 (%)	none	D424-54T
Plasticity index (%)	none	D424-54T
Spec. gr., (evacuation)	2.50	C128-42
Unit wt., dry rodded (pcf)	88.3	C29-55T
Voids vol., dry rodded (%)	0.425	C30-37
pH	7.0	

INVESTIGATION

The stability of heat-altered shale in combination with asphalt and chemical additives was investigated by the following methods:

1. Modified Marshall method — for design and control of paving mixtures (ASTM Designation: D 1559-58T).
2. Relative test — for effect of water on cohesion of compacted bituminous mixtures.
3. Evaluation of mixtures — made by subjecting specimens to accelerated weathering tests and comparing respective stabilities.

Previously, others had achieved successful use of 2 and 4 percent of the additives used in this investigation for improving asphalt as a stabilizer. These percentages served as a guide for this study.

TABLE 3

LABORATORY TESTS ON ASPHALT CEMENTS FOR THE BITUMINOUS STABILIZATION OF HEAT-ALTERED SHALE

Material	Husky 150 A.C.	Husky 160 A.C. Rubberized	Socony 85-100 A.C.
Soft. point, F	105	–	114
Pen. at 77 F (100 g, 5 sec)	150	160	95
Pen. at 32 F (200 g, 60 sec)	31	–	–
Pen. at 115 F (50 g, 5 sec)	T.S.	T.S.	–
Duct. at 77 F (5 cm/min)	100+	150+	196+
Duct. at 39.2 F (5 cm/min)	40	150+	–
Flash, deg F, C.O.C.	535	535	540
Vis. at 325 F, s.f.s.	41	112	95
Loss on heat, 325 F, 5 hr, %	0.03	0.03	0.06
Pen. after L.O.H.% of orig.	84	84	85
Spot test	Neg	Neg	Neg
Sol in CS_2, %	99.8+	99.0+	–
Sol in CCl_4, %	99.8	99.0	99.89
Water, %	none	none	–
Specific gravity at 60 F/60 F	1.029	1.029	1.0105 at 77
Float at 122 F, sec.	–	–	–
Res of 100 pen.	–	–	–

TABLE 4

AMINE SPECIFICATIONS AND PHYSICAL PROPERTIES (6)

Product		Melting Point			Water Content	Cost
Name	Formula	(°C)	Sp. Gr.	Amine Number	(%)	($/lb)
Diamine No. 21	(N-Coco trimethylene diamine) $RCH_2NH(CH_2)_3NH_2$	20-25	–	375 min	2	0.63
Alamine No. 26	(primary tallow amine) RCH_2NH_2 or $(RCH_2)_2NH$	35	0.85	192 min	–	0.33

Modified Marshall Test and Relative Test

A total of 355 Marshall test specimens with an asphaltic range from 9 to 15 percent, and an additive range from 0 to 4 percent were prepared. The specimens were compacted in the specified manner (that is, proper temperature, mix time and proportions; 10-lb hammer drop of 18 in. and 50 blows to each side of the sample).

However, the Marshall method was modified in the following manner: Four representative specimens of each mixture were compacted for the modified Marshall stability tests. Following mass and dimension measurements, the samples were placed in a constant temperature oven (140 F) for four days. Then, two of these specimens were completely immersed in a water bath (140 F) for 24 hr, while the other two samples remained in the dry oven. Immediately preceding the stability tests, the dry cured specimens were immersed in this water bath for 20 min.

Marshall test property curves (unit weight; Marshall stability; flow; percentage of voids — total mix; percentage of aggregate voids — filled) were plotted following measurements and testing. The optimum percentages of asphalt — 11 and 12 percent — were determined from these curves and are summarized in Figures 6 and 7. Large variations were observed between the strength of an untreated asphalt cured "dry" at 140 F for

TABLE 5

PENETRATIONS, SPECIFIC GRAVITIES, AND CONTACT ANGLES OF ADDITIVES IN ASPHALT MEDIUMS

Medium	Penetration[a]	Specific Gravity[b]	Average Contact Angle[c]
Husky Rubber AC160	160	1.028	52.5
Mobil AC85-100	95	1.010	56.0
Husky AC150	150	1.025	52.1
2% phosphoric acid in AC	112	1.028	98.0
4% phosphoric acid in AC	75	1.035	
2% diamine No. 21 in AC	Too soft	1.017	53.4
4% diamine No. 21 in AC	Too soft	1.014	
2% alamine No. 26 in AC	259	1.017	56.2
4% alamine No. 26 in AC	Too soft	1.009	
AC 150 on detergent	—	—	49.1
AC 150 on phosphoric acid	—	—	neg
AC 150 on alamine No. 26	—	—	48.6
AC 150 on diamine No. 21	—	—	46.8
Water on alamine No. 26	—	—	18.0
Water on detergent	—	—	neg

[a]Penetration at 77 F (100 grams, 5 sec) of asphalts and asphalts containing additives. (ASTM Designation D 5-52).
[b]Specific gravity at 77/77 F of asphalts and asphalts containing additives. (ASTM Designation D 70-520).
[c]Contact angles formed between drops of various mediums and a glass plate surface.

TABLE 6

ADDITIVES IN ASPHALT MEDIUMS AND INDICES OF RETAINED STRENGTH FROM EFFECT OF WATER

Additive and Medium	Index of Retained Strength
Mobil AC 85-100	68
Husky rubber AC 160	92
Husky AC 150	71
2% phosphoric acid on aggregate	61
2% phosphoric acid in AC	82
4% phosphoric acid in AC	0
2% diamine No. 21 on aggregate	96
2% diamine No. 21 in AC	101
4% diamine No. 21 in AC	100
2% alamine No. 26 on aggregate	68
2% alamine No. 26 in AC	101
4% alamine No. 26 in AC	81
2% lime on aggregate	98
4% lime on aggregate	65

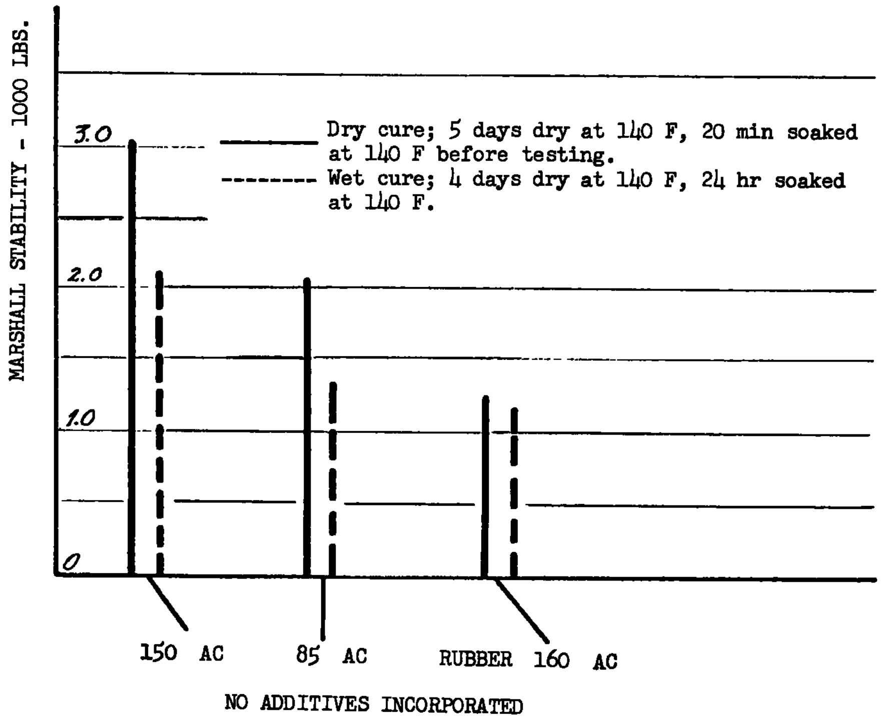

Figure 6a. Optimum Marshall stability for asphalts and for additives.

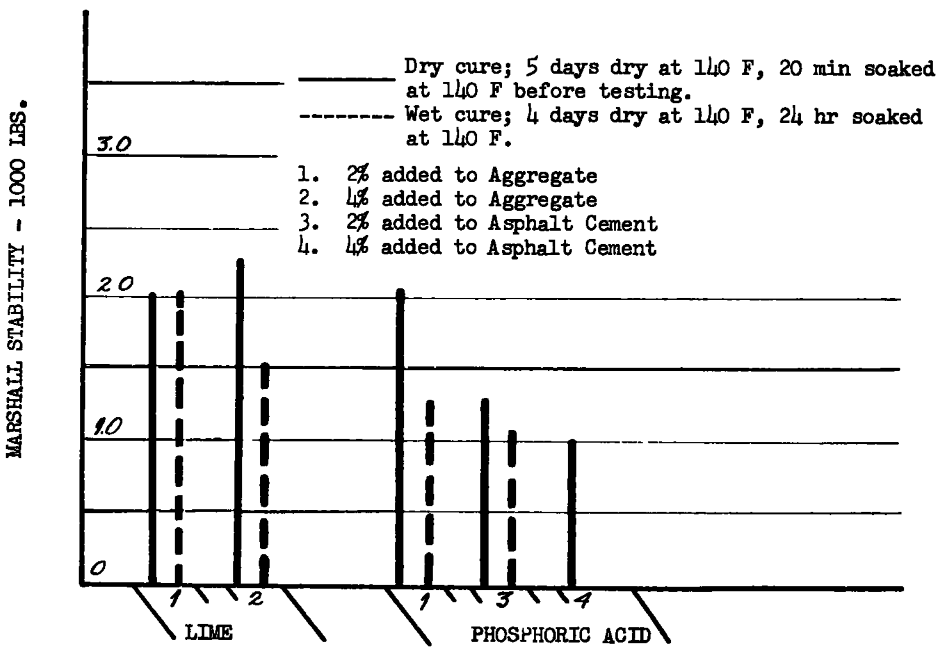

Figure 6b. Optimum Marshall stability for asphalts and for additives.

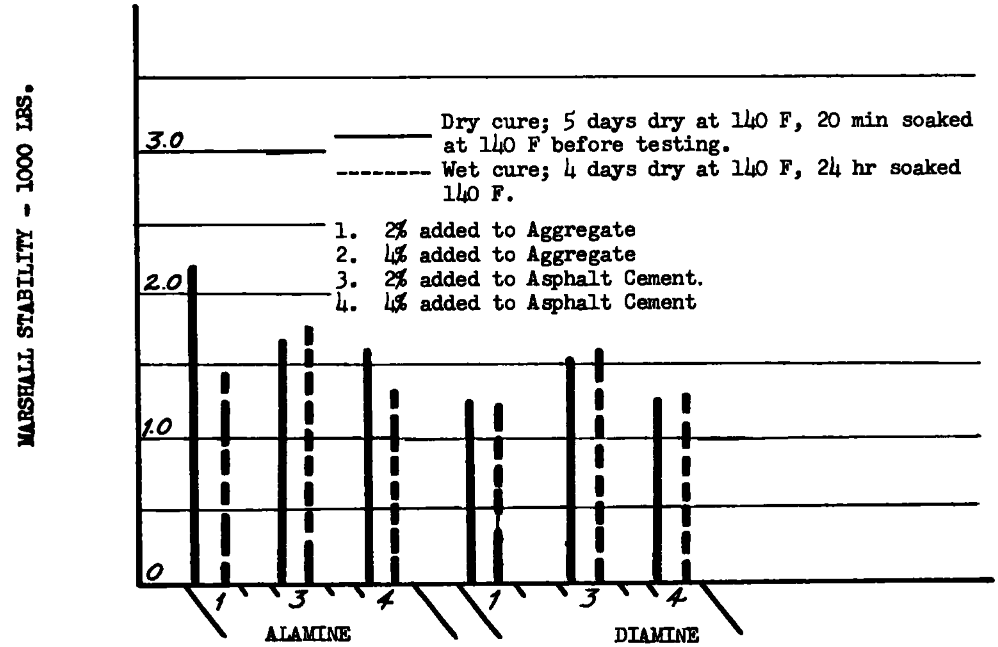

Figure 6c. Optimum Marshall stability for asphalts and for additives.

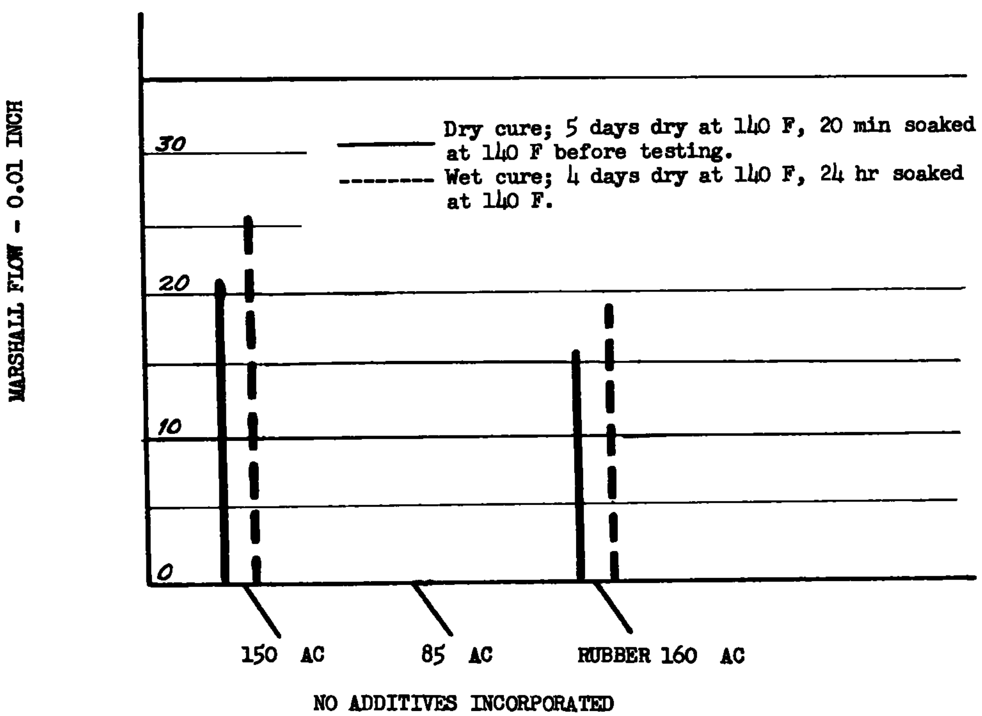

Figure 7a. Flow values for optimum Marshall stabilities shown in Figure 6.

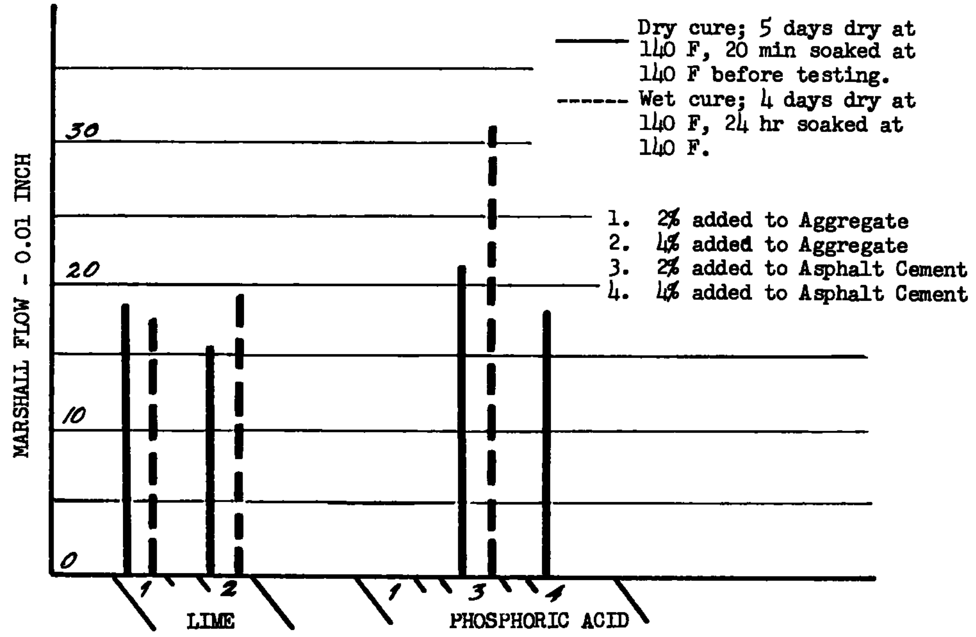

Figure 7b. Flow values for optimum Marshall stabilities shown in Figure 6.

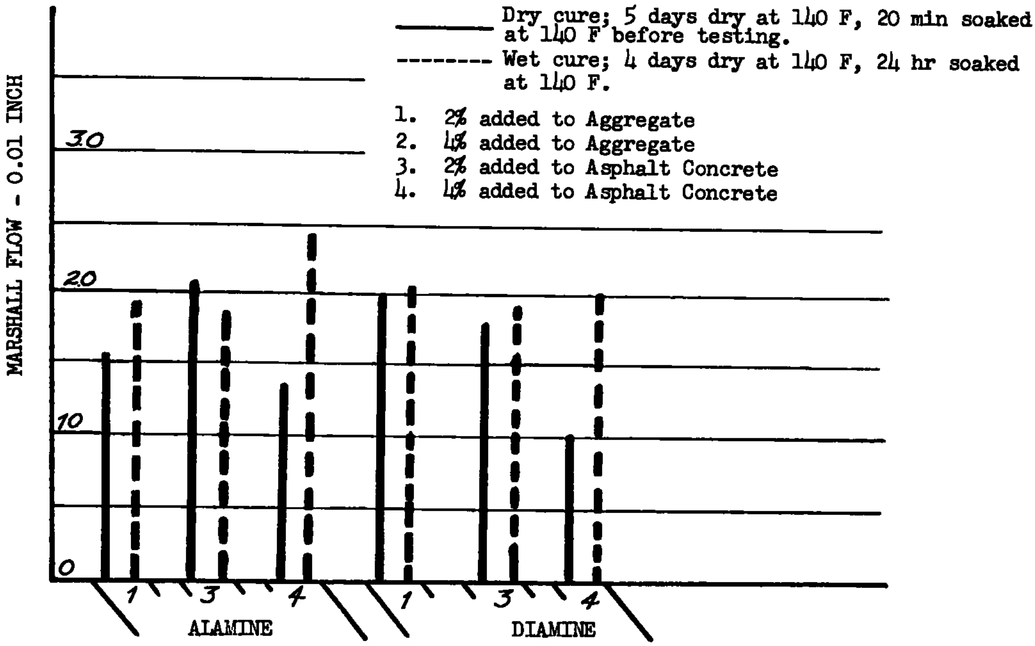

Figure 7c. Flow values for optimum Marshall stabilities shown in Figure 6.

five days and one cured "wet" at the same temperature. The Marshall stability values were lowered when the sample was soaked in water at a temperature of 140 F which tended to soften the asphalt. The indices in Table 6 were computed from the resulting values in Figure 6. This was considered the relative test for the effect of water on cohesion of compacted bituminous mixtures.

Weathering Test

A desirable characteristic of major importance to an asphalt stabilized aggregate is its ability to withstand exposure to weather. Marshall test specimens of asphalts with or without additives presenting favorable results in earlier investigations were subjected to duplicable accelerated weathering conditions.

A weathering test device designed for the testing of coal briquettes was used for the weathering of asphalt compression specimens. "The device itself supplies dry air, heat, ultraviolet radiation and wetting under any cycle desired. The effect of freezing was included by removing specimens, at a specified time each day, and placing them in a freezer at -10 F. Ultraviolet radiation was supplied from a type S-1 mercury lamp at a distance of 15 in. This is approximately nine times the intensity of a normal mid-day sun. The infrared radiation from the lamp, combined with the heat from a 500-watt heating coil, maintains a temperature of 150 F during the heating cycle. A blower circulates air at the rate of 42 cu ft per min, giving a complete change of air every 9 sec. The three atomizing sprayheads in the top of the chamber provide a fine mist of water which wets all specimens on the shelves."

The daily cycle for 28 days of simulated weathering, as followed in the study, was:

1. Twenty-hour heating period, employing ultraviolet radiation (S-1 mercury lamp at 15 in.) and hot air (150 F) circulated on specimens.

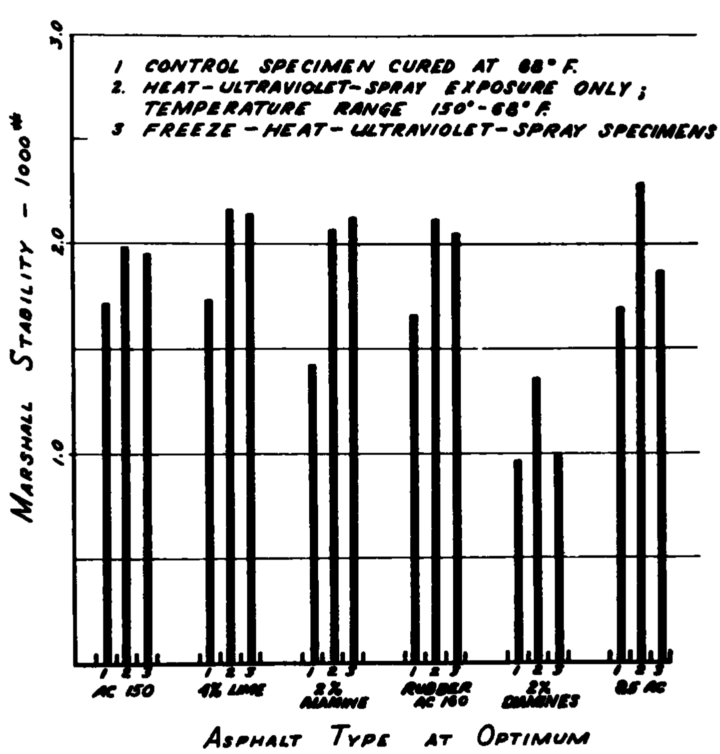

Figure 8. Weathering investigation, comparison of stabilities following 28 days of exposure.

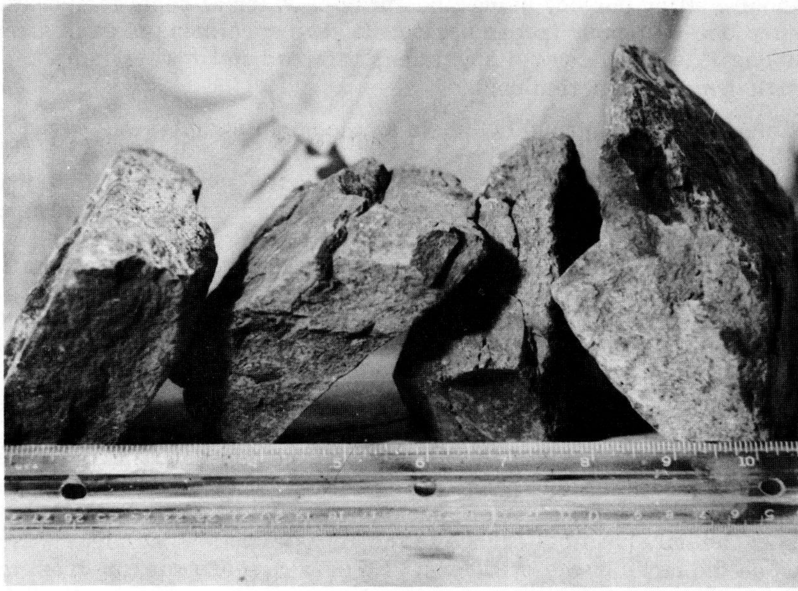

Figure 9. Heat-altered shale clinkers prior to subjection to the accelerated weathering exposure.

Figure 10. Clinkers following exposure to accelerated weathering test. The two pieces on the left were exposed for 28 days of alternating heat-spray-rest cycles. The samples on the right were subjected to spray-freeze-rest-heat cycles.

2. Two-hour wetting period, using fine spray of water at room temperature.
3. One- and one-half-hour freeze period: (a) two specimens of each sample subjected to -10 deg F, and (b) control specimen, retained under water spray.
4. One-half-hour acclimation period.

One specimen from each group (withheld as a control sample) was cured for 28 days at room temperature.

Figure 8 represents the Marshall stabilities when the specimens of various asphalts and admixes had been subjected to 28 days of accelerated weathering and soaked in the water bath (140 F) prior to compression.

A distinguishable difference is noted in each case between the control "air cured" specimen and specimens that actually received weathering exposure. The fact that some of the exposure specimens were also frozen as part of the test seemed to have little significant effect. The reader in comparing Figures 6a, 6b, 6c and Figure 8 will also note a distinct effect of the curing process on the stabilities. (Note especially Rubber AC 160 pen.)

It was interesting to compare the damage that occurred to large pieces of aggregate shale clinkers subjected to the same weathering as the asphaltic specimens (Figs. 9 and 10). The clinkers shown in Figure 10 had actually cleaved or broken apart, and thus for the extent of the test, the test appeared complete for the purposes of this investigation. The only physical change to the asphalt-shale specimens was a slight discoloration on the faces of the cylinders. No breakage or cracking from the weathering test cycles, either heat or freezing, was observed. Visual inspection after the weathering test was completed thus indicated that the asphalt-aggregate specimens held up better than the shale clinkers.

CONCLUSIONS

The modified Marshall curing period used, simulates conditions in the field. Immediately following compaction and a minimum curing time, traffic loads are allowed on a finished roadway. Thus, the short-time cured specimens closely represent the newly finished roadway. In contrast to this, the stabilities received from the long-time weather cured specimens indicate the expected strengths of a roadway over a period of months. The effective weathering test gives a qualified answer to the question: "What will the condition of the roadway be in a few years?"

The following conclusions have been derived from the data:

1. Considering the minimum design criteria for main streets and roads receiving heavy traffic (1,800 lb Marshall stability), highway construction would be justified by using asphalt cements 85-100 and AC 150 penetration without additives.

The 85-100 penetration asphalt cement was used as a control asphalt. Resulting test values were for 12 percent AC: 2,110 lb dry, 1,310 lb wet, for the short cure and 2,560 lb weathered.

The resistance to the accelerated weathering and the high stabilities for AC 150 penetration warrants the use of this asphalt with heat-altered shale as an aggregate in highway construction (12 percent 150 AC pen. — 3,000 lb dry, 2,125 lb wet, and 1,900 lb weathered).

2. The addition of lime to the baked shale in small percentages gave a high index of retained strength. Under the short cure time used for the Marshall test, the stabilities did not exceed the compressive strength of the heat-altered shale with normal 150 penetration asphalt cement; however, the values were above the 1,800 lb — designated as the minimum. Examining the weathering results, it is noted that the lime additives produce stabilities higher than the asphalt-aggregate system using 150 penetration asphalt cement. Stabilities received were: 2 percent lime — 12 percent AC produced 2,050 lb dry, 2,025 lb wet, and 2,300 lb weathered. Lime added in small amounts for increased stability is justified.

3. Rubberized asphalt 160 penetration gave the following stabilities: 12 percent R AC 160 — 1,280 lb dry, 1,180 lb wet and 2,100 lb weathered. The rubberized asphalt had a high index of retained strength and had stabilities comparable to normal asphalt.

Rubberized asphalt has a quality of high temperature stability coupled with low temperature flexibility.

4. The Marshall stabilities and weathering investigation demonstrated that diamines and phosphoric acid additives would not be usable in a baked shale aggregate system.

5. The retained strength from the effect of water on compacted specimens containing the amine group of additives signified that the additives definitely aid in the resistanc to the detrimental effect of water. However, the stability values are lower than those of heat-altered shale with normal asphalt of 150 penetration, under the short cure. The weathered specimens, of the alamine amine however are above those given by normal 150 penetration asphalt. The stabilities produced would make questionable the use of the alamine additive. The proper way in which to evaluate the effectiveness of the additive would be its incorporation into a highway test strip.

RECOMMENDATIONS

Recommendations for highway test road construction using Wyoming heat-altered shale and additives are as follows:

1. Highway test strips of heat-altered shale should be constructed and better additives and asphalts should be evaluated. Test strips should be compared to parallel sections of equivalent normal asphalt cement using aggregate of proven quality.

2. The test strips should be constructed in accordance with strict specifications, presently in effect, to produce a successful roadway.

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