STORAGE OF BITUMINOUS CONCRETE IN INERT GAS

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Possible problems associated with hot storage of bituminous concrete mixes can be migration of asphalt, segregation of the mix, and aging of the asphalt cement due to prolonged storage at high temperature. Laboratory and field investigations were conducted to evaluate the effect of prolonged hot storage on the properties of a bituminous paving mix. No additive was used in the asphalt cement. Storage of mixes was attempted with and without inert gas for as long as 10 days. Field tests were conducted with inert gas only to extend the storage period. Migration of asphalt and segregation of mix did not pose any problem. In the pilot tests, mix stored without inert gas hardened at a faster rate than that stored with an inert atmosphere. If the maximum allowable storage time is determined from the criterion of minimum percentage of retained penetration (AASHO M 20-70) on TFOT residue, this can vary depending on the asphalt source, composition of the bituminous mix, temperature, and time of mixing. No storage might be possible in some cases; however, based on these criteria, the allowable storage period using inert gas was found to be 24 hours in this field study. It is yet to be established to what extent the durability of the pavement is sacrificed because of premature hardening of the asphalt in the hot storage bin.

•THE installation of hot-mix surge and storage-bin systems has considerably increased throughout the United States during the past 2 or 3 years. This study was undertaken to investigate the effects of prolonged storage at high temperature on the properties of a bituminous paving mixture.

The research program consisted of three phases: Phase 1 was the laboratory hot storage study to evaluate the rheological properties of the asphalt cement on being stored at higher temperatures for a considerable length of time, phase 2 was a semi-field test using two pilot storage bins (3 ft in diameter and 3 ft deep) placed in the yard outside the laboratory, and phase 3 was a full-scale field test using 200-ton hot storage bins supplied with inert gas. Phase 3 involved storage of base course and wearing course bituminous mixtures in inert atmosphere for as long as 10 days.

The objectives of this study were to ascertain if there is segregation in the resulting mix; evaluate possible migration of asphalt during storage; determine the effects of storage on the physical properties of the mixture such as Marshall stability, flow, stiffness, and so forth; and evaluate the changes in rheological properties of the asphalt cement caused by prolonged storage at high temperatures.

REVIEW OF LITERATURE

The effect of hot storage on bituminous concrete mixes has been studied by various research agencies with variable results. It has been stated (1) about hot-mix surge storage that safe storage periods are not so much a function of bin design as they are a function of the nature of the mix and its uniformity.

Middleton et al. (2) studied the effects of hot storage on a fine-graded bituminous paving mix (using silicone treated 85 to 100 penetration grade asphalt cement) that was

Publication of this paper sponsored by Committee on Characteristics of Bituminous Materials.

stored in a silo for a total of 97 hours. Results showed that there was a marked change in the properties of the asphalt cement during mixing but that any change that may have occurred during storage was practically negligible.

Parr (3) has reported about storage of a fine-graded bituminous base course mix using sand-gravel as aggregate and 40 to 50 penetration grade asphalt cement. Two series of tests of storage were conducted: Series A contained asphalt that was untreated, and series B contained asphalt treated with 2 ounces of an additive containing silicone materials for each 5,000 gal of asphalt. With the treated asphalt, no significant change in either penetration or ductility occurred for 72 hours of hot storage. However, with untreated asphalt, significant changes in both penetration and ductility were observed in samples taken after 24-hour storage at the hot-bin temperature.

Vallerga and White (4) reported significant hardening of asphalt cement after 4 hours. The rate of hardening was significantly decreased when silicone fluid was added to the asphalt.

Brock and Cox (5) reviewed the results of tests on actual installations and concluded that hot bituminous mix may be stored without detrimental effects for 10 days in a heated silo blanketed with an inert gas and for more than 14 days if the mix contains silicone and is held in a heated silo blanketed with an inert gas.

A series of gradation tests were conducted (6) on surface-course mix obtained from the silo, but no appreciable change in gradation was noted due to storage.

Four series of 72-hour tests conducted by the National Asphalt Pavement Association (7) indicated that the use of an inert atmosphere increased the storage time from 6 to more than 72 hours for mix made with nontreated asphalt. The use of silicone thus increased storage time from about 24 to more than 72 hours in the inert atmosphere and from about 2 to 6 hours in air.

Parr and Brock (8) conducted a statistical study of effects of hot storage on the composition and properties of a bituminous concrete binder mixture stored for 7 days in a sealed silo equipped with a hot-oil heating jacket and purged with an inert gas inside. They concluded that the mix composition before and after storage was not significantly different. However, some hardening of the asphalt did occur, but it was not severe because the drop in penetration was less than that permitted by specifications for the thin-film oven test (TFOT).

Because the Pennsylvania Department of Transportation does not permit the use of silicones as an additive in the asphalt cement, this additive was excluded from this study.

Effect of hot storage on the rheology of the asphalt cement has not been studied in greater detail. Hardening of asphalt cement because of storage has mostly been observed by noting change in penetration only. Recent research (9, 10, 11) has indicated that, besides absolute viscosity, other parameters such as shear susceptibility and temperature susceptibility are needed to specify the complete rheological behavior of paving grade asphalts. The major factors affecting the durability and performance of the pavements have been observed to be the aging index (based on viscosity at 77 F) and the gain in shear susceptibility of the asphalt cements (12). An attempt has been made in this study, therefore, to evaluate the effects of hot storage on these rheological parameters also.

PRELIMINARY LABORATORY AND SEMI-FIELD TESTS

Laboratory Study

This study was undertaken to simulate the condition when the bituminous concrete mix is stored at high temperature in a sealed silo without inert gas. Bituminous wearing course mix made of limestone aggregates and AC-2000 asphalt cement was stored in 1-quart friction top cans and kept in an oven at 290 F for as long as 15 days. After specified time intervals, the cans were taken out and chilled by immersion in ice water to minimize the effect of continued heat. Asphalt was recovered by the Abson method, and the rheological tests were conducted on the recovered asphalt. Complete data are given elsewhere (13).

Because the cans were sealed, hardening of asphalt continued until the limited oxygen in the air was utilized in about 24 hours of storage, and thereafter further hardening was negligible for as long as 15 days. During the first 24 hours, penetration dropped from 67 to 53, viscosity at 77 F increased from 3.0 to 4.0 megapoises, viscosity at 140 F increased from 3,450 to 5,290 poises, and viscosity at 275 F increased from 647 to 793 cs.

Semi-Field Study

Bituminous mixes were stored in two CMI pilot bins mounted on a trailer for demonstration purposes. The complete unit, which was placed in the back yard of the BMTR laboratory, consisted of two hot-mix containers 3 ft in diameter and 3 ft deep and an oil heating and inert gas system. One container was supplied with inert gas and jacketed with hot oil; the other was only jacketed with hot oil. Bituminous mixtures were fed in these preheated containers at a temperature of 290 F. The mixtures were stored for as long as 10 days.

Tests similar to the laboratory study were run on the recovered asphalt, and the complete data obtained are given elsewhere (13). The mix stored without inert gas hardened at a faster rate than that stored with inert atmosphere in the bin. However, in both mixes, there was a significant drop in asphalt penetration and ductility and significant increase in viscosity and shear susceptibility during the first 5 to 6 hours. Asphalt cement hardened more than that permitted by specifications for the TFOT. This indicated that the rate of hardening during storage depends primarily on the asphalt source (if storage conditions are held the same). It may be mentioned, however, that, because of problems in maintaining temperature and regulating the inert gas, the data would not relate to the ideal conditions.

FIELD TEST RESULTS

The storage system of the H. R. Imbt Co. (asphalt plant 6) located at Bossardsville, Pennsylvania, was used for this field study. The storage system consisted of the following:

1. Three 200-ton circular bins heated by hot-oil jackets and insulated—Automatic controls were available to maintain the unit at the desired temperature. The bin is a tall, vertical unit on top with a tapered hopper that tends to remix the bituminous concrete while discharging. Each bin is provided with a bottom air lock below a clam-type discharge gate and top air lock to seal the unit and hold the inert gas at a constant low pressure to prevent outside atmosphere from entering.
2. Thermal oil heater—Hot oil from the heater is pumped to the storage unit by a high-volume pump.
3. Inert gas generator—When the generator is operating, inert gas is pulled from the exhaust of the pre-mix burner and is then cooled, dehydrated, compressed, and stored in a pressure tank.
4. Drag chain conveyor—The conveyor is a continuous type and is enclosed to prevent spillage of mix and to maintain temperature.
5. Control room—The room contains consoles consisting of all controls necessary for discharging mix, bin level indicators, and controls for heating and inert gas systems.

Only two bins were used. In one bin, Pennsylvania ID-2 wearing course mix was stored; in the other bin, bituminous concrete base course (BCBC) mix was stored. Sampling of the bituminous concrete was done at specified intervals during the 10-day storage period. Samples were obtained as per ASTM D 979-51. After each storage period, two 1-gal cans were filled with bituminous mix, chilled in cold water, and then shipped to the BMTR laboratory for testing. Samples were also used in compacting three Marshall specimens immediately at the plant laboratory. These were stored for 24 hours and then tested for Marshall stability and flow.

Marshall data obtained on samples of ID-2 wearing course mix are shown in Figure 1. The temperature of discharged mixture from the two bins at the time of sampling is shown in Figure 2.

Asphalt cement was extracted from the duplicate samples of bituminous mix received in the central laboratory. Based on extraction test data, the gradation number, the surface area of the recovered aggregate, and the bitumen index have been determined

Figure 1. Marshall test data (ID-2 wearing course mix).

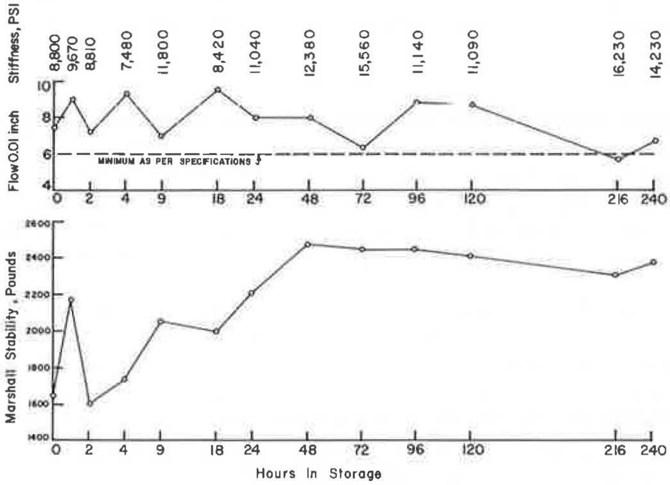


Figure 2. Temperature of discharged mix (field study).

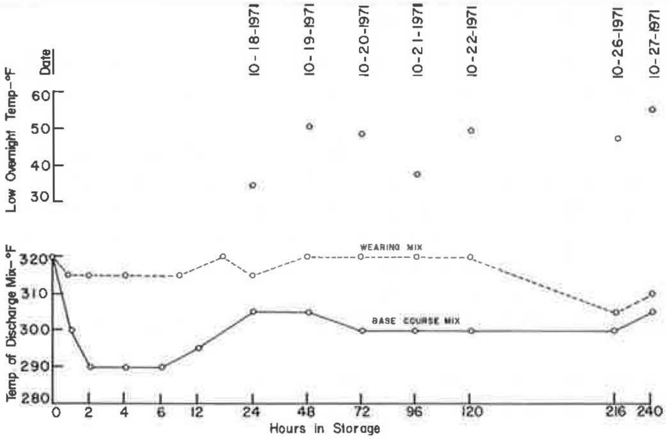


Table 1. Mix data.

Hours in Storage	Asphalt Content (percent)	Gradation Number	Aggregate Surface Area (ft^2/lb)	Bitumen Index (lb/100 ft^2)
0	3.6	2.068	17.74	0.211
1	3.8	2.336	20.18	0.196
2	3.8	2.149	18.49	0.214
4	3.6	2.293	19.66	0.190
6	3.5	2.333	19.38	0.187
12	3.6	2.404	19.81	0.189
24	3.5	2.381	19.27	0.188
48	3.7	2.572	20.09	0.191
72	3.4	2.349	18.07	0.195
96	3.7	2.468	18.98	0.202
120	3.6	2.335	17.86	0.209
216	3.6	2.154	17.34	0.215
240	3.6	2.356	18.64	0.200
Average	3.6	2.323	18.89	0.199
Standard deviation	0.11	0.135	0.936	0.012

to examine the variability in the mix sampled. These data are given in Table 1 for the base course mix. The gradation number is similar to fineness modulus except it is the sum from the ½-in. and smaller sieves of the percent fractions passing divided by 100.

The recovered asphalt cement was tested for penetration at 77 F; ductility at 60 F, 5 cm/min; absolute viscosity at 77 F, 0.05 sec^{-1} shear rate and shear susceptibility; absolute viscosity at 140 F; and kinematic viscosity at 275 F.

Figures 3, 4, 5, and 6 show these data versus the storage period for both the mixtures.

Absolute viscosity, using the sliding plate microviscometer, was also determined at 50, 104, and 122 F. Temperature susceptibility data based on these results are given in Table 2 for the base course mix only.

Storage aging indexes, based on viscosity at 77 F, have been determined for both mixtures and are shown in Figure 7. These have been determined as follows:

$$\text{Storage aging index} = \frac{\text{viscosity after storage}}{\text{viscosity before storage}}$$

DISCUSSION OF TEST RESULTS

Effect of Storage on Properties of Bituminous Concrete

Changes in the properties of bituminous concrete resulting from storage in the full-scale storage bins are as follows.

Migration of Asphalt Cement—Extraction test data indicate reasonable uniformity of asphalt content in the two mixtures sampled after various storage intervals for as long as 10 days. If the migration of asphalt cement in either of the mixes had taken place toward the bottom of the bins, the asphalt content would have increased with storage period. The standard deviation in the asphalt content for the samples tested was observed to be 0.14 and 0.11 percent for the wearing course and base course mixtures respectively. Migration of asphalt would perhaps occur if the storage is attempted at temperatures higher than 320 F or the bituminous concrete stored is rich in asphalt.

Segregation—Possible segregation of the mixtures due to storage was evaluated by subjecting to sieve analysis the aggregate recovered by the extraction test from the sampled mixtures. The gradation of the recovered aggregate from both the mixtures falls within the tolerance limits as per specifications. Only the normal variation that is inherent in a bituminous concrete plant has been observed.

Gradation number and bitumen index have been used (8) to evaluate the extent of segregation and the uniformity of asphalt film thickness. The aggregate surface area of each sample was calculated to determine the bitumen index. Normally a coarse mix would tend to segregate more than a fine mix. Data given in Table 1 for base course mix do not indicate any segregation problem.

Marshall Stability and Flow—Marshall specimens were compacted from the wearing course mix samples and tested for stability and flow with an automatic recorder. In the specifications for Pennsylvania ID-2 wearing course, a minimum stability of 1,200 lb and a flow value between 6 and 16 are required. The average stability and flow of the mix out of the pug mill was observed to be 1,600 lb and 8 units respectively. Figure 1 shows the changes in these values with the storage period. As would be expected if the asphalt hardens, there was a general trend of increase in stability and decrease in flow due to storage. However, the mix stored for as long as 10 days still met the requirements of the specifications. Most of the increase in stability took place in the first 48 hours of storage. This is explained by a similar increase in asphalt viscosity measured at 140 F (Fig. 5).

Van der Poel developed data indicating that, in case of mixtures containing dense-graded aggregates and asphalt cements, which were well compacted (approximately 3 to 5 percent air voids), the stiffness of a mixture is dependent on the stiffness of the asphalt that it contains and the volume concentration of the aggregate (14). At very short times of loading or low temperatures or both, the behavior of asphaltic concrete is almost elastic in the classical sense, and the stiffness, S, is analogous to an elastic modulus, E. At longer times of loading and higher temperatures, the stiffness is

Figure 3. Penetration and ductility (field study).

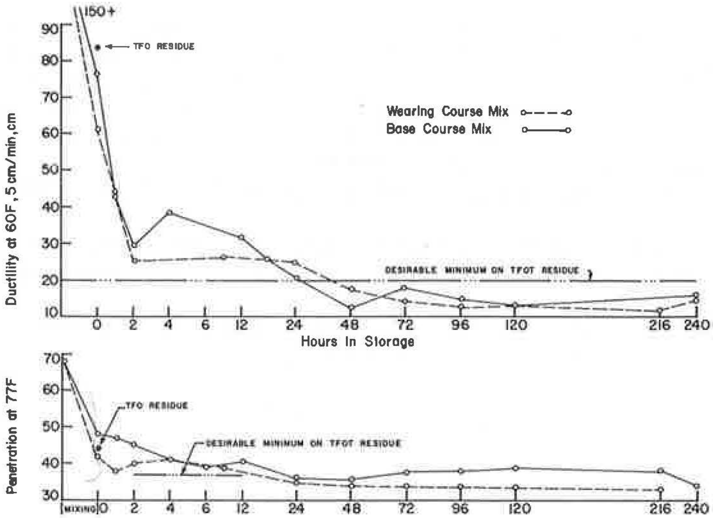


Figure 4. Viscosity and shear susceptibility (field study).

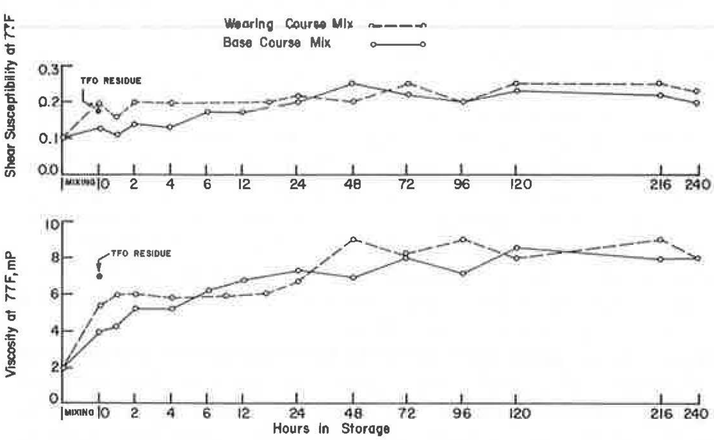


Figure 5. Absolute viscosity at 140 F (field study).

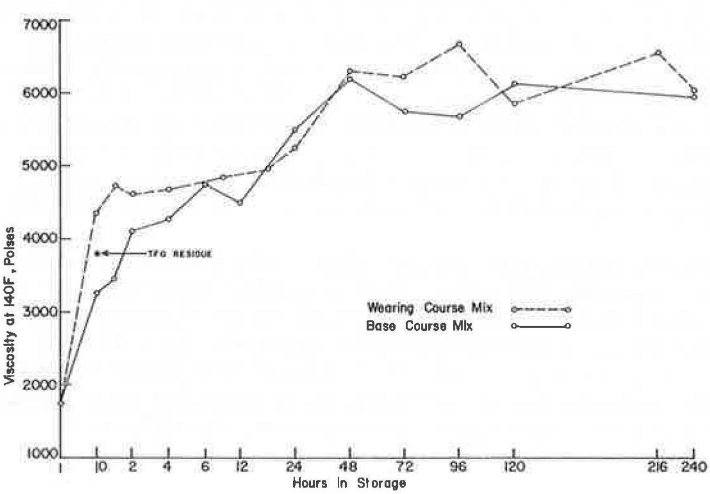


Figure 6. Kinematic viscosity at 275 F (field study).

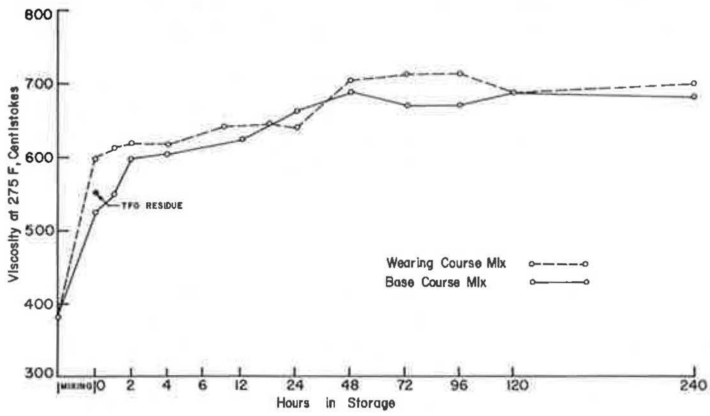
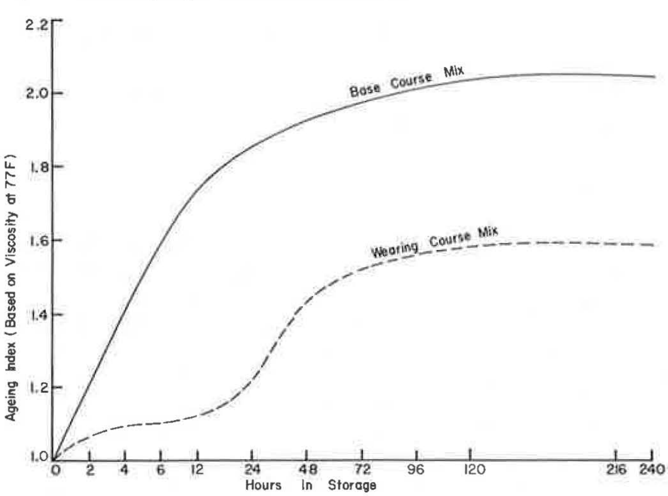


Table 2. Temperature susceptibility of base course mix.

Hours in Storage	Temperature Susceptibility of Recovered Asphalt Cement		
	50 to 77 F	77 to 140 F	140 to 275 F
0 (pug mill)	3.62	3.60	3.48
12	3.16	3.57	3.47
24	3.14	3.40	3.50
72	3.13	3.40	3.51
120	3.12	3.38	3.51
240	3.13	3.20	3.50

Figure 7. Storage aging index (field study).



simply a relation between the applied stress and the resulting strain. Stiffness of the stored wearing course mix, calculated from the stress-strain data obtained in Marshall tests, is shown in Figure 1. Stiffness of the mix increased by 50 percent in the first 48 hours.

Temperature of Stored Mix—Figure 2 shows the temperature of the mix discharged from the storage silo at the time of sampling. There were some initial drops in temperature of base course mix, but the temperature stabilized after 24 hours. On the whole, temperature of the wearing course mix was 15 to 20 F higher than that of the base course mix.

Effect of Storage on Properties of Asphalt Cement

Laboratory and semi-field tests confirmed field tests reported by others that inert gas extends storage time, and, because the intent was to extend the storage to 10 days, only inert gas was tested in the field. No problems with respect to the functioning of the heating and inert gas systems were encountered, so the results can be considered as realistic in a practical sense. However, the results would be valid only for the asphalt cement actually used in the two mixtures. Though the same asphalt cement was used in the wearing course and base course mixes, more hardening occurred in the pug mill in case of wearing course mix. The effect of storage on the various properties of asphalt cement is discussed in the following.

Penetration—Penetration of original asphalt cement was 68. After mixing in the pug mill, the penetration of asphalt was noted to be 42 and 48 in the wearing course and base course mixes respectively. Drop in penetration of asphalt cement (Fig. 3) occurred from 42 to 34 in the wearing course mix and from 48 to 36 in the base course mix during the first 48 hours of storage period, and then there was no appreciable change. Most of the drop in penetration took place in the first 24 hours. According to AASHO M 20-70 on penetration graded asphalt cements, penetration of residue after TFOT for the asphalt cement used can be permitted as low as 37 (that is, 54 percent of original). If this is assumed as a criterion for acceptance, these mixes would be acceptable if stored for 24 hours or less (Fig. 3). However, this may not be applicable in general. More hardening in the pug mill itself can take place in some cases, and thus storage may not be possible at all. The following data taken from two projects (15) using an asphalt cement from the same source would indicate this:

	Penetration Values			Percentage Retained Penetration		
Mix	Original	After Mixing	After Compaction	Original	After Mixing	After Compaction
LR 219	59	36	31	100	61	53
LR 101	59	44	40	100	75	68

It would not have been possible to store the LR 219 mix to meet the same criterion, whereas LR 101 mix could possibly be stored for some period. Asphalt from the same source was used in these projects, but different aggregate sources and aggregate gradations were used. Thus, it would seem that the allowable storage time depends on the asphalt source and the composition of the bituminous mix. It is evident that hardening in 24 hours of storage can be equivalent to about 20 months of hardening in the pavement based on penetration (Fig. 8). The two asphalts compared are from different sources but had almost the same penetration after pug mill mixing. These asphalts had almost identical viscosity at 77 and 140 F.

Ductility—There was significant drop in ductility (60 F) in the first 2 hours of storage (Fig. 3). The ductility dropped to 25 and 30 cm in the wearing course and base course mixtures respectively. The desirable minimum ductility on the TFOT residue is 20 cm at 60 F; therefore, these mixtures would not be acceptable after 24 hours. The change in ductility value beyond 48 hours was negligible.

Viscosity at 140 F and 275 F—Viscosity at 140 F of asphalt cement increased from 4,350 to 6,300 poises in wearing course mix and from 3,250 to 6,200 poises in the base

Figure 8. Penetration and viscosity of asphalt (silo versus pavement).

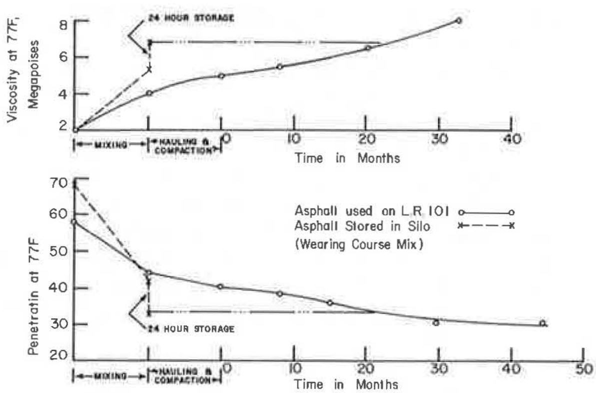
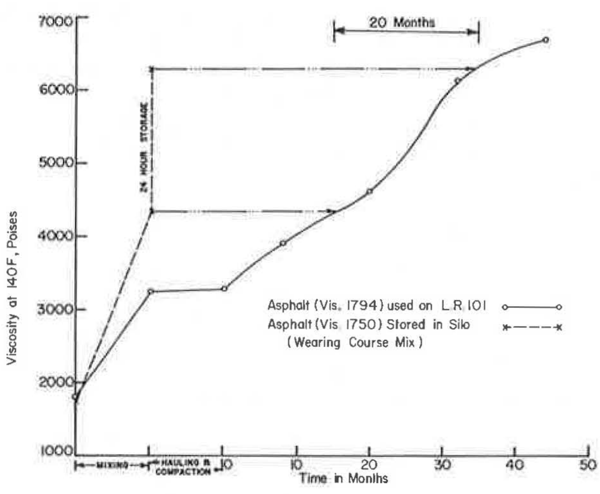


Figure 9. Viscosity at 140 F of asphalt (silo versus pavement).



course mix during 48 hours of storage, and then it leveled off (Fig. 5). Maximum permissible viscosity after the TFOT is 8,000 poises, which was not reached in 10 days of storage. However, when compared to hardening in the pavement based on viscosity, the hardening due to 24 hours of storage is equivalent to that that occurred in pavement in about 20 months (Fig. 9).

Viscosity data at 275 F also indicate the same trend (Fig. 6).

Absolute Viscosity at 77 F—Increase in absolute viscosity at 77 F was noted during the first 48 hours of storage, most of which occurred in the first 24 hours. Thereafter, no significant increase took place (Fig. 4). Increased viscosity at 77 F reduces the capability of the pavements to compact under traffic at ambient temperatures (12). Hardening in 24 hours of storage can be equivalent to about 20 months of hardening in the pavement based on viscosity at 77 F (Fig. 8). The two asphalts compared had original viscosity of 2.0 megapoises.

Because the same asphalt in the wearing and base course mixes did not harden to the same extent in the pug mill, storage aging index is a better indicator of relative hardening in the storage silo. Asphalt in the base course mix hardened at a faster rate than the asphalt in the wearing course mix (Fig. 7) as would normally be expected. However, ultimate hardening of the asphalt was greater in the wearing course mix than the base course mix.

Shear Susceptibility—There was no significant increase in shear susceptibility (77 F) of the asphalt in the wearing course mix, whereas the shear susceptibility increased during the first 24 hours of storage in the base course mix (Fig. 4). However, in both cases, the shear susceptibility does not seem to be excessive.

Temperature Susceptibility—Temperature susceptibility data for asphalt in the base course mix are given in Table 2, based on the viscosity data at 50, 77, 104, 122, 140, and 175 F. Because of shear dependent viscosities at temperatures lower than 140 F, slopes of the temperature viscosity lines deviate from those obtained between 140 F and higher temperatures. Therefore, the temperature susceptibility of the asphalts has been determined in three temperature ranges: 50 to 77 F, 77 to 140 F, and 140 to 275 F. It will be observed that the temperature susceptibility values of the asphalt have shown a decreasing trend with storage aging in the first two temperature ranges, whereas the increase in the range of 140 to 275 F is not significant.

General Data—Most of the hardening during the 10-day storage period in inert atmosphere occurred in the first 24 to 48 hours. It seems that some air is trapped by the mixture when it is dropped from the pug mill onto the conveyor used in charging the silo. The oxygen in this air is not displaced by the inert silo atmosphere and undoubtedly oxidizes the asphalt of the stored mix at the high-temperature storage conditions. This was also observed by Parr and Brock (8). The significant increase in asphalt viscosity (77 and 140 F) and drop in penetration because of 24 hours of storage in inert atmosphere has been observed to be equivalent to 20 months of hardening in the pavement using asphalt of the same consistency based on viscosity. Though the drop in penetration and increase in viscosity is less than that permitted by AASHO specifications for the TFOT (AASHO M 20-70 and M 226-70), the amount of hardening that should be classed as detrimental is a moot question.

CONCLUSIONS

The following conclusions were drawn with regard to laboratory and semi-field tests:

1. Mix stored without inert gas hardened at a faster rate than that stored with inert atmosphere in the bin, and
2. The aging index is a better indicator of relative hardening when asphalts from different sources are used or mixtures of different composition are stored.

Full-scale field tests were conducted using inert gas only, and the following conclusions are drawn:

1. No migration of asphalt cement in the bituminous concrete was observed during 10-day storage.
2. All silo discharge samples met the specification requirements for gradation of wearing and base course mixes, so there was no segregation problem.
3. Wearing course mix samples obtained from the storage silo for as long as 10 days still met the specification requirements for Marshall stability and flow. However, the stiffness of the mix increased by 50 percent in the first 48 hours because of increased Marshall stability and reduced flow.
4. Based on the aging index, base course mix hardened at a faster rate than the wearing course mix.
5. Most of the hardening during the 10-day storage period occurred in the first 48 hours. It seems the air entrapped in the mix is not displaced completely by the inert silo atmosphere and thus oxidation continues for a limited period.

The following general conclusions were made:

1. If the maximum allowable storage time is determined from the criterion of minimum percentage of retained penetration (AASHO M 20-70) on TFOT residue, it can vary depending on the asphalt source, composition of bituminous mix, temperature, and time of mixing. Although no storage might be possible in some cases, based on these criteria, the allowable storage period using inert gas was found to be 24 hours in this study.

2. Asphalt hardening (aging) during 24 hours of storage of bituminous concrete in inert atmosphere can be equivalent to 20 months of hardening in the pavement. It is yet to be established to what extent the durability of the pavement is sacrificed because of this premature hardening in the hot storage bin. Further investigations should be attempted to include comparison of asphalt properties from pavements laid from silo-stored materials with those of asphalt from pavements laid directly from the pug mill.

ACKNOWLEDGMENTS

This report is the result of a research project sponsored by the Pennsylvania Department of Transportation. The opinions, findings, and conclusions expressed are those of the authors and not necessarily those of the Pennsylvania Department of Transportation. CMI Systems, Chattanooga, Tennessee, and the Pennsylvania Asphalt Pavement Association cooperated in this study.

Thanks are due to W. S. Myers for assistance in the field study, P. G. Kaiser for physical testing of the bituminous mixtures, and H. R. Basso, Jr., for the testing of recovered asphalt.

Appreciation is also expressed to Edward Macko for illustrations and June Viozzi for compilation of research data.

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DISCUSSION

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The authors of this paper are to be congratulated on bringing information on such an interesting and timely subject to us. The use of hot storage bins has increased rapidly in the past few years, and the reduction in cost that comes from greater flexibility in scheduling hot-mix operations should not be ignored. It is my understanding that the reduction in cost is significant, and, in a competitive market, this reduction will soon be passed on to the buyer.

This paper points out the importance of silicone fluid in reducing the rate of hardening of asphalt binders, thus making possible longer storage periods without significant hardening of the mix. Previous papers (2, 3, 5, 7) have shown that asphalt mix could be stored for several days with only negligible hardening. The authors point out that silicone liquid was used in each case in contrast to the present paper where silicone was not used and where a storage period of only a day or less would be allowable. It is interesting to note that the data of this paper like those of other papers (2, 3, 5, 7) show no migration of asphalt cement in the mixture and no segregation problem.

The matter that causes me the greatest concern in this paper is that there is no indication of the random variation of the testing procedures. This is not so much a criticism of the paper or the authors as it is of the general current practice in the bituminous field. We know that the precision of our bituminous testing methods such as the Abson recovery method, Marshall stability, flow, penetration, ductility, viscosity, and shear susceptibility are poor. When they are added together as they are here, the errors become very large. Some indication of the confidence spread about each of the time series lines in Figures 1 through 9 would be a tremendous help in evaluating the meaning of these figures.

AUTHORS' CLOSURE

Samples were tested in duplicate, and reported results are the average of these values. It is realized that precision of bituminous testing methods is poor. It must be pointed out that each of the specific tests cited was performed by only one trained operator in each case. This procedure should tend to cancel the influence of variances among operators.

We admit that it would be highly desirable to have data that would permit establishment of the confidence spread about each of the time series lines. However, the scope of this investigation and the manpower and testing hours precluded development of these data.

Any future work on this study will be expanded to permit determination of the confidence limits. We are also considering the Orsat analysis of the inert gases to determine the effect of oxygen and carbon monoxide on the asphalt properties.