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Iowa Highway Research Board
Project HR-124 conducted by the
Engineering Research Institute,
Iowa State University for the
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PROGRESS REPORT NO. 3

DEVELOPMENT OF A DURABILITY TEST FOR ASPHALTS

HR-124

1 October 1968 to 31 October 1969

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Iowa Highway Research Board Project HR-124
conducted by the
Engineering Research Institute, Iowa State University
for the
Iowa State Highway Commission

ENGINEERING RESEARCH INSTITUTE
IOWA STATE UNIVERSITY AMES

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I. INTRODUCTION

Research Project HR-124, "Development of a Laboratory Durability Test for Asphalts," was initiated in 1966 as a long range comprehensive program. Its ultimate objective was to develop a simple, rapid laboratory test that could be used by highway engineers to select paving asphalt according to quality, to identify inferior asphalts, and to reasonably predict the useful life of asphalts once they were incorporated in the pavements.

The original proposed study on asphalt durability involves work in the following phases:

1. Critical review of the state-of-the-art on the durability of paving asphalts, the identification of predominant factors causing hardening during mixing, laying and in-road service.
2. Development of an accelerated laboratory durability test to simulate changes in asphalt both during short time production and long term road service.
3. Correlation of hardening and other changes in asphalts during the developed laboratory durability test and changes in some asphalts in pavements.
4. Establishment of durability criteria and functional approach specifications by means of established laboratory durability tests on original asphalt.

Work in phases 1 and 2 was accomplished in the original HR-124 (1966 – 1967) project and was presented in Progress Report No. 1, a paper published in Highway Research Record 231 and a Special Report on the state-of-the-art on asphalt durability. The first 11 mo field correlation studies in phase 3 were conducted in HR-124 (1967 – 1969) and were presented in Progress Report No. 2 (October 1968). This report, designated Progress Report No. 3, summarizes the work accomplished during the second year of the HR-124 extension (1967 – 1969), i. e., the period from 1 October 1968 to 31 October 1969.

II. OBJECTIVES

The objectives of HR-124 (1967 – 1969) were:

- refinement of the durability test procedure developed in HR-124 (1966 – 1967),
- durability tests on asphalts used for actual paving projects in Iowa,
- determination of changes in asphalts incorporated in the various paving projects from the plant and at 6-mo intervals thereafter, and
- field correlations for a period up to 24 mo

III. MATERIALS AND METHODS

Asphalt cements, plant mixes and core samples were received from four paving projects each during the 1967 and 1968 construction seasons. The materials received and tested as of 31 October 1969 are given in Table 1. In addition to asphalts received from the Iowa State Highway Commission, an essentially asphaltene-free asphalt cement was obtained from the Esso Research and Engineering Co. and is included in the study to evaluate the role of asphaltenes in the performance of a paving asphalt. The locations of the eight pavement projects are shown in Table 2 and Fig. 1. Same procedures as described in Appendix A, Progress Report No. 2 were followed.

Table 1. Materials received and tested.

Code	County	Type of Mix	Thickness in.	Asphalt	Plant Mix	0-mo core	6-mo core	12-mo core	18-mo core	24-mo core
1	Chickasaw	3/8 in. Type A	3/4 in.	X	X	X	X	X	X	X
2	Dickinson	3/8 in. Type A	1 in.	X	X	X	X	X	X	X
3	Harrison	3/8 in. Type A	1 in.	X	X	X	X	X	X	Nov 69
4	Story-Polk	3/8 in. Type A	1 in.	X	X	X	X	X	X	X
5	Esso	-	-	X	-	-	-	-	-	-
6	Monona	3/4 in. Type A	1 3/4 in.	X	X	X	X	X	Oct 69	April 70
7	Bremer	3/8 in. Type B	1/2 in.	X	X	X	X	X	Nov 69	May 70
8	Keokuk	3/8 in. Type A	1/2 in.	X	X	X	X	X	Nov 69	May 70
9	Jackson	3/4 in. Type B	2 in.	X	X	X	X	X	Jan 70	July 70

Table 2. HR-124 pavement project locations.

No.	County	Location	Date laid
1	Chickasaw	On US 63 north of New Hampton	Nov 1967
2	Dickinson	On Iowa 327 from Iowa 276 east and north	Oct 1967
3	Harrison	On US 75 out of Mo. Valley, north into Mondamin	Nov 1967
4	Story-Polk	On US 69 between Huxley and Ankeny	Oct 1967
6	Monona	On US 75 from Harrison Co. line north into Ottawa 11 mi.	April 1968
7	Bremer	On Iowa 3	May 1968
8	Keokuk	On Iowa 92 from Sigorney east	May 1968
9	Jackson	On US 52	June 1968

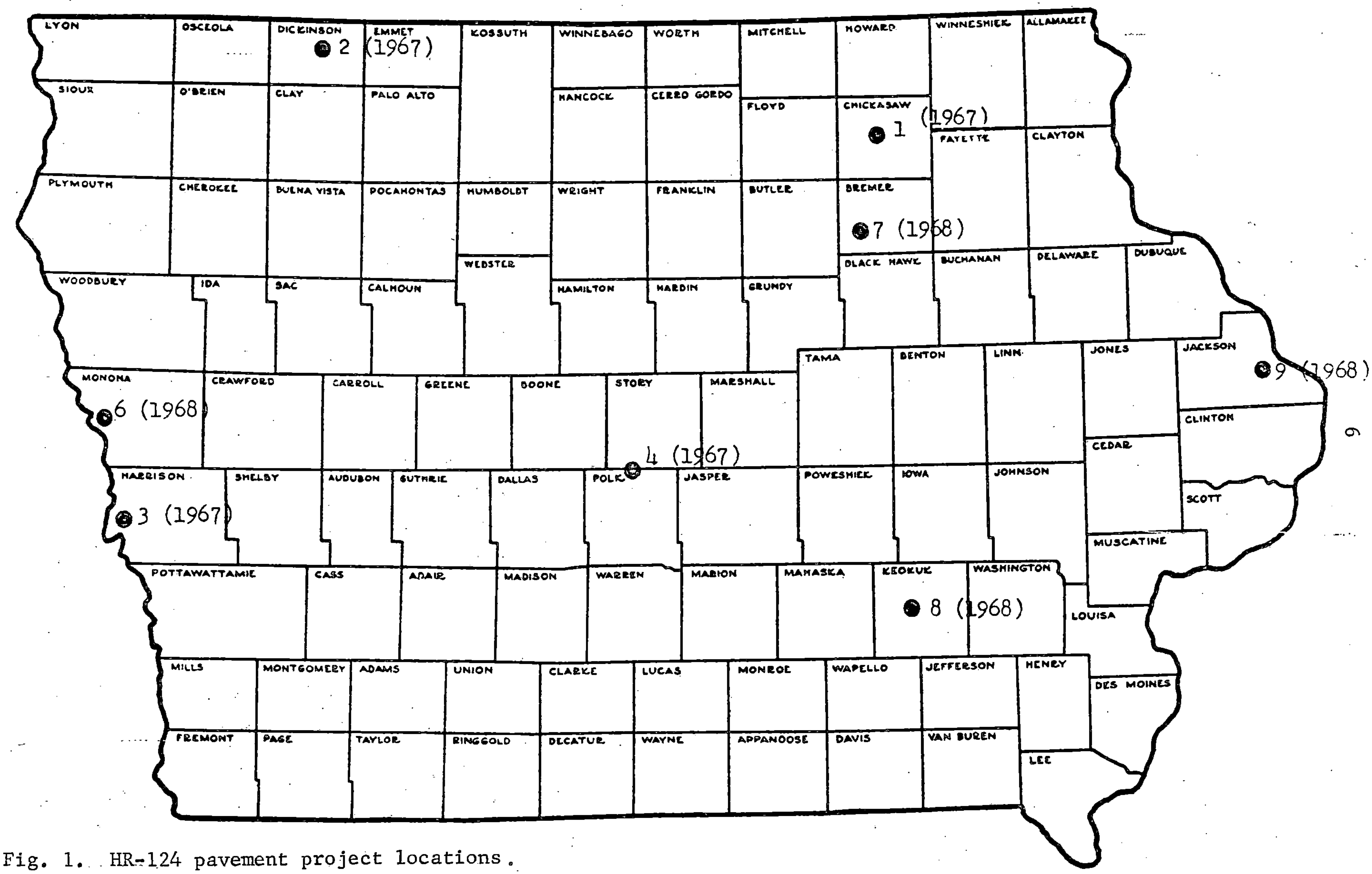

Fig. 1. HR-124 pavement project locations.

IV. BEHAVIOR OF ASPHALTS DURING IOWA DURABILITY TEST

During the first 24 mo of the study, nine asphalt cements were subjected to the laboratory developed durability test (TFO Test plus pressure-oxygen treatment at 150°F and 20 atm), which is hereafter called the Iowa Durability Test. Eight of those asphalts were of 85 – 100 pen. paving grade used in eight paving projects throughout Iowa during the 1967 – 68 construction seasons. The ninth asphalt, also of 85 – 100 pen. grade but essentially asphaltene-free, was obtained from the Esso Research and Engineering Co. and was included in the study to evaluate the role of asphaltenes in the performance of a paving asphalt. Changes in asphalts during Iowa durability tests were measured by viscosity, penetration, softening point, ductility, asphaltenes and oxygen contents, and brittle point.

Aging and Consistency Change in Asphalts Consistency changes of asphalts in terms of penetration, viscosity and softening point are a hyperbolic function of time for all asphalts as shown in Fig. 2 for penetration and Fig. 3 for viscosity at 77°F. This is significant because it agrees with actual asphalt hardening in service as reported by other investigators[1 – 4]. It also suggests that:

- the Iowa Durability Test is realistic, and
- a definite correlation between field hardening and hardening in the Iowa Durability Test is possible.

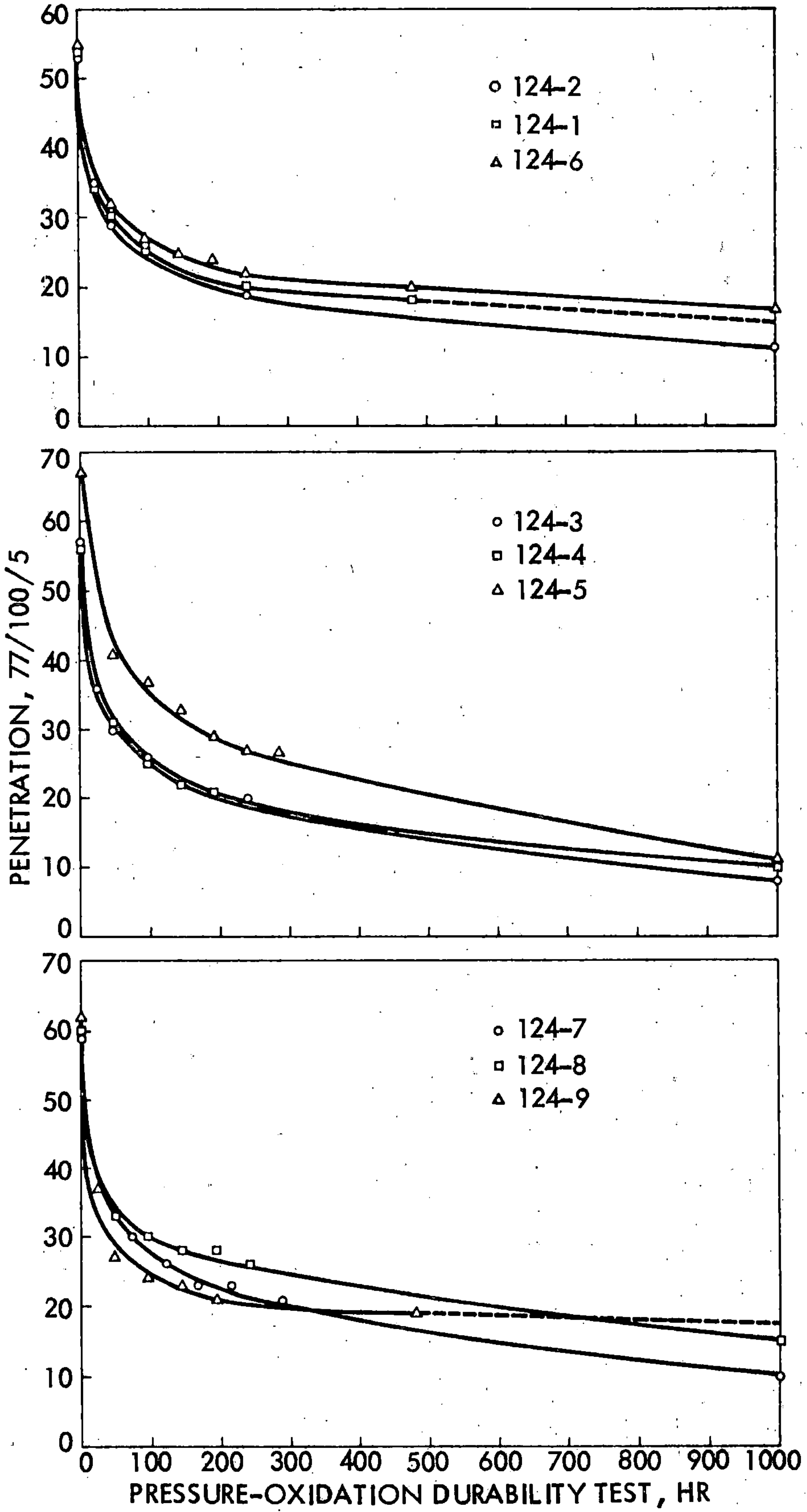

Fig. 2. Penetration change during Iowa Durability Test.

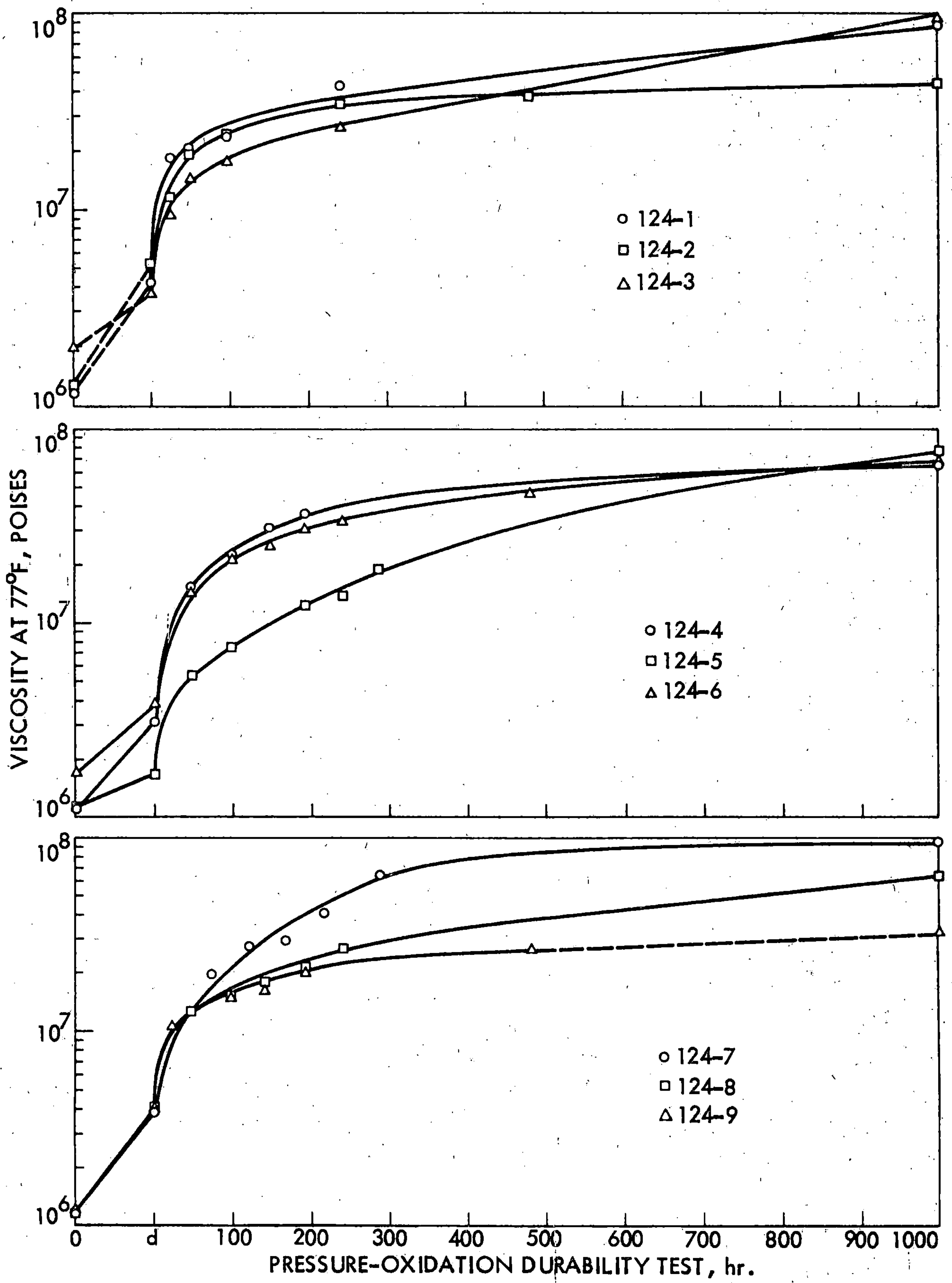

Fig. 3. Viscosity change during Iowa Durability Test.

<u>Aging and Increase in Brittleness of Asphalts</u> The increase in the hardness of an asphalt is generally considered its most important physical change, from a practical viewpoint. Asphalt hardening for asphalts studied in this project, both laboratory aged and field samples, has been determined directly by penetration, softening point, ductility, viscosity at 140°F and at 77°F, and indirectly by such parameters as temperature susceptibility change, penetration index, shear susceptibility, aging index, etc. However, viscosity of aged asphalts in a brittle state of subzero temperatures could not be determined due to instrumentation limitations.

This problem was solved by the modified Frass brittle point test. The test has been used by Lee and Dickinson[5] in Great Britain and Jones[6,7] in Canada to evaluate low temperature behaviors of coal tars and roofing asphalts.

Studies[8] have shown that the brittle point temperature is an equiviscous temperature corresponding to a viscosity of 4×10^9 poises. Thus the test in fact gives the temperature at which the asphalt has a viscosity of 4×10^9 poises, and, as hardness or brittleness increases, there is a corresponding increase in brittle point.

Brittle point determinations for asphalts studied in HR-124 were made on samples with a film thickness of 0.55 mm and rate of cooling of 1°C per minute. Figure 4a shows the rate at which the brittle point temperature changes with the length of exposure of three of the asphalts in the accelerated durability test. A progressive increase in the brittle temperatures is observed for all three asphalts. A slow rate of change of brittleness would indicate a durable material capable of withstanding severe exposure conditions.

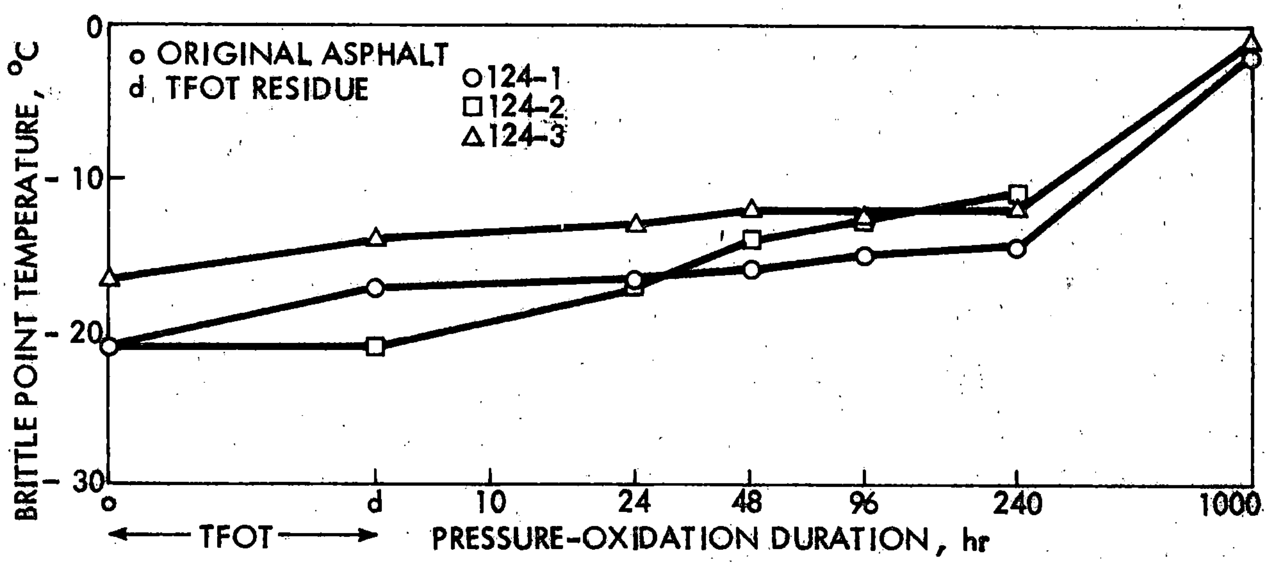

Fig. 4a. Change in brittle point during Iowa Durability Test.

Figure 4b shows brittle point temperature changes in asphalts 4, 5 and 6.

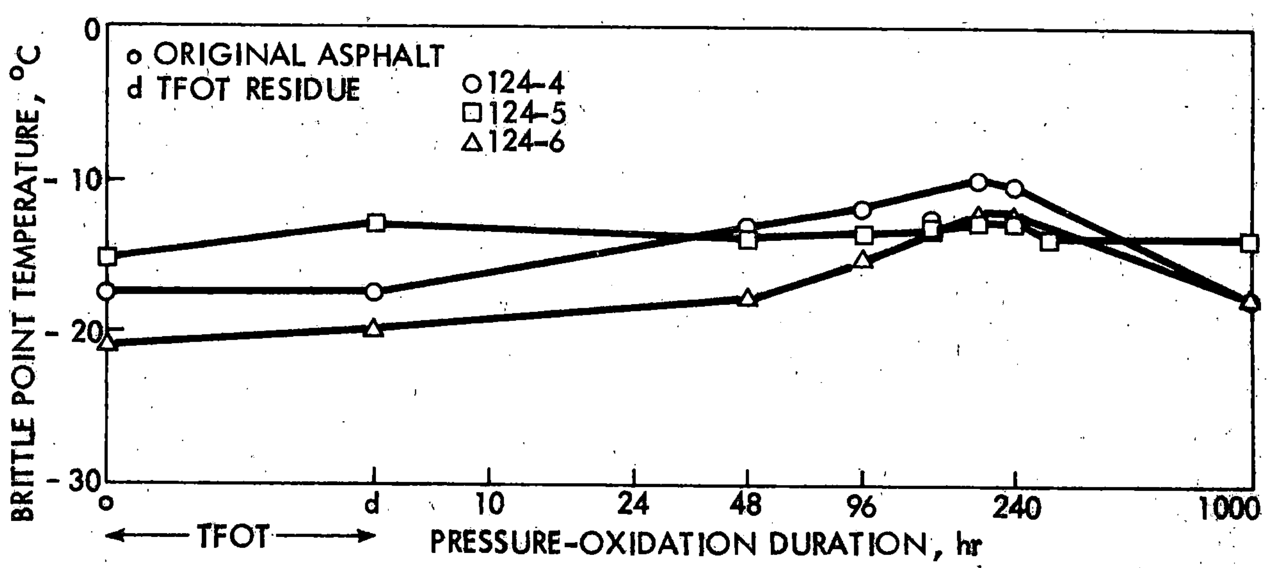

Fig. 4b. Change in brittle point during Iowa Durability Test.

Asphalts 4 and 6 have initial increases in brittleness followed by progressive decreases as the sample aged beyond 200 hr. According to Jones[9], this is characteristic of very durable asphalts. Whether this is the case for asphalts 4 and 6 in HR-124, only extended field correlation beyond two years can answer. Also significant is the very slight change in brittle point for asphalt 5, which is a special asphalt, nearly asphaltene-free.

Due to limited data collected during relatively short periods of field correlation studies thus obtained in HR-124, it is too early to draw final conclusions on the usefulness of the brittle point test in evaluating, predicting and specifying paving asphalt relative to durability and low temperature behavior of asphalts. However, the following statements are justified:

1. The determination of the brittle point temperature, in the absence of instrumentations for very low temperature viscosity measurements, provides a useful method to study changes in the low temperature behavior of asphalts exposed to both field and laboratory accelerated weathering.
2. Variation exists among asphalts in brittle point change during aging.
3. A variation in the initial brittle point temperature or the residue from the TFOT of asphalts and the rate of brittle point temperature change in the accelerated durability test may be related to the performance of the materials in the field.
4. Studies should be continued to evaluate the use of these concepts for asphalts in field service conditions for longer periods to establish correlation in functional terms.

Aging and Chemical Changes in Asphalts Chemical approach of durability study of asphalts has been criticized as premature due to lack of definitive knowledge on chemical composition and structure of asphalt. However, this is a fundamental approach. It has been proved that chemical composition does have direct effects on rheological and durability properties of asphalt and that relationship between asphalt durability and chemical composition could be established[10] and durability grouping of asphalts by chemical composition was possible[11].

Asphaltene content and percent oxygen have been used as parameters for chemical changes. Asphaltene content was determined by precipitation with a Skelly F, and oxygen content was determined by combustion method using a Coleman Oxygen Analyzer. Due to inherent weaknesses in the design of the instrument and intermittent malfunction, the oxygen content results have been incomplete and often misleading. However, gradual increase in oxygen content during aging (both during durability test and road service) is obvious (Table 3). The degree and rate of oxidation is also indicated by weight increase during pressure-oxidation treatment (Table 4 and Fig. 6).

Asphaltene content changes of AC 124-1, 3 and 4 are shown in Fig. 5. It is obvious that

- aging is accompanied by increase in asphaltene content,
- change in asphaltene content is a hyperbolic function of time, and
- rates of asphaltene content change are different for different asphalts.

Table 3. Percent weight gain during pressure-oxidation test.

AC	48	72	96	144	192	240	480	1000	1000A[a]
1								2.62	
2							1.46		
3								2.98	
4								2.60	0.43
5	0.40							3.14	
6	0.56		0.68	0.95	1.03		1.26	2.00	0.40
7		0.65						2.73	
8	0.63		0.76	0.94	0.98	1.02		2.40	
9	0.52		0.66	0.74	0.81		1.30		

[a] 1000A: 1000 hr at 150°F and 1 atm air.

Table 4. Change in oxygen content during Iowa Durability Test.

Hours pressure - oxidation treatment

AC	o[a]	d[b]	24	48	96	144	192	240	1000
1	1.21	1.49	1.63	1.79	2.19			2.06	
2	1.30	1.58	1.69	1.68	1.74			2.49	
3	0.98	1.08	1.11	1.82	1.74			1.92	
4	1.26	1.37		1.33	1.47	2.36	2.77	2.33	
5	1.01	1.19		1.44	1.59	1.70	2.65	2.00	

(a) o = original sample

(b) d = TFOT residue

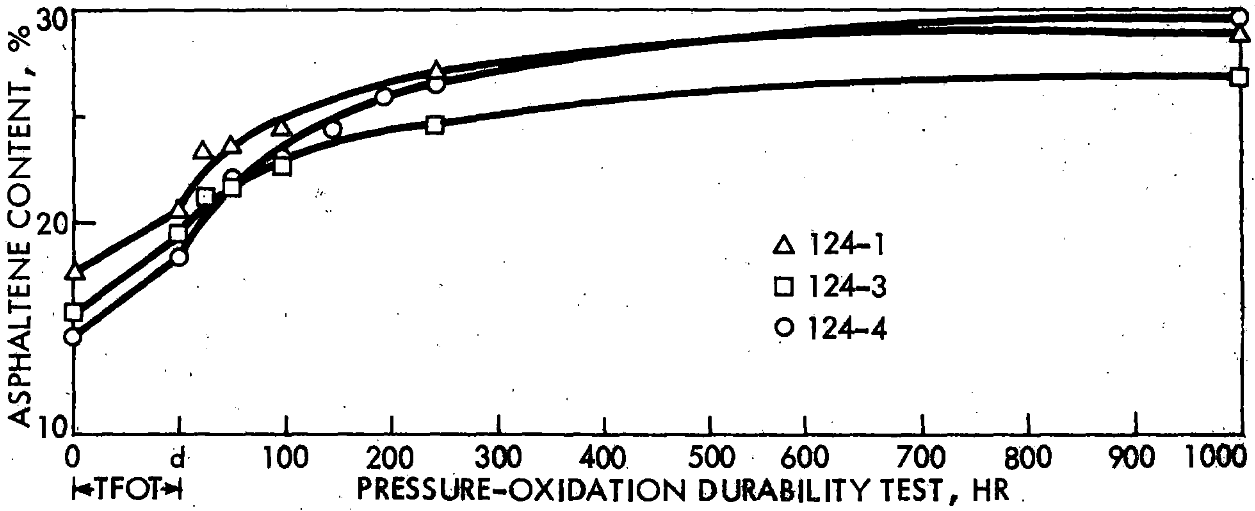

Fig. 5. Change in asphaltene content during Iowa Durability Test.

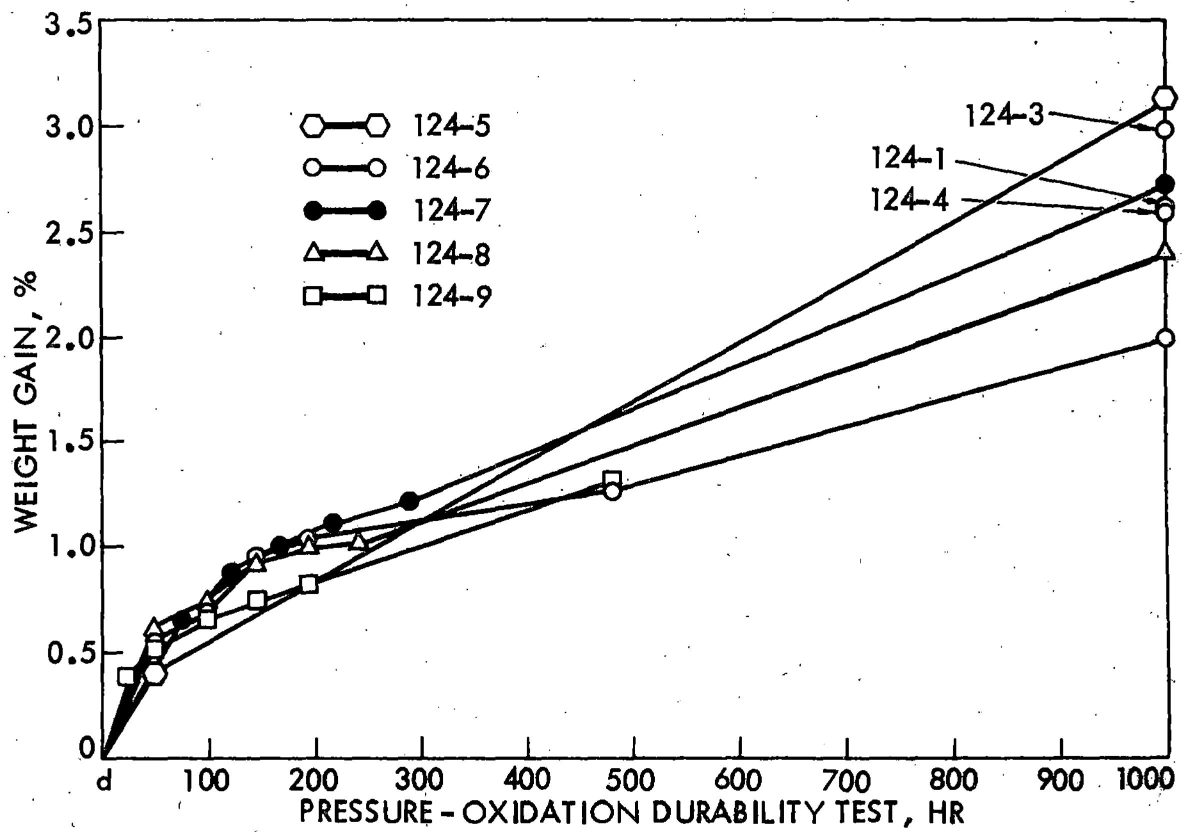

Fig. 6. Oxidation of asphalt during Iowa Durability Test as measured by weight change.

V. DENSITY — VOIDS CHANGE OF THE PAVEMENTS

Changes of density and voids of the pavements are being studied because they have been found to affect the rate of asphalt hardening, fatigue resistance and water susceptibility of the mixture, and thus they are two relevant factors to be considered in the final analysis of asphalt durability, correlation and critical asphalt property values.

Bulk specific gravity (d) is determined by water displacement method at 77°F (25°C). Percent voids (V) is calculated from bulk specific gravity and maximum theoretical specific gravity (D) data. The latter is determined following Rice's method, essentially that described in the ASTM D2041-64T. Values of bulk specific gravity, maximum specific gravity and percent voids for both specimens in wheeltracks (WT) and between wheeltracks (BWT) up to 24 mo are tabulated in Table 5. Data of bulk specific gravity represent averages of 4 to 6 specimens and those of maximum specific gravity were results of duplicate determinations. Changes in density were also plotted in Figs.7a and 7b, and changes in void contents were plotted in Figs. 8a and 8b. Examining the trends and comparing with designed mix data, as supplied by the Iowa State Highway Commission (Table 6), the following preliminary observations can be made:

1. There were no appreciable initial differences on density-voids values between specimens in wheeltracks and those between wheeltracks.
2. None of the 8 pavements reached as-built densities of the designed Marshall densities.
3. Pavements constructed during second period (Projects 6 — 9) of warmer weather approached higher density and lower voids relative

Table 5. Density - voids changes of the pavements.

	Age of pavement, mo									
	0		6		12		18		24	
	WT	BWT	WT	BWT	WT	BWT	WT	BWT	WT	BWT
1	d	2.220	2.240	-	2.294	2.288	2.303	2.313	2.324	2.292
	D	2.430	2.363	-	2.350	2.356	2.385	2.392	2.390	2.390
	V	8.64%	4.94%	-	2.38%	2.89%	3.44%	3.30%	2.76%	4.10%
2	d	2.144	2.146	2.152	2.170	2.171	2.188	2.178	2.198	2.202
	D	2.376	2.383	2.383	2.405	2.405	2.405	2.405	2.429	2.438
	V	9.76%	9.95%	9.69%	9.77%	9.73%	9.03%	9.44%	9.50%	9.68%
3	d	2.151	2.178	2.154	2.209	2.195	2.184	2.181		
	D	2.433	2.417	2.417	2.406	2.397	2.402	2.402		
	v	11.59%	9.89%	10.88%	8.19%	8.45%	9.08%	9.20%		
4	d	2.108	2.110	2.098	2.132	2.156	2.176	2.176	2.197	2.182
	D	2.404	2.377	2.377	2.397	2.362	2.384	2.384	2.408	2.403
	V	12.30%	11.23%	11.76%	11.06%	8.72%	8.72%	8.72%	8.76%	9.20%
6	d	2.309	2.373	2.325	2.377	2.333				
	D	2.443	2.487	2.464	2.461	2.461				
	V	5.49%	4.58%	5.46%	3.41%	5.20%				
7	d	2.114	2.241	2.238	2.260	2.249				
	D	2.274	2.382	2.381	2.406	2.406				
	V	7.04%	5.92%	6.00%	6.07%	6.50%				
8	d	2.214	2.232	2.194	2.292	2.260				
	D	2.373	2.394	2.394	2.426	2.426				
	V	6.70%	6.77%	8.35%	5.52%	6.84%				
9	d	2.303	2.356	2.337	2.350	2.363				
	D	2.425	2.448	2.433	2.436	2.436				
	V	5.03%	3.76%	3.95%	3.53%	3.00%				

d: Bulk specific gravity 77/77^{o}F

D: Maximum theoretical specific gravity, 77/77^{o}F

V: Percent air voids

Table 6. Laboratory mix design data.

Project number	AC content, %	Marshall density, gm/cc	Voids, %
1	7.50	2.275	6.8
2	6.25	2.275	6.1
3	6.25	2.260	6.6
4	7.50	2.180	9.3
6	5.00	2.326	6.8
7	7.00	2.291	6.1
8	5.25	2.311	3.5
9	5.50	2.355	5.8

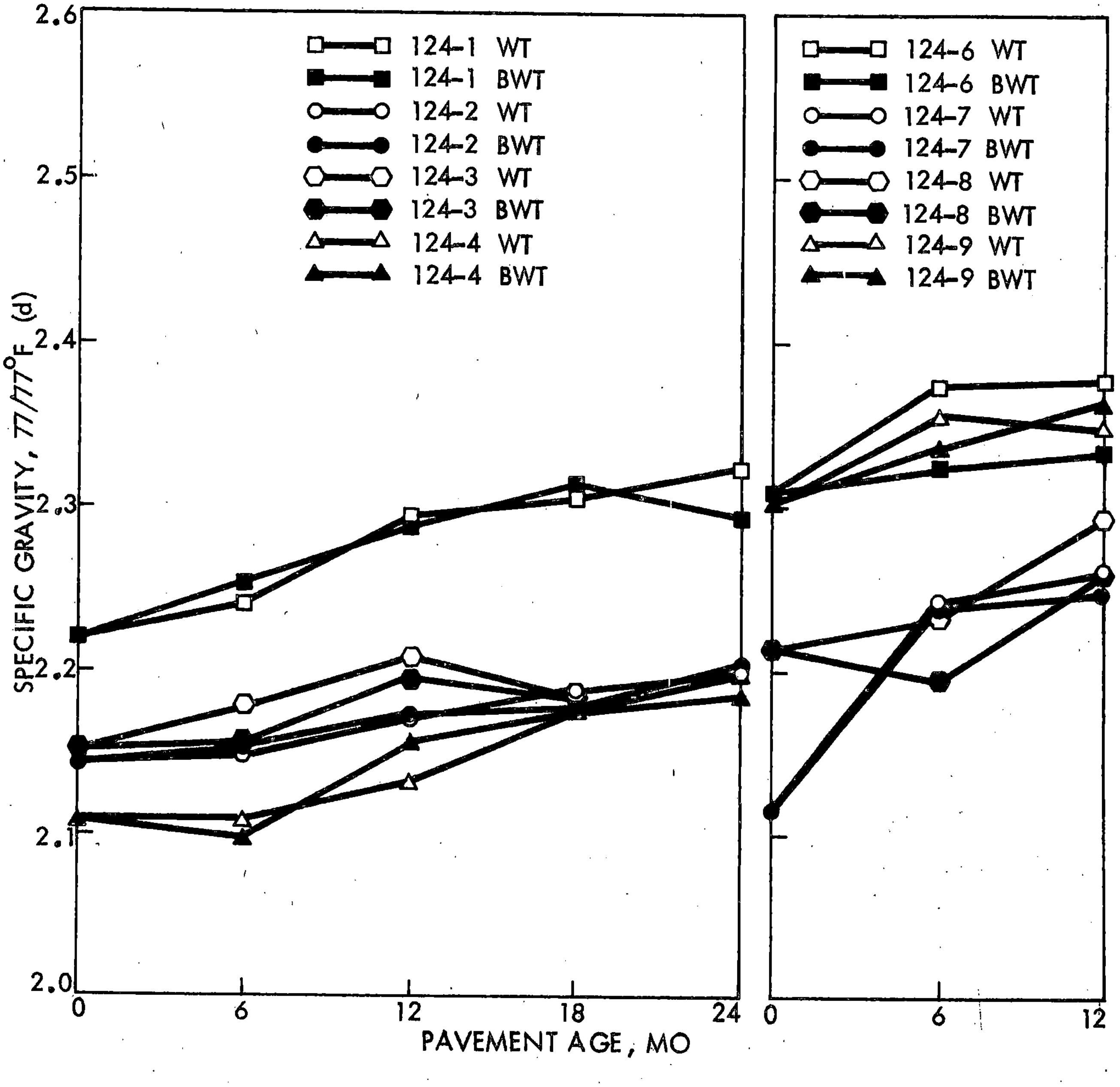

Fig. 7. Density change of the pavements.

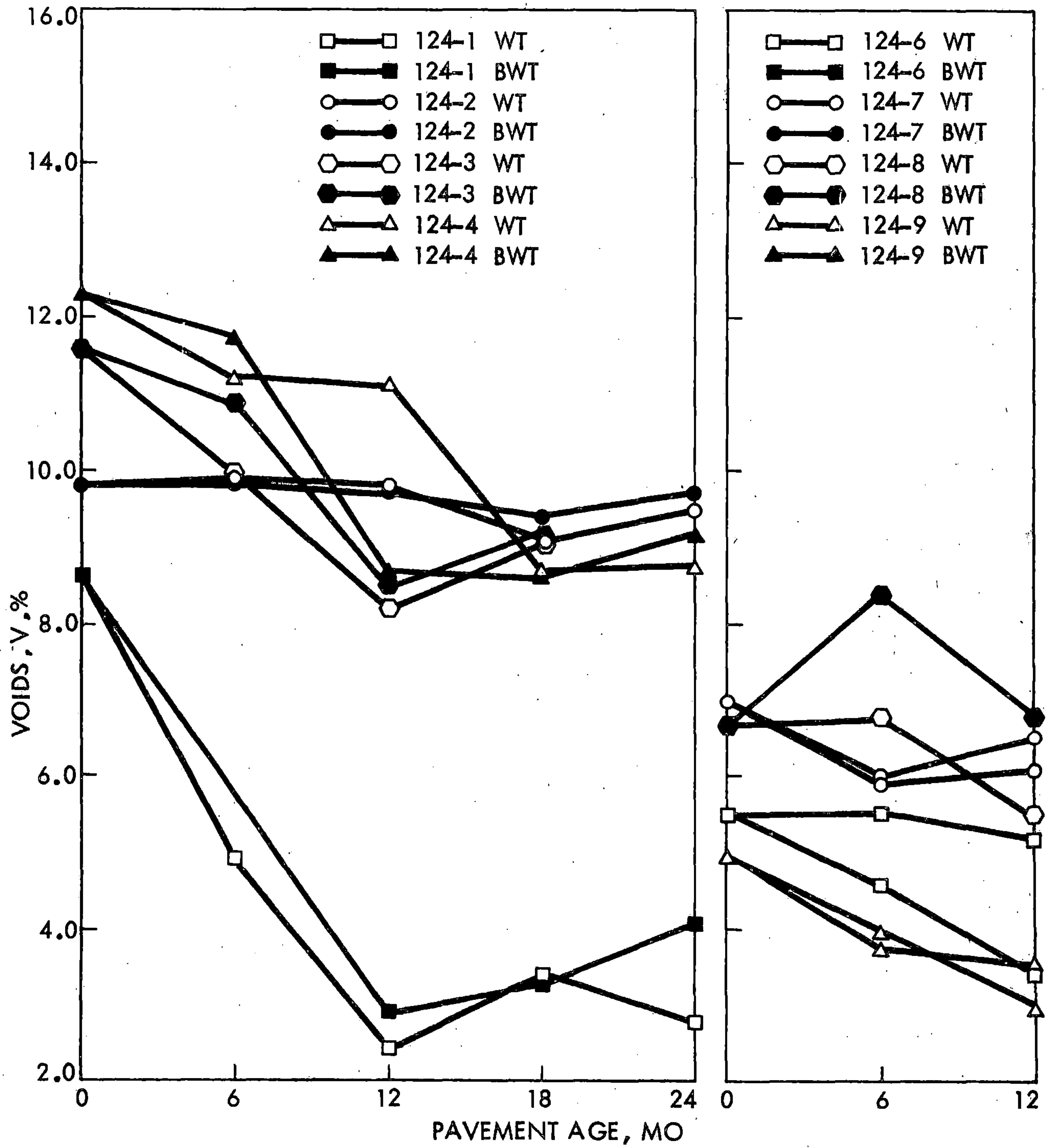

Fig. 8. Voids change of the pavements.

to design density and voids than those built during the first period (Projects 1 – 4).

4. Half of the projects reached design density and voids at the end of one year or after one summer of traffic densification.

5. Changes of density and voids appeared to have leveled off after 1 yr of traffic compaction.

6. Due to the inherent problems in sampling, inexactness of density determinations and sensitive dependence of voids on density, trends of density and voids with respect to time lack desired consistency; however, it can be stated that, after traffic, the areas in wheeltracks have displayed greater densification than areas between wheeltracks.

VI. BEHAVIOR OF ASPHALTS IN THE PAVEMENTS

Eight asphalt cements and paving projects using these asphalts were selected by the Iowa Highway Commission engineers. The project location and mix type were carefully selected to represent different types of mixes used in Iowa and different weathering conditions throughout Iowa. The time of the pavement construction was also well distributed, from October 1967 to July 1968. (See Table 2 and Fig. 1).

Physical and chemical changes in asphalts in pavements were determined for 18 mo on 4 projects and for 12 mo on the other 4 projects. The trend or shape of the time-property curves (e. g., time-penetration curves) can not be precisely defined at this time, due to short correlation time and limited points. Nevertheless, the decreasing rate of change with time can be observed, which indicates the hyperbolic nature of the curves (Fig. 9). It is expected that the shapes of these time-rate-of-change curves should be definable after 30 – 36 mo.

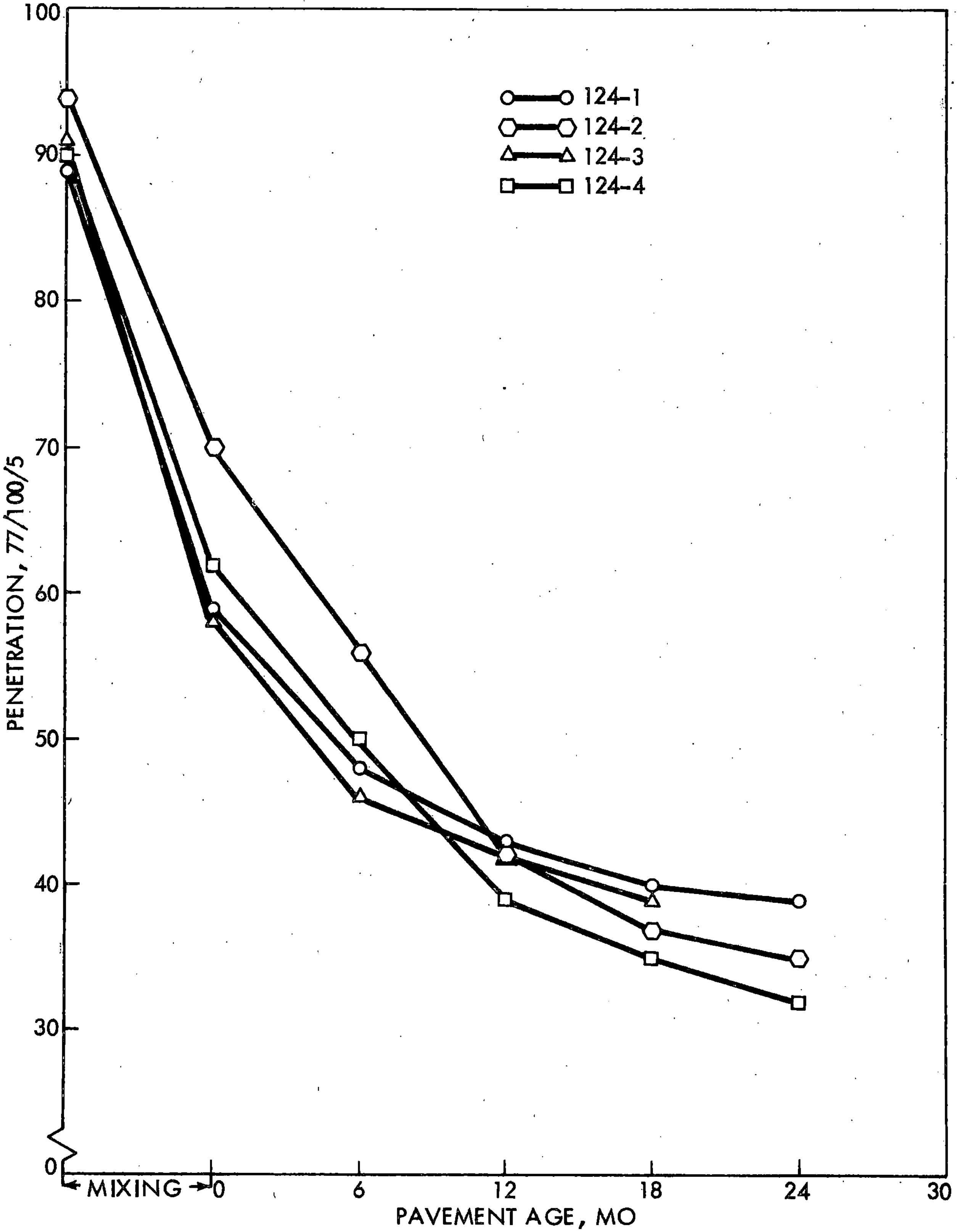

Fig. 9. Penetration change of the asphalt in service.

VII. PRELIMINARY CORRELATION AND ENGINEERING IMPLICATIONS

It has been pointed out that a correlation curve between the laboratory durability test and pavement service aging cannot be established until the behavior curves of asphalts in pavements are defined, which would require data at least extended to 30 – 36 mo. However, based on available data on four projects and penetration equivalency, a preliminary correlation curve is constructed (Fig. 10). As expected, there is a dispersion of points from different projects due to differences in asphalt, asphalt content, voids, etc. However, a general trend is obvious and a time-equivalency curve is definable. Based on this preliminary curve, a treatment of 24 hr in the Iowa Durability Test is approximately equal to 24 mo of pavement service time for Iowa conditions and, if this trend continues, an exposure of 40 hr in the Iowa Durability Test would produce aging of asphalts equivalent to 5 yr in the field under Iowa conditions. It is to be noted that the shape of the curve may change when all projects are considered and more data are collected. It should be noted also that when different criteria are used, the correlation curve may also be different. It is expected that a final weighted correlation curve will be established with consideration of all projects and all critical parameters at the end of 48-mo pavement service studies.

The ultimate goal of HR-124 is the establishment of functional approach asphalt specifications with more emphasis on durability. For engineering application of the Iowa Durability Test and the final correlation curve, the following approach and procedures are suggested:

1. Correlation or time-equivalency curve (Fig. 10) can be constructed based on selected parameters such as viscosity, ductility,

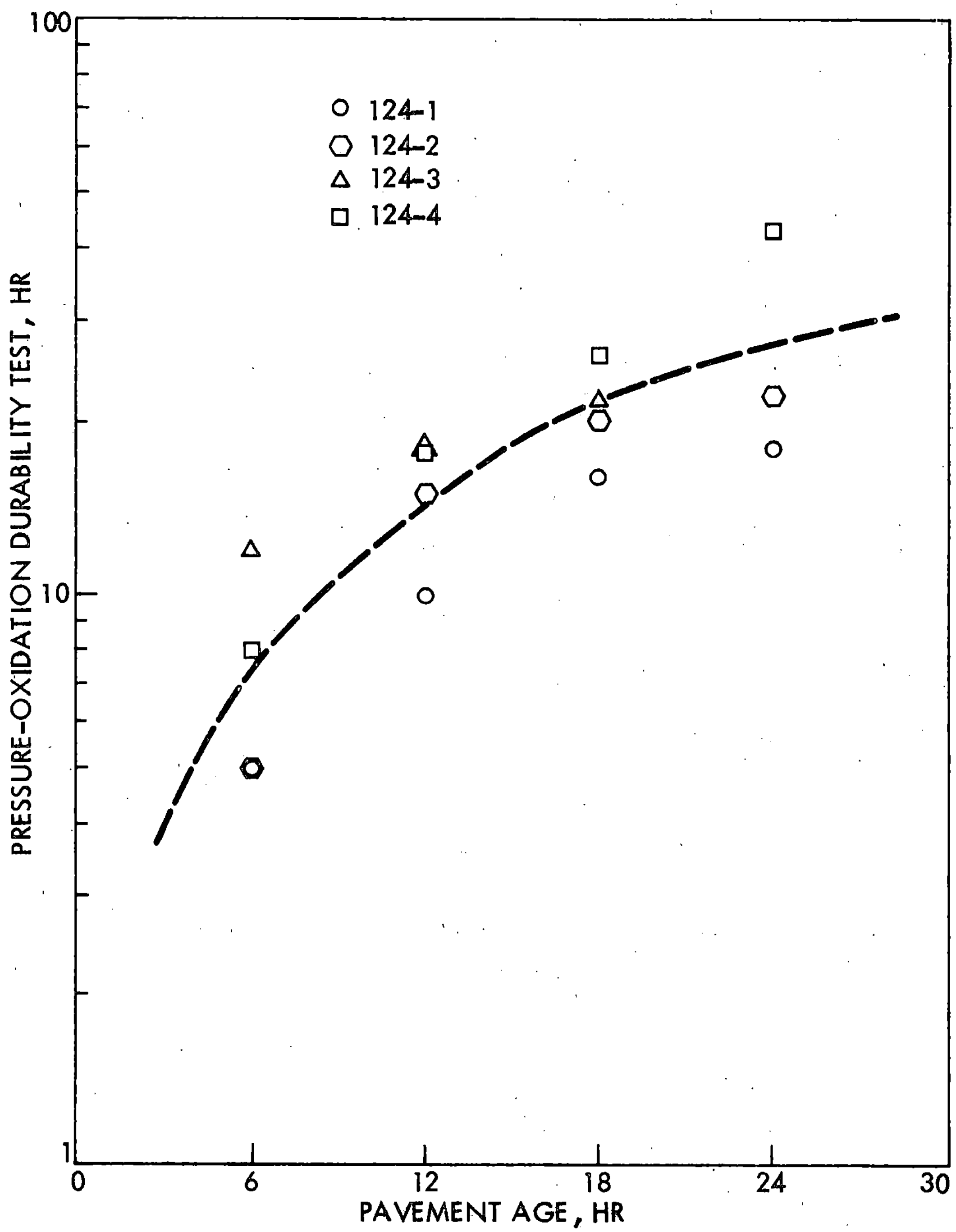

Fig. 10. Preliminary correlation curve based on penetration criteria.

Table 7. Correlation of pavement condition with physical properties of recovered bitumen[a].

Data source	Pavement locations	Pavement condition	Tests of recovered bitumen: Penetration (77°F)	Ductility 77°F (cm)	Softening point (°F)
Shattuck	Detroit, Mich.	Very good	20+	50+	-
		Good	20+	25+	-
		Badly cracked	20-	25-	-
Thomas	Minn.	Good[b]	41	-	-
		Fair[b]	26	-	-
		Poor[b]	20	-	-
Hubbard and Gollomb	Ohio, Mich., N.Y., Ind., D. Col.	Sound	30+	-	-
		Prone to crack	30-	-	-
		Cracking type	20-	-	-
Vokac	Ohio, Pa., Md., Va., Mo., Ill., Ind., Mich., N.Y., N.J., D. Col.	Sound	25+	24+	-
		Prone to crack	18-25	4-24	160-
		Cracking type	18-	4-	160+
Powers	Arizona	Good	10+	10+	160-
		Cracked	10-	10-	160+
Pub. Rds. Admin.	Cuba	Good[b]	9	1.5	199
		Cracked[b]	5	0.5	217

[a] After Lewis and Welborn. Proc. Assoc. Asph. Pav. Tech. 17: 228 (1948).

[b] Average values.

Table 8. Summary of critical recovered asphalt properties for acceptable performance of asphaltic surfacing .

Reference	Penetration (0.1 mm)	Ductility 77°F, 5 cm	Ductility 45°F 1 cm	Softening Point (°F)	Viscosity (poises)
Dow	38-90				
Lewis and Welborn	< 20	< 10		> 150	
Hubbard and Gollomb	< 20				
Shattuck	< 20	< 25			
Thomas	< 20				
Hubbard	< 20				
Vokac	< 25	< 24		> 160	
Powers	< 10	< 10		> 160	
Halstead[a]	50	100			
	25	10			
Clark		< 15			
Doyle			< 8		
Hveem *et al.*[b]		16		151	
Simpson *et al.*					10^7-10^8 [b]

[a] Critical penetration (arithmetic)-ductility (logarithms) relationship as established by line connecting these values.

[b] Based on results of Zaca-Wigmore test project.

penetration, etc. or a combination of them using data collected during correlation studies.

2. Critical values of selected significant properties such as values in Tables 7 and 8 will be established based on pavement performance observations and information from other studies.

3. Functional specifications can be expressed in terms of the durability test by selecting design life which determine durability test duration (from Fig. 10) and setting limiting values of critical properties from Table 7 or 8. For example, if design life is selected as 60 mo, from Fig. 10, durability test duration will be 40 hr. If penetration of 20 is considered as the critical value, then the specification can be read as: penetration of asphalt after 40 hr of durability test exposure should not be less than 20.

Note that this proposed approach of durability test application has advantages over all current specifications, including the Asphalt Institute's and California's research specifications. It not only specifies durability, it also considers design life (balanced design) and it can predict behavior of asphalt throughout pavement life.

VIII. FUTURE WORK

In order to continue the collection of data on changes in asphalts in the eight pavements toward completion of correlation between behavior of asphalts in the Iowa Durability Test and in pavement service in terms of acceleration factors or time-equivalency curves so that the laboratory durability test procedures can ultimately be incorporated in specifications as quality criteria and used to predict the durability of asphalt under Iowa conditions in reasonably exact terms, a proposal for extension of HR-124 for two more years (1969 – 1971) was submitted to and approved by the Iowa Highway Research Board in October 1969. This will bring the total correlation to 48 mo, which is believed to be sufficient to define and establish the correlation curve.

The procedures and approach in examining physical-chemical changes of asphalts in the eight projects at 6-mo intervals will be the same as in the first 24 mo except that:

- field observation and assessment of pavement performance conditions will be made with the Iowa Highway Commission engineers at 6-mo intervals,
- infrared spectroscopic analysis will be made on both new and old samples to determine the relationship between asphalt durability and carbonyl index change as suggested by Greenfeld and Wright[12],
- chemical analysis of some asphalts will be made as suggested by Rostler and White[13] and Halstead <u>et al.</u>[14] to determine the relationship between asphalt durability and $(N + A_1)(A_2 + P)$, and

- limited work toward establishment of "critical" value or values of critical property or properties of asphalt under Iowa construction, traffic and climate conditions.

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