NATIONAL BUREAU OF STANDARDS REPORT

5422

INTERIM REPORT ON PROJECT 4847
STANDARDS FOR BUILT-UP ROOFS

by

W. C. Cullen
L. R. Kleinschmidt
H. R. Snoke

NBS

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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Floor, Roof and Wall Coverings Section
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I. INTRODUCTION

This report gives the results of field studies of 101 low-pitched, mineral-surfaced, asphalt and coal-tar pitch, built-up roofs in the Western, Mid-Western and Eastern States, and of laboratory studies of 14 asphalts furnished by roofing manufacturers as representative of materials sold by them for the construction of dead-level, mineral-surfaced, built-up roofs. Three samples of coal-tar pitch, furnished by two producers as representing eastern and western production of one and eastern production of the other, were included in some of the laboratory studies.

While this was primarily a study of asphalts intended for use in the construction of low-pitched, mineral-surfaced, built-up roofs, it was considered essential for proper evaluation to compare the behavior of asphalt and coal-tar-pitch roofs in the field and to compare the results of laboratory tests on representative samples of asphalt and coal-tar pitch where such tests were applicable to both materials.

Complete details of the field studies are described in Appendix 1 of this report and of the laboratory studies in Appendix 2. Appendix 3 contains a discussion of the requirements in the proposed specification for asphalts for low-slope roofs.

II. SUMMARY AND CONCLUSIONS

A. Field Studies

Figure 1 shows graphically the results of the field studies. In this and subsequent graphs, length of service is plotted against 100 minus the numerical rating; consequently, the lower the slope of the curve, the better the performance rating. Figures 2 to 7 and Table 2, Appendix 1, show details of the field studies in the various areas. No data are shown for coal-tar-pitch roofs on the West Coast because coal-tar pitch has become generally available there only recently.

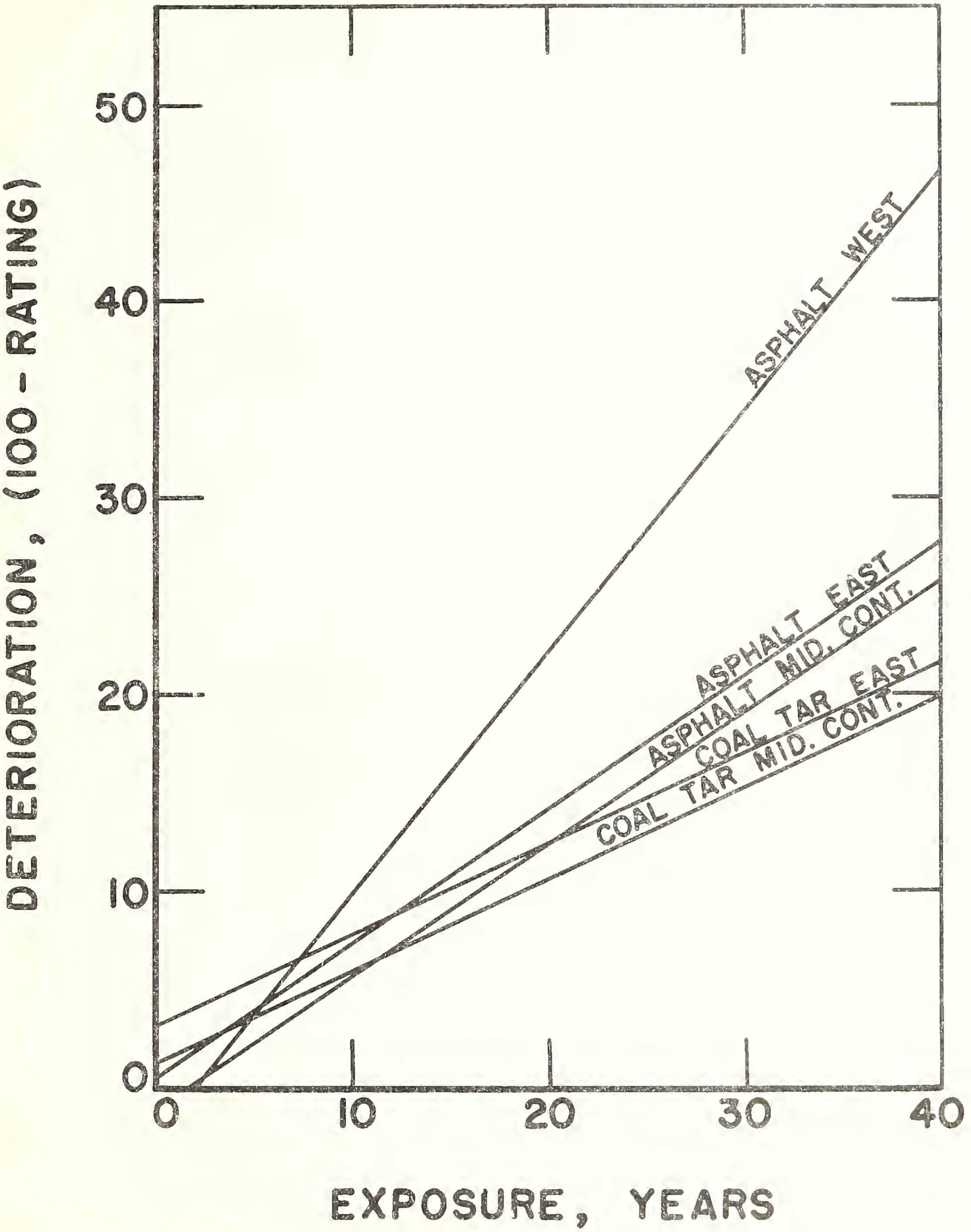


FIGURE 1. RESULTS OF FIELD STUDY.

Conclusions drawn from the field studies were:

1. Numerous low-pitched, mineral-surfaced, asphalt and coal-tar pitch, built-up roofs were observed in good, serviceable condition in all areas after more than twenty years of exposure. Roofs of both types thirty years old were not uncommon.

2. Coal-tar-pitch roofs were rated slightly higher, generally, than asphalt roofs, in the Eastern and Mid-Western States. This was most apparent in roofs exposed more than twenty years.

3. Asphalt roofs in the Eastern and Mid-Western States were rated significantly higher than those in the Western States. Asphalt roofs in the Mid-Western States were rated slightly higher than those in the Eastern States.

4. It is our strong opinion that good service from any conventional, bituminous, built-up roof depends more on initial workmanship and subsequent maintenance than on the materials that are used. This conclusion is not entirely a product of this investigation, but is based, rather, on considerable field experience.

B. Laboratory Studies

In any appraisal of the results of these laboratory studies, it should be borne in mind that specifications for asphalt and coal-tar pitch have always been based largely on physical characteristics. Through the years specifications have evolved which insure materials that can be handled well, will stay in place on a roof, etc., but which give no assurance of the "quality" of the product, if one defines "quality" as service on a roof. That there is no general agreement, even as regards physical characteristics, is shown by the wide variations in the properties of the materials submitted for these tests.

We have made the standard tests to determine properties that are usually listed in purchase specifications. In addition, we have made other tests, many of which show

differences among the materials tested. Some of these tests are reported here for the first time. We would expect that different bituminous technologists would interpret the results of some of these tests differently.

Conclusions drawn from the laboratory studies are:

1. Types of Asphalt: Two types of asphalt are being furnished by roofing manufacturers for low-slope, mineral-surfaced, built-up roofs. Type I is largely characteristic of asphalt produced for many years for this use. Type II asphalt is characterized chiefly by high ductility at 77°F, embrittlement at 40°F, and a higher susceptibility factor than Type I asphalt. Eleven samples of Type I and three samples of Type II asphalt were furnished by manufacturers for this study.

2. Compliance with current specifications: All specimens of Type I asphalt submitted complied with the requirements of Federal Specification SS-A-666, Type 1, Class A, for surfaced, built-up roofs, except in minor respects, and all complied with American Society for Testing Materials Specification D312, "Mineral-Surfaced Flat".

   The three Type II asphalts complied with Federal Specification SS-A-666, Type 1, Class A, except for penetration at 32°F and 77°F. They complied with A.S.T.M. Specification D312, "Mineral-Surfaced Flat", except that for two of them the penetration at 77°F was lower than the minimum specified.

   The three samples of coal-tar pitch complied with Federal Specification R-P-381, Type I, for built-up roofing.

3. Component distribution in asphalts: The component distribution in asphalts cannot be used alone as a criterion of quality.

4. Deterioration tests:

   4.1 Accelerated weathering tests (Exposure to heat, light and water):

   4.1.1 Hardening: Both asphalts and coal-tar pitches showed marked hardening as evidenced by increases in softening point.

4.1.2 Shrinkage: The asphalts showed varying amounts of shrinkage on long exposure. The two that shrank most gave positive "spot" tests and contained the largest percentage of matter insoluble in n-pentane. The coal-tar pitches showed no detectable shrinkage on long exposure.

4.1.3 Matter insoluble in n-pentane: All asphalts showed marked increases in matter insoluble in n-pentane on long exposure.

4.1.4 Photochemical reactions: Water-soluble acidic compounds formed from the asphalts were markedly greater than those from the pitches. The relatively large amounts of acidic materials formed when asphalts are exposed to light and moisture emphasize the very important role of mineral-surfacing materials in built-up roofs.

4.1.5 Self-healing characteristics: Both asphalts and coal-tar pitches exhibited self-healing properties after 4500 hours of accelerated weathering. The coal-tar-pitch specimens healed more rapidly than the asphalt specimens in all cases. Variations in the rate of self-healing among the asphalts could not be attributed to the type of asphalt.

4.2 Outdoor weathering tests:

4.2.1 Hardening: Both asphalts and coal-tar pitches hardened during outdoor exposures as evidenced by increases in softening point.

4.2.2 Self-healing: Both asphalt and coal-tar pitch exhibited self-healing properties during summer exposures; neither showed self-healing properties during winter exposures.

4.3 Heat susceptibility tests (Exposure to heat along):

4.3.1 Component distribution: Changes in the component distribution of the asphalts when exposed to heat alone were essentially the same as those found by exposure to accelerated weathering.

4.4 Water absorption tests:

4.4.1 Long-time immersion: The amounts of water absorbed by the asphalts in long-time immersion tests varied widely, but were not indicative of the type or source of asphalt. The water absorption of the coal-tar pitches fell within the range of the asphalts that absorbed the least.

From 0.05 to 0.1 ml of o.01 N alkali solution was required to neutralize material leached from each of the asphalts and the coal-tar pitch. This is in marked contrast to the results of the accelerated weathering test (Exposure 3) where from 20 to 80 ml of 0.01 N alkali were required to neutralize the material formed from different asphalt specimens during 900 hours of exposure. Only 1.6 ml of 0.01 N alkali were required to neutralize material formed from the different coal-tar pitches exposed similarly.

5. Specification: Laboratory examination of the asphalts furnished indicates the desirability of a specification covering two types of asphalt, with requirements that are more restrictive than those of the Federal and A.S.T.M. specifications. A study of specifications furnished by manufacturers lends weight to this conclusion.

III. PROPOSED SPECIFICATION FOR ASPHALT FOR LOW-SLOPE ROOFS

Table 1 lists chemical and physical requirements of two types of asphalt for use in the construction of mineral-surfaced, built-up roofs on slopes up to one inch per foot. Type I asphalts are characteristic of low-slope asphalts covered by Federal and A.S.T.M. specifications, but the requirements in the proposed specification are more restrictive than in either of those specifications. Type II asphalts are processed to approximate, initially, some of the physical characteristics of coal-tar pitch, notably, high ductility at 77°F.

TABLE 1. PROPOSED SPECIFICATION FOR ASPHALT FOR LOW-SLOPE ROOFS.

Type	I Min.	I Max.	II Min.	II Max.
Softening point (R & B),[1] °F	135	145	135	145
Ductility at 77°F[2], 5 cm/ min., cm.	15		100	
Ductility at 40°F, 0.25 cm/ min., cm.	3		---	
Penetration[3] at				
32°F, 200 g, 60 sec.	10		5	
77°F, 100 g, 5 sec.	25	60	15	25
115°F, 50 g, 5 sec.	100		90	
Weight loss, 5 hrs. at 325°F[4], %		0.5		0.5
Penetration of residue at 77°F, percent of original	85		75	
Bitumen soluble in CCl_4[5], %	99		99	
Bitumen insoluble in n-pentane[6], %		30		30
Flow test at 140°F, 5 hrs.[7], cm.	2	9	2	9
Spot test[8]		Negative		Negative
Susceptibility factor[9]	2		5	
Flash point, C.O.C.[10], °F	375		375	

(Methods of Tests)

1/ A.S.T.M. D36.

2/ A.S.T.M. D113.

3/ A.S.T.M. D5.

4/ A.S.T.M. D6.

5/ A.S.T.M. D4.

6/ N.B.S. RP-2577.

7/ F.S. SS-S-164 (Modified).

8/ A.A.S.H.O. T102 (Naphtha).

9/ $\frac{\text{Pen. at 115°F - Pen. at 32°F}}{\text{Pen. at 77°F}}$

10/ A.S.T.M. D92.

The proposed specification contains some requirements that are not normally included in specifications for asphalt for use in the construction of built-up roofs, namely, matter insoluble in n-pentane, spot test, susceptibility factor, and flow test, applicable to both types. In addition, a minimum requirement for ductility at 40°F is included for Type I asphalt. The significance of the specification requirements is discussed in Appendix 3. under "Discussion of Requirements in the Proposed Specification for Asphalts for Low-Slope Roofs".

Type I asphalt should be readily available in all areas. Type II asphalts have been available nationally for a relatively short time. Consequently, most of the long-service roofs reported in the field study were of Type I asphalt.

IV. ACKNOWLEDGMENT

The authors acknowledge the assistance of the Asphalt Roofing Industry Bureau in furnishing the list of older asphalt roofs of known history, and of manufacturers of asphalt and coal-tar-pitch roofings for the locations of roofs, assistance in making inspections, and the samples of asphalt and coal-tar pitch that they furnished. The assistance of Dr. William S. Connor of the Applied Mathematics Division, N.B.S., in the statistical aspects of the field survey is also acknowledged.

APPENDIX 1. FIELD STUDIES OF BUILT-UP ROOFS.

1. Introduction

In planning the field studies of built-up roofs, the following criteria were established as of primary importance:

a) The inspection areas should represent the widely different climatic conditions in the United States.

b) The asphalt roofs should represent the principal sources of asphalt used in this country.

c) Only roofs of known history should be examined.

d) The system for selecting roofs for inspection should be entirely unbiased.

e) The system of rating the condition of a roof should be on a numerical basis.

It was realized of course that many factors other than the quality of the bitumen must be considered in evaluating the performance of built-up roofs and that serious defects may occur that are in no sense related to the quality or kind of bitumen used. For example, it is our opinion that more premature roof failures are caused by faulty workmanship than by faulty materials. Proper maintenance is also of great importance in determining the service of any built-up roof.

While the principal purpose of this field study was to evaluate the performance of mineral-surfaced, built-up roofs on low slopes, other roofs of particular interest were inspected where available. Among these were: smooth-surfaced, built-up roofs; built-up roofs in which glass mat replaced the conventional felts; double-surfaced, built-up roofs; and one application of an elastomeric sheet (Neoprene) on a concrete deck. This report is confined to conventional mineral-surfaced, built-up roofs.

2. Selection of Roofs for Inspection

In order to obtain roofs of known history for inspection, it was necessary to rely upon the roofing industry. The Asphalt Roofing Industry Bureau furnished a list of 86 mineral-surfaced, asphalt, built-up roofs known to be 10 years old or older. Individual manufacturers of asphalt roofings who submitted asphalt specimens furnished lists of more than 400 such roofs applied with the asphalts they are selling currently, all less than two years old. Manufacturers of coal-tar pitch furnished the locations of coal-tar pitch roofs in the inspection areas. Frequently the local representatives of the roofing manufacturers and roofing contractors were able to point to older roofs of known history.

As it was obviously impracticable to inspect all of the roofs suggested, the initial list of older roofs was divided into three geographical areas, East Coast, Mid-West, and West Coast, and each of these groups were broken down into seven sub-groups, by age, as follows: Less than 10 years, 10 to 12, 13 to 15, 16 to 18, 19 to 21, 22 to 24, and 25 years and older. Actual roofs to be inspected were selected by lot from each sub-group. Where it was found that a roof selected had been destroyed or reroofed, an attempt was made to find a replacement. One-hundred-one mineral-surfaced, built-up roofs were inspected in this study, distributed as shown in Table 2.

TABLE 2. DISTRIBUTION OF ROOFS INSPECTED

	ASPHALT ROOFS		
Location:	East Coast	Mid-West	West Coast
Age, Yrs.	No.	No.	No.
10 or less	2	3	5
10 to 20	11	10	4
20 to 30	5	9	8
+30			1
	COAL-TAR-PITCH ROOFS		
Age, Yrs.	No.	No.	No.
10 or less	6	7	
10 to 20	10	5	
20 to 30	6	7	
+30	1	1	

3. Areas Included in the Studies

Inspections were made in 23 cities in 16 States and the District of Columbia, as follows:

1. Seattle and Tacoma, Washington.
2. San Francisco and Los Angeles, California.
3. Albuquerque, New Mexico.
4. El Paso, Texas.
5. Kansas City, Missouri.
6. Kansas City and Wichita, Kansas.
7. New Orleans, Louisiana.
8. Oklahoma City, Oklahoma.
9. Jacksonville, Florida.
10. Atlanta and Macon, Georgia.
11. Columbia, South Carolina.
12. Gastonia and Red Springs, North Carolina.
13. Washington, D. C.
14. Baltimore, Maryland.
15. Marion and Columbus, Ohio.
16. Philadelphia, Pennsylvania.
17. New York, New York.

4. Methods of Inspecting and Rating Roofs

Practically all roofs were inspected in company with a representative of the manufacturer of the roofing materials and, frequently, with the owner or manager of the building.

In order to secure the greatest possible uniformity in the inspections a form was developed which included the history of the roof and a check list of the elements that are important in a built-up roof. Each element was rated numerically in accordance with its importance as a factor in determining the condition of the roof. A copy of the inspection form with the ratings assigned to the various elements follows:

ROOF INSPECTION FORM

Rating _____
Date ________________

Historical

1. Building ________________ 2. Location ____________________

3. Used for ________________ 4. Year Applied _______________

5. Slope ______ 6. Sunshine Index _____ 7. Rainfall Index ___

8. Roofing Contractor ____

9. Manufacturer ______________ 10. Spec. No. ________________

11. Bonded: Yes ___ No ___ 10 ___ 15 ___ 20 ___

12. Deck: Wood ___ Concrete ___ Metal ___ Gypsum ___ Other ___

13. Type of Felt _____________ Ply: 2 __ 3 __ 4 __ 5 __

14. Insulation: Yes ___ No ___ Type ________ Thickness __________

15. Character of Bitumen: Asphalt ___ Coal Tar ___ Other ________

16. Source ______ Soft. Pt. ____ Pen. ____ Duct. ____ Other ______

17. Wt. of Bitumen per square: Between plies ____ Top pouring ___

18. History of Repairs____

Observations

1. Water Tightness: No leaks (8)___ Leaks with long-continued rain (4)___ Leaks every rain (0)___ Cause: ________________

2. Repaired Areas: None (10)___ Few (5)___ Many (0) ___
   Nature: ____

3. Blistering: None (12)___ Moderate (6)___ Serious (0) ______
   Between plies ______ Between deck & membrane _______
   Remarks: ____

4. Cracking of Felts: None (12)___ Few (6)___ Many (0)_______
   Remarks: ____

5. Surfacing: Adhesion to Bitumen: Good (4)_____ Fair (2)_____
   Poor (0) _____ Remarks: ______________________________

ROOF INSPECTION FORM (Continued)

6. Bare Areas: None (6)____ Few (3)____ Many (0)____

7. Felts: Good (8)____ Fair (4)____ Poor (0)____
Condition:____

8. Bitumen: Areas water standing or likely to stand:
   a) Condition: Good (4)_____ Fair (2) _____ Poor (0) _____
   b) Hardness: Soft (2)_____ Medium (1)_____ Hard (0)______
   c) Blisters, Pits, etc: None (2)_____ Few (1)____ Many (0)___
   d) Alligatored: None (6)_____ Moderately (3)____ Severely (0)__
   e) Cracking, Flaking, etc: None (6)___ Moderate (3)___
   Severe (0)___ Other Observations:_________________________

9. Bitumen: Areas water unlikely to stand:
   a) Condition: Good (4)_____ Fair (2)_____ Poor (0)_____
   b) Hardness: Soft (2)____ Medium (1)____ Hard (0)______
   c) Blisters, Pits, etc: None (2)___ Few (1) ____
   Many (0)_____
   d) Alligatored: None (6)_____ Moderately (3)_____
   Severely (0)_____
   e) Cracking, Flaking, etc: None (6)_____ Moderate (3)_____
   Severe (0)_____ Other Observations:________________________
   ____.

Under this system of rating, the various elements are weighted in accordance with the degree of deterioration. A roof with no defects would be rated 100 and the final rating of a roof is the sum of the ratings of the elements. This system was adopted as the method most likely to insure uniform procedures in the inspections. It is certain that the numerical ratings are more readily evaluated than mere descriptive terms.

This rating system might be used as a basis for determining the treatment necessary in long-time maintenance of built-up roofs, though, obviously, it would not apply universally. The following is a suggested application:

Deterioration	Action Indicated
0 - 5	Yearly inspection and preventive maintenance.
5 - 15	Yearly inspection and minor maintenance.
15 - 30	Extensive maintenance and minor repair.
30 - 50	Major repair.
Above 50	Reroofing probably indicated.

5. Results of Field Studies

The results of the field studies are shown graphically in Figures 2 to 6 inclusive. The age and deterioration of each roof inspected are shown by the points in Figures 2 to 6. The positions of the graphs were determined by the method of least squares and are presented as a means for comparing the general ratings of asphalt and coal-tar-pitch roofs in the different areas.

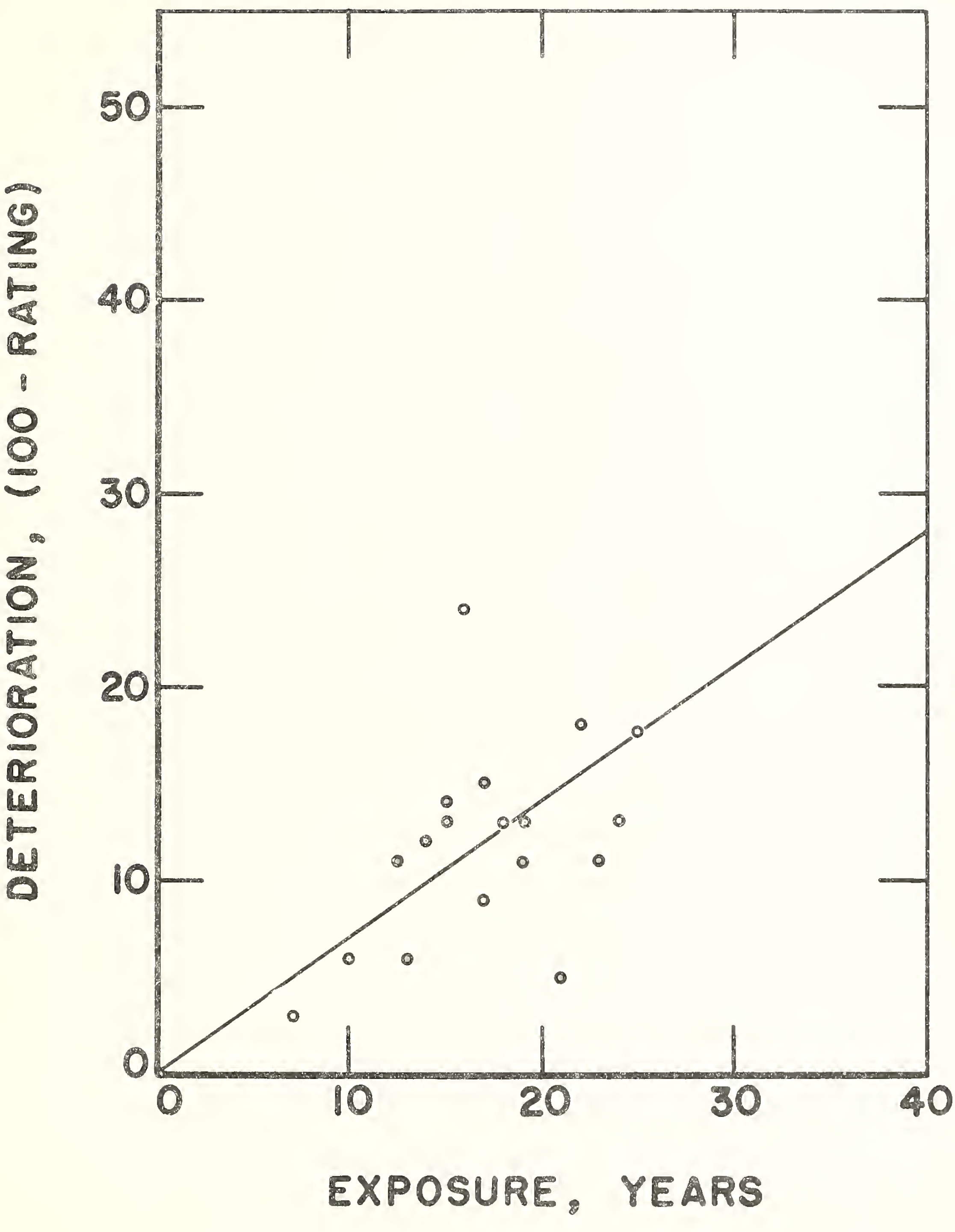


FIGURE 2. ASPHALT ROOFS - EASTERN STATES.

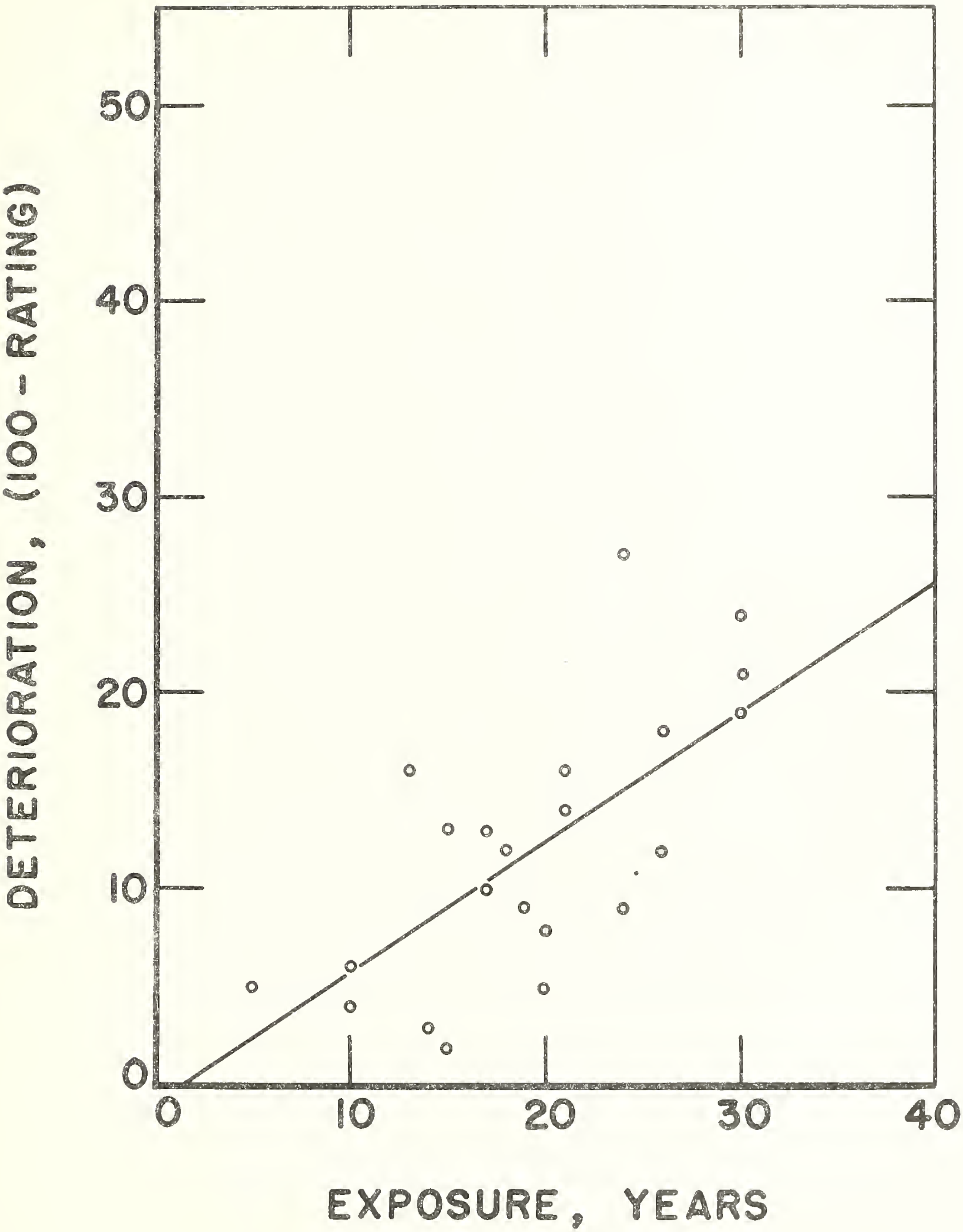


FIGURE 3. ASPHALT ROOFS - MID-WESTERN STATES.

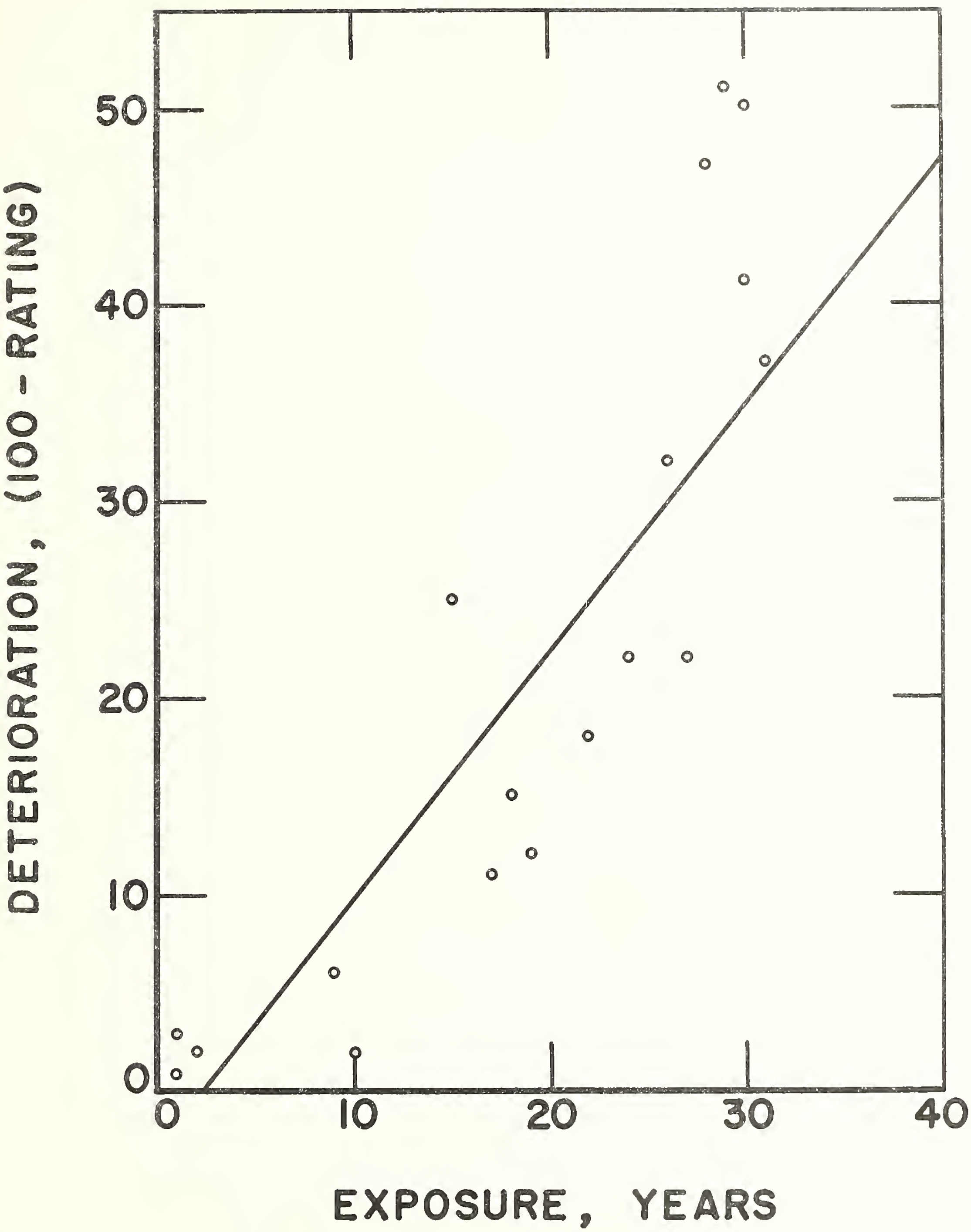


FIGURE 4. ASPHALT ROOFS - WEST COAST STATES.

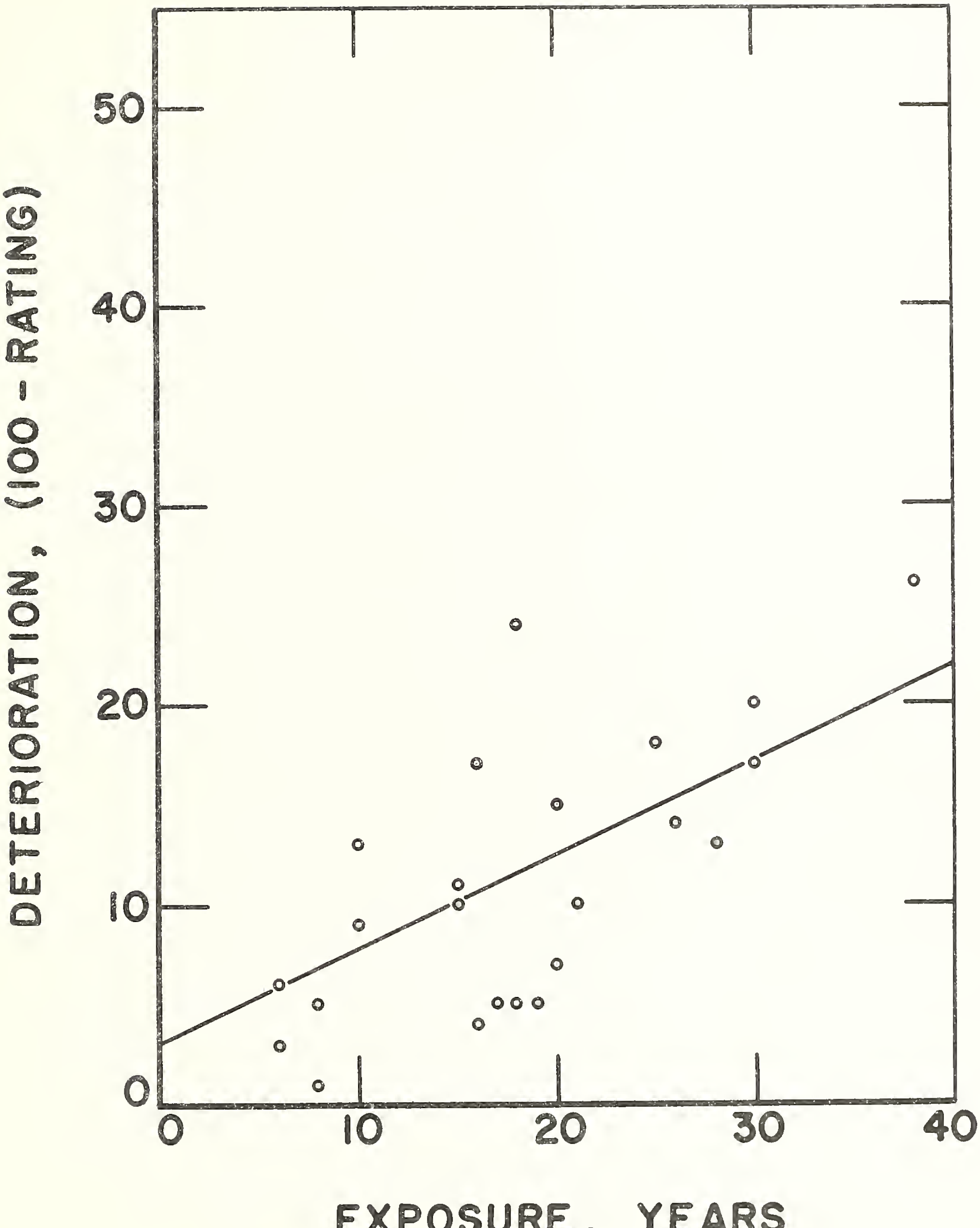


FIGURE 5. COAL-TAR PITCH ROOFS - EASTERN STATES.

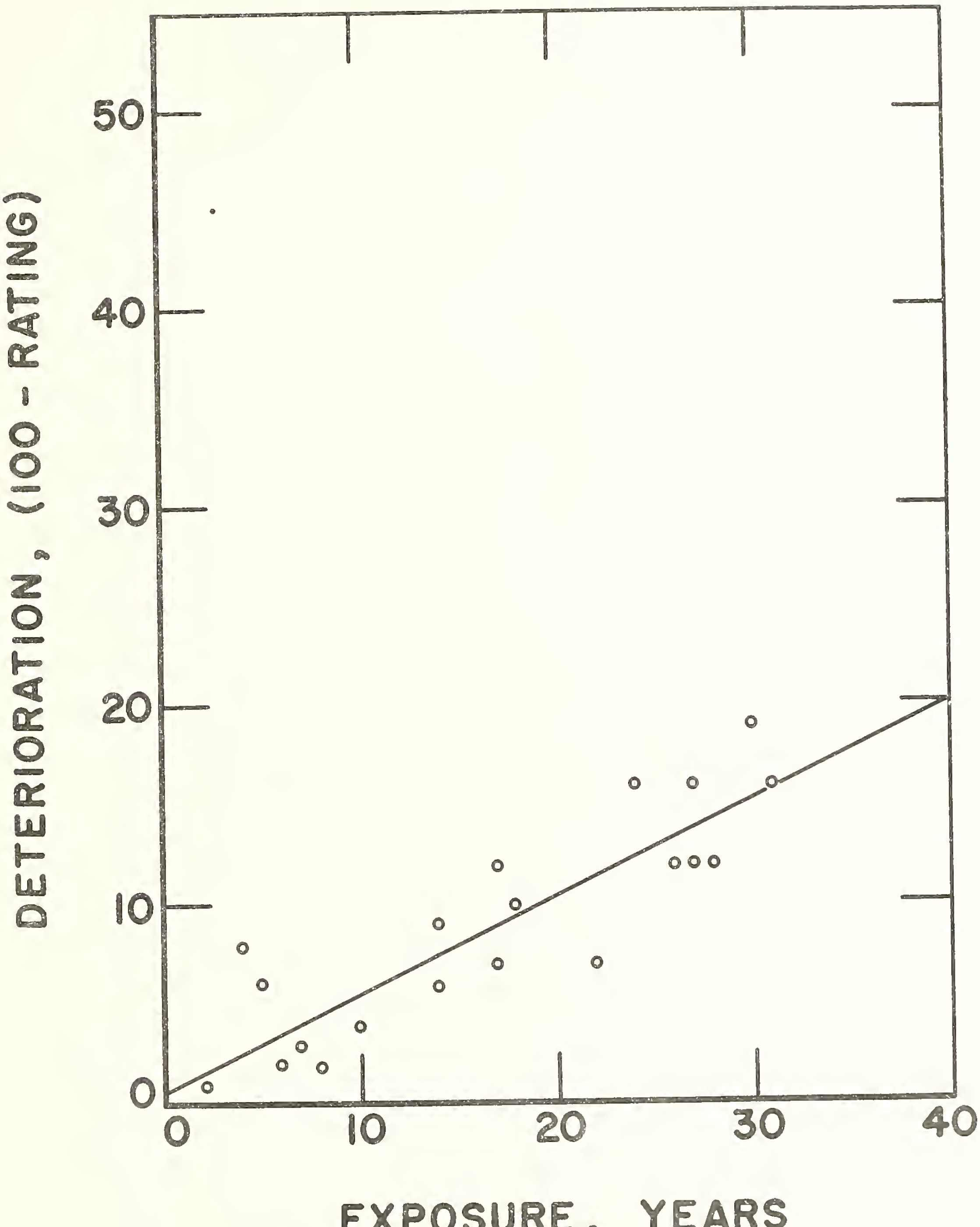


FIGURE 6. COAL-TAR PITCH ROOFS - MID-WESTERN STATES.

APPENDIX 2. LABORATORY STUDIES

1. Introduction

The 14 samples of asphalt and 3 samples of coal-tar pitch were examined in the laboratory by chemical and physical tests that are normally used to define the characteristics of asphalts and pitches used in built-up roofs. While limiting values under these tests may be used in specifications to control the physical characteristics of asphalts and pitches to insure materials that can be handled satisfactorily and will remain in place on a roof, they give no assurance of the quality of the product, when quality is considered in terms of long-time service on a roof.

Additional laboratory tests were made to determine differences among the asphalts and, wherever possible, between the asphalts and coal-tar pitches. In devising these tests, consideration was given to the conditions to which the bitumens in low-slope, mineral-surfaced, built-up roofs are subjected. Among these were exposure to elevated temperatures, alternate wetting and drying accompanied by sunlight, where water runs off quickly and where it evaporates in place. That a membrane may be cracked in cold weather by movements in the roof deck and subjected to long-continued exposure to water without sunlight were also considered.

Asphalts, when stored in closed containers at room temperature, are hardened and become more brittle, as evidenced by increases in softening point and in the amount of material insoluble in n-pentane, and decreases in the penetration values. These reactions are accelerated by elevated temperatures and will therefore apply in service to the moppings between felts as well as to the surface coatings.

Exposure of asphalt to the sun induces a photochemical reaction at the exposed surface, resulting in a thin surface film of light-degraded material which is largely soluble in water. When the slope of a roof is such that water runs off readily, this light-degraded material is largely removed, the coating loses weight and weathers to a dull, matt surface. On roofs where standing water evaporates in place, the light-degraded material is first dissolved and then deposited as a film when the water evaporates. On further drying, this film causes a shrinkage of the surface of the coating. So-called "alligatoring" may result from surface shrinking, with cracking the eventual result.

Cracked coating and mopping layers of bitumen may not be too important if the bitumen has the ability to "self-heal" in a reasonable time. Inability to self-heal could result in early failure of a membrane. The flow test and the special self-healing test are measures of the ability of a bitumen to self-heal.

Knowledge concerning the importance of the initial distribution of components or groups of compounds in an asphalt is insufficient for use in purchase specifications, but changes in the distribution of components, as in the increase of n-pentane insoluble matter, give an indication of the extent of degradation that has occurred.

The spot test usually appears in specifications for road asphalts as a control of quality.

2. Physical and Chemical Characteristics

In Table 3 are reported the source of the crudes from which the 14 asphalts included in this study were produced and the conventional physical and chemical characteristics normally used in purchase specifications. These data show that although the asphalts as a group fall within a narrow softening point range, other physical characteristics are markedly different. The ductilities and penetrations at 77°F and 32°F of samples 7, 12 and 13 indicate the desirability of including two types of material in a purchase specification. Consequently, asphalts 1, 2, 3, 4, 5, 6, 8, 9, 10, 11 and 14 will be referred to hereafter as Type I asphalts, and Nos. 7, 12 and 13 as Type II asphalts.

All Type I asphalts complied with the requirements of Federal Specification SS-A-666, Type I, Class A, for surfaced built-up roofs, except in minor respects, and all complied with American Society for Testing Materials Specification D312, "Mineral-Surfaced Flat".

The three Type II asphalts complied with Federal Specification SS-A-666, Type I, Class A, except for penetration at 32°F and 77°F. They complied with A.S.T.M. Specification D312, "Mineral-Surfaced Flat", except that for two of them, the penetration at 77°F was lower than the minimum specified.

TABLE 3. PHYSICAL AND CHEMICAL

Sample No.	Source of Crude	Soft.[1] Point (R & B)	Ductility[2] At 77°F 5 cm/min.	Ductility[2] At 40°F 0.25 cm/min.	Penetration[3] 77°F 100 g. 5 sec.	Penetration[3] 32°F 200 g. 60 sec.	Penetration[3] 115°F 50 g. 5 sec.
		°F	cm.	cm.			
1	Wyo.	145	9	3.3	42	22	117
2	"	148	13	3.5	26	9	90
3	"	146	23	4.0	29	11	108
4	Texas	158	7	4.6	30	17	73
5	Calif.	144	21	5.4	42	19	132
6	Mid. Con.	141	38	6.0	31	13	113
7	Calif.	143	150+	1.5[10]	16	5	95
8	Mid. Con.	149	14	4.2	31	12	89
9	Mid. Con.	142	11	3.6	46	22	122
10	Calif.	146	26	5.7	36	18	130
11	Ven.	148	23	4.6	28	15	98
12	Calif.	143	110	L1.0[10]	16	5	99
13	-----	140	150+	L1.0[10]	20	7	120
14	Mid. Con.	148	8	3.6	41	21	105

1/ A.S.T.M. D-36.
2/ A.S.T.M. D-113.
3/ A.S.T.M. D-5.

4/ $\frac{\text{Pen. at 115°F minus Pen. at 32°F}}{\text{Pen. at 77°F}}$

5/ F. S. SS-S-164 (Modified).

CHARACTERISTICS OF ASPHALTS.

Susceptibility Factor[4]	Flow at 120°F[5] 5 Hrs.	Heat Test, 5 Hrs.[6] at 325°F: Loss	Heat Test, 5 Hrs.[6] at 325°F: Penetration of Residue	Solubility: Carbon[7] Disulfide Soluble	Solubility: N-Pentane[8] Insoluble	Spot Test[9]: Naphtha
	cm.	%		%	%	
2.26	2.0	L0.1	40	99.7	30.0	Negative
3.11	1.7	"	25	99.7	29.8	"
3.34	2.2	"	27	99.8	28.5	"
1.86	0.7	"	28	----	34.4	"
2.69	1.9	"	38	----	35.9	Positive
3.23	3.6	"	29	----	24.2	Negative
5.62	3.6	"	13	99.8	29.7	"
2.48	1.2	"	30	99.8	30.6	"
2.18	1.0	"	43	99.9	27.4	"
3.12	1.4	"	36	99.9	41.5	Positive
2.96	1.4	"	28	99.9	31.5	Negative
5.87	2.4	"	14	99.7	29.7	"
5.65	4.4	"	15	----	24.8	"
2.05	1.0	"	39	----	27.2	"

6/ A.S.T.M. D-6.
7/ A.S.T.M. D-4.
8/ N.B.S. RP2577.
9/ A.A.S.H.O. T-102 (Naphtha).
10/ Fractures.

The three samples of coal-tar pitch complied with Federal Specification R-P-381, Type I, for built-up roofing.

The examination of these materials also indicated that the softening point and penetration ranges contained in both the Federal and A.S.T.M. specifications for asphalt can be reduced to insure a better defined material. Although a "susceptibility factor" and flow test are not included in either the Federal or A.S.T.M. specifications, these give a numerical evaluation of the "relatively susceptible asphalts" and "self-healing" properties, which the A.S.T.M. specification states are desirable.

3. Component Distribution

In Table 4 are reported the source of the crudes from which the asphalts were produced, their softening points, and components distributions. The component distributions were determined according to the procedures described in Research Paper 2577, NBS J. Res. Vol. 54, No. 3, March 1955. Asphalt samples 1 to 6, 8 to 11, and 14 were produced by vacuum reduction or vacuum reduction accompanied by air blowing. Samples 7, 12 and 13 were produced by special processes. Component analysis did not distinguish between these two types of asphalt.

As reported in a subsequent section of this report, component analysis can be used to determine changes in asphalts occurring during accelerated durability exposures. The asphaltene content as determined by this component analysis may give some indication of the quality of the asphalt. Asphalts of the same softening points having the lower asphaltene content would normally be preferred. When used in conjunction with the spot test, it could eliminate materials prepared from cracked residuums.

TABLE 4. SOURCE, SOFTENING POINTS AND COMPONENT DISTRIBUTION OF ASPHALTS

Sample No.	Source	Soft. Point (R & B)	Component Distribution			
			Asphaltenes	Water White Oils	Dark Oils	Asphaltic Resins
		°F	%	%	%	%
1	Wyo.	145	30.0	25.0	32.5	10.9
2	Wyo.	147	29.8	22.3	37.4	11.3
3	Wyo.	146	28.5	19.2	40.7	11.3
4	Texas	158	34.4	24.6	28.3	11.3
5	Cal.	144	35.9	22.8	23.8	17.2
6	Mid. Con.	141	24.2	16.9	41.5	16.9
7	Cal.	143	29.7	26.8	29.2	14.3
8	Mid. Con.	149	30.6	26.0	29.5	14.2
9	Mid. Con.	142	27.4	24.8	33.2	14.3
10	Cal.	146	41.5	17.0	25.4	14.6
11	Ven.	148	31.5	21.4	33.7	13.5
12	Cal.	143	29.7	15.3	32.0	22.2
13	----	140	24.8	17.1	41.8	14.3
14	Mid. Con.	148	27.2	25.0	32.0	15.1

4. Susceptibility to Heat

Five grams of the asphalts contained in 250 ml beakers were heated for 10 days at 200°F in an enclosed oven. The source of the crudes from which the asphalts were produced and their component distribution before and after exposure are reported in Table 5. All the asphalts showed marked susceptibility to heat when evaluated by the increases in their asphaltene content.

After exposure at 200°F for ten days, asphalt samples 6, 8, 9 and 14, which had been produced from Mid-Continent crudes, contained less n-pentane insoluble material (asphaltenes) than samples 1, 2, 3, 4, 5, 10 and 11, which had been produced from Wyoming, Texas, Venezuelan and California crudes. These samples would be classified under Type I material of the suggested specification. The two asphalt samples 7 and 12, which were produced from California crudes and which would be classified as Type II materials under the suggested specification, showed essentially the same asphaltene content as samples 5 and 10, Type I materials, which were also produced from California crude oils. The asphaltene content of sample 13, a Type II material, was slightly higher after exposure than that of asphalts produced from the Mid-Continent crude oils. No detectable weight losses occurred during the heat exposure. Weight gains in the order of 0.4 percent occurred during these heat exposures.

These data indicate the changes that will occur in the asphalt moppings between the plies of felts as well as in the top coatings of asphalt built-up roofs, because of their susceptibility to heat.

TABLE 5. DISTRIBUTION OF COMPONENTS IN ASPHALTS BEFORE AND AFTER 10 DAYS EXPOSURE AT 200°F.

Sample No.	Source of Crude	Asphaltenes		Water White Oils		Dark Oils		Asphaltic Resins	
		A	B	A	B	A	B	A	B
		%	%	%	%	%	%	%	%
1	Wyo.	30.0	37.5	25.0	23.3	32.5	25.9	10.9	13.8
2	Wyo.	29.8	41.2	22.3	16.6	37.4	29.7	11.3	14.0
3	Wyo.	28.5	38.7	19.3	15.4	40.7	32.0	11.4	14.0
4	Texas	34.4	40.8	24.6	24.4	28.3	22.2	11.4	12.2
5	Cal.	35.9	46.0	22.8	20.0	23.8	21.2	17.2	13.4
6	Mid. Con.	24.2	33.2	16.9	16.8	41.5	32.4	16.9	17.0
7	Cal.	29.7	45.6	26.8	14.3	29.2	22.2	14.3	18.3
8	Mid. Con.	30.6	36.5	26.0	23.0	29.5	23.4	14.2	15.0
9	Mid. Con.	27.4	34.7	24.8	23.0	33.2	26.5	14.3	14.3
10	Cal.	41.5	45.5	17.0	18.4	25.4	20.0	14.6	13.6
11	Ven.	31.5	41.1	21.4	17.3	33.7	26.2	13.5	15.1
12	Cal.	29.7	42.2	15.3	13.7	32.0	23.8	22.2	21.1
13	----	24.8	37.5	17.1	15.2	41.8	30.2	14.3	18.3
14	Mid. Con.	27.2	34.5	25.0	21.7	32.0	28.0	15.1	15.1

A = Before Exposure. B = After Exposure.

5. Accelerated Durability Exposures

5.1 Exposure 1

To simulate roof conditions where there are no run-offs and standing water evaporates in place.

The specimens were prepared by depositing a uniform film of the melted bitumens over the bottom of 3-1/2- X 1-in. petri dishes. The asphalt specimens were prepared at a spreading rate of 60 lbs/100 ft^2; the coal-tar-pitch specimens at a spreading rate of 70 lbs/100 ft^2.

The coatings were exposed in a horizontal position in a low-intensity, enclosed carbon arc durability machine (see Figure 7). They were exposed for 21 hours to light only. The petri dishes were then filled three-quarters full with distilled water and re-exposed to the radiation of the arc, allowing the water to evaporate in place, which required from 6 to 8 hours. The temperature of the dry specimens during their exposure was from 125°-130°F.

Shrinkage of coatings during exposure: The shrinkage of the coatings from the sides of the dishes after 2500, 3000, 3500, 4000 and 4500 hours of exposure is reported in Table 6. Included in this table are the weight changes which occurred during 4500 hours of exposure.

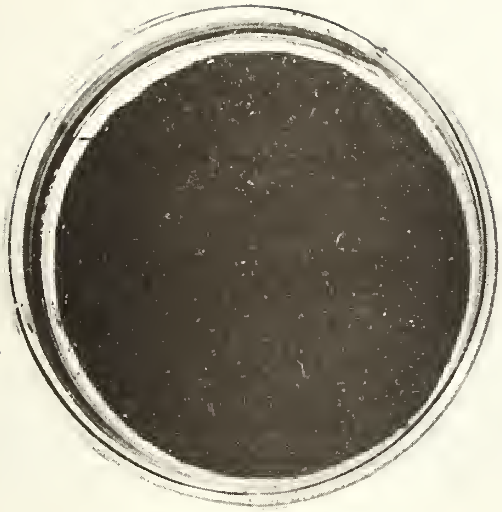
The coal-tar-pitch coatings showed no shrinkage from the sides of the petri dishes after 4500 hours of exposure.

Type I asphalts, samples 6, 8, 9, 11 and 14, showed less shrinkage from the sides of the dishes than samples 1, 2, 3, 4, 7 and 10. The shrinkages of samples 3, 5 and 10 were essentially the same, approximately 0.6 inch, and were the most pronounced of any of the Type I asphalts. Samples 7 and 12, Type II, produced from California crudes, showed much less shrinkage than Type I asphalt, samples 5 and 10, also produced from California crude. Sample 13, Type II, showed less shrinkage than the averages shown by Type I asphalts produced from Mid-Continent crudes. Figure 8 shows the shrinkage which occurred after 4500 hours of exposure in coal-tar-pitch sample 4C and asphalt samples 4, 5 and 11.

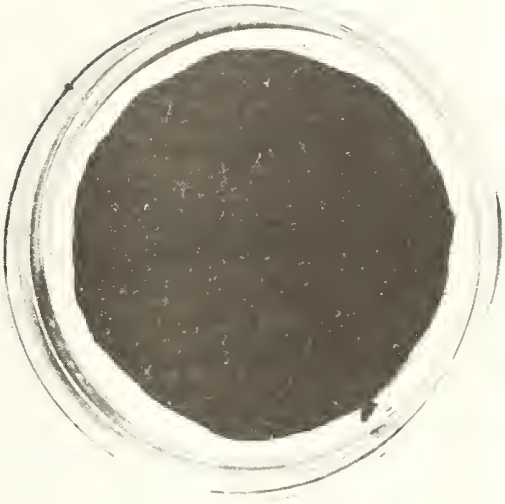
FIGURE 7. ACCELERATED DURABILITY TEST APPARATUS.

TABLE 6. SHRINKAGE FROM SIDES OF DISH OF TEST SPECIMENS AND WEIGHT CHANGES IN ACCELERATED DURABILITY TEST. (EXPOSURE 1).

Sample No.	Source of Crude	Shrinkage from Sides of Dish After Exposure, Hours					Weight Change After 4500 Hrs.
		2500	3000	3500	4000	4500	
		Inch	Inch	Inch	Inch	Inch	Gms/100 Ft.^{2}
1	Wyo.	0.12	0.12	0.12	0.20	0.20	5
2	Wyo.	0.16	0.20	0.20	0.28	0.28	23
3	Wyo.	0.16	0.32	0.32	0.39	0.51	23
4	Texas	0.12	0.12	0.12	0.20	0.24	136
5	Cal.	0.16	0.35	0.47	0.51	0.51	136
6	Mid. Con.	Tr.	Tr.	0.20	0.20	0.20	45
7	Cal.	Tr.	0.12	0.16	0.16	0.20	136
8	Mid. Con.	Tr.	Tr.	0.08	0.08	0.08	18
9	Mid. Con.	Tr.	Tr.	0.04	0.04	0.08	9
10	Cal.	0.16	0.35	0.63	0.63	0.71	---
11	Ven.	Tr.	Tr.	Tr.	Tr.	0.08	9
12	Cal.	Tr.	Tr.	0.08	0.08	0.28	5
13	----	----	None	----	0.08	0.08	Tr.
14	Mid. Con.	----	0.04	----	0.04	0.08	131
2C	----	None	None	None	None	None	254
3C	----	"	"	"	"	"	50
4C	----	"	"	"	"	"	91



11.



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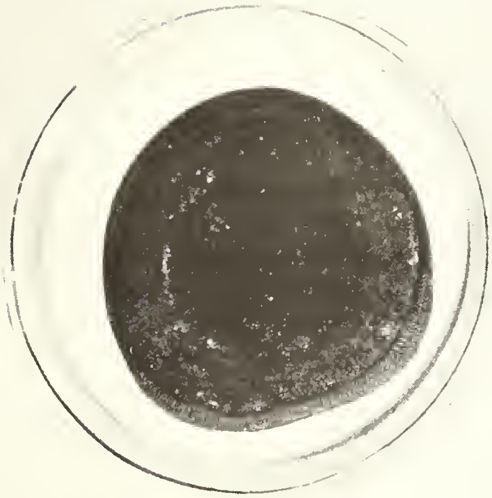

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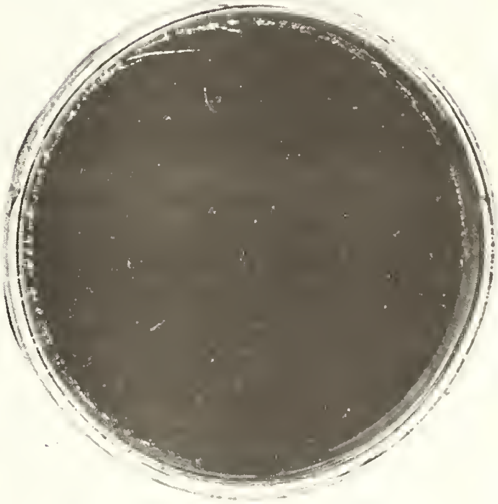

4C.

FIGURE 8. EXTREMES IN THE SHRINKAGE OF ASPHALT AND COAL-TAR-PITCH SPECIMENS AFTER 4500 HOURS OF EXPOSURE 1.

Water-soluble, photochemical, degradation products: After 1000, 3000, 3500, 4000 and 4500 hours of exposure to light, the petri dishes were filled with distilled water, allowed to stand for 18 hours, and the extracted material titrated with 0.01N alkali solution. These data are reported in Table 7. This titratable material was a photochemical degradation product. The relative amounts were markedly greater for the asphalts than for the coal-tar pitches. See Exposure 3 for the amounts of titratable materials formed when they are removed after each 21-hour exposure to light.

Changes in physical characteristics: In Table 8 are reported the softening points and penetrations of the asphalts before and after exposure. All asphalts showed marked hardening and embrittlement, as evidenced by the increases in the softening points and decreases in penetrations.

Changes in distribution of components: In Table 9 are reported the component distribution of the asphalts before and after 4500-hours exposure (Exposure 1). As in the exposures of the asphalts to heat alone, there were marked increases in the n-pentane insoluble components. Although these exposures were made at temperatures of 125°-130°F, which were appreciably lower than maximum roof temperatures, heat degradation still occurred.

The percentage of n-pentane insoluble material in samples 6, 8, 9 and 14 after exposure was appreciably lower than that found in samples 1, 2, 3, 4, 5, 10 and 11. All of these samples would be classified as Type I materials under the suggested specification. Samples 7 and 12, Type II, produced from California crude oils, contained less n-pentane insoluble material than samples 5 and 10, Type I, also produced from California crudes. The n-pentane insoluble content of sample 13, Type II, after exposure, was slightly less than the average of that formed in samples 6, 8, 9 and 14, produced from Mid-Continent crudes. These increases in n-pentane insoluble material, which were formed at the expense of the water-white oils and dark oils, resulted in a hardening of the asphalts as discussed under "Change in physical characteristics". Asphalts containing the lesser amounts of n-pentane insoluble material after exposure would be preferred. For example, if a choice had

TABLE 7. GRAMS SODIUM HYDROXIDE REQUIRED TO NEUTRALIZE WATER-SOLUBLE MATERIAL FORMED IN ACCELERATED DURABILITY TEST (EXPOSURE 1).

Sample No.	Source of Crude	Grams Sodium Hydroxide To Neutralize Water-Soluble Materials Formed Exposure, Hours					
		1000	3000	3500	4000	4500	Total
		G/100 ft^2	G/100 ft^2	G/100 ft^2	G/100 ft^2	G/100 ft^2	G/100 ft^2
1	Wyo.	7.3	7.8	7.8	8.5	10.5	41.9
2	Wyo.	8.0	6.7	6.2	6.5	7.0	34.4
3	Wyo.	6.0	8.9	6.0	6.3	7.0	34.2
4	Texas	8.0	10.2	10.2	10.8	11.6	50.8
5	Cal.	9.3	10.0	9.8	10.0	12.5	51.6
6	Mid. Con.	5.3	4.7	4.4	4.9	5.0	24.3
7	Cal.	8.0	7.2	6.8	6.5	6.0	34.5
8	Mid. Con.	5.3	6.1	6.5	6.5	7.3	31.7
9	Mid. Con.	5.3	6.9	7.3	7.5	8.0	35.0
10	Cal.	8.0	9.4	4.4	4.2	4.1	30.1
11	Ven.	3.3	5.5	7.5	7.8	9.8	33.9
12	Cal.	7.3	6.4	4.9	5.5	6.9	31.0
2C	----	Trace	0.6	0.3	0.3	0.2	1.4
3C	----	Trace	0.2	0.3	0.3	0.4	1.2
4C	----	Trace	0.4	0.2	0.4	0.3	1.3

TABLE 8. SOFTENING POINTS OF ASPHALTS AND COAL-TAR PITCHES AND PENETRATIONS OF ASPHALTS BEFORE AND AFTER 4500 HOURS EXPOSURE TO ACCELERATED DURABILITY TEST (EXPOSURE 1).

Sample No.	Source of Crude	Soft. Pt. (R & B)		Penetrations					
				77°F, 100 g, 5 sec.		32°F, 200 g, 60 sec.		115°F, 50 g, 5 sec.	
		A	B	A	B	A	B	A	B
		°F	°F	1/10 mm.	1/10 mm.	1/10 mm.	1/10 mm.	1/10 mm.	1/10 mm.
1	Wyo.	145	183	42	22	22	13	117	42
2	Wyo.	148	171	26	15	9	8	90	36
3	Wyo.	146	174	29	16	11	9	108	40
4	Texas	158	195	30	15	17	11	73	27
5	Cal.	144	192	42	15	19	10	132	32
6	Mid. Con.	143	171	31	17	13	10	113	35
7	Cal.	143	170	16	7	5	2	95	25
8	Mid. Con.	149	178	31	17	12	10	89	35
9	Mid. Con.	142	185	46	22	22	14	122	38
10	Cal.	146	183	36	13	18	7	130	34
11	Ven.	148	183	28	13	15	8	98	30
12	Cal.	143	172	16	6	5	1	99	20
13	----	140	167	20	11	7	3	120	34
14	Mid. Con.	148	181	41	25	21	14	105	44
2C	----	131	160						
3C	----	132	146						
4C	----	138	149						

A = Before Exposure. B = After Exposure.

TABLE 9. DISTRIBUTION OF COMPONENTS IN ASPHALTS BEFORE AND AFTER 4500 HOURS OF EXPOSURE TO ACCELERATED DURABILITY TEST (EXPOSURE 1).

Sample No.	Source of Crude	N-Pentane Insoluble		Water White Oils		Dark Oils		Asphaltic Resins	
		A	B	A	B	A	B	A	B
		%	%	%	%	%	%	%	%
1	Wyo.	30.0	42.3	25.0	20.7	32.5	21.6	10.9	14.3
2	Wyo.	29.8	42.0	22.3	16.1	37.4	25.0	11.3	16.3
3	Wyo.	28.5	43.0	19.3	15.6	40.7	26.1	11.3	15.0
4	Texas	34.4	44.5	24.6	22.2	28.3	19.0	11.4	15.0
5	Cal.	35.9	52.5	22.8	19.8	23.8	15.5	17.2	13.4
6	Mid. Con.	24.2	34.0	16.9	16.0	41.5	29.1	16.9	20.3
7	Cal.	29.7	45.0	26.8	14.1	29.2	20.3	14.3	20.9
8	Mid. Con.	30.6	29.2	26.0	22.3	29.5	21.1	14.2	16.7
9	Mid. Con.	27.4	38.6	24.8	20.8	33.2	19.9	14.3	16.8
10	Cal.	41.5	54.4	17.0	17.6	25.4	15.9	14.6	12.3
11	Ven.	31.5	42.7	21.4	18.4	33.7	22.4	13.5	16.3
12	Cal.	29.7	43.8	15.3	14.5	32.0	20.1	22.2	21.9
13	----	24.8	34.4	17.1	15.0	41.8	29.5	14.3	20.8
14	Mid. Con.	27.2	34.8	25.0	21.5	32.0	24.5	15.1	18.9

A = Before Exposure. B = After Exposure.

to be made among samples 5, 7, 10 and 12, all produced from California crude oils, samples 7 and 12, Type II materials, would be preferred. By the same reasoning, sample 13 would be preferred to sample 11.

5.2 Exposure 2

Exposures made to simulate conditions to which a cracked membrane may be subjected.

The specimens were prepared and exposed as described under Exposure 1. At fixed intervals, a groove 1/16 in. wide was cut through the coatings along a diameter. The specimens were then exposed dry to the radiation of the arc until the groove had healed. The temperature of the specimens during their dry exposures was 125°-135°F. In Table 10 are reported the extent of self-healing in specimens which had been exposed for 4500 hours before being grooved. The self-healing in the coal-tar-pitch coatings was much more rapid than in the asphalt specimens. Asphalt samples 2, 3, 5, 7, 8, 9, 10, 11, 13 and 14 showed satisfactory self-healing within 96 hours of exposure. The percentages reported represent the approximate closure of the grooves at the times indicated. Asphalt samples 1, 4, 6 and 12 were not included because of lack of space in the test equipment.

5.3 Exposure 3

Exposures made to simulate roof conditions where water-soluble, light-degraded products were partially removed as by dew or rain.

Specimens were prepared for this exposure as for Exposure 1, except that the coatings were applied in petri dishes at a spreading rate of 18 lb/100 ft^2, to accelerate degradation.

The dry specimens were exposed horizontally to radiation from the carbon arc (Figure 7). After 21 hours of exposure the petri dishes were filled with water and allowed to stand, without light, for one hour, and the water poured off. This process was repeated until the coatings had been exposed to light for 900 hours. The

TABLE 10. SELF-HEALING OF SPECIMENS EXPOSED 4500 HOURS. (EXPOSURE 2).

Sample No.	Source of Crude	Self-Healing After 4500 Hours of Exposure			
		24 Hrs.1/	48 Hrs.1/	72 Hrs.1/	96 Hrs.1/
		%	%	%	%
2	Wyo.	50	90	100	---
3	Wyo.	90	100	---	---
5	Cal.	50	60	60	90
7	Cal.	90	100	---	---
8	Mid. Con.	90	100	---	---
9	Mid. Con.	50	60	70	100
10	Cal.	50	70	90	100
11	Ven.	90	100	---	---
13	----	100	---	---	---
14	Mid. Con.	50	80	100	---
2C		100 2/			
3C		100 2/			
4C		100 2/			

1/ Hours of additional exposure.

2/ Healed completely within 3 hours.

coatings were dried and weighed after 200, 400, 600 and 900 hours of exposure, and the accumulated washings to these periods titrated with 0.01N alkali. The coatings were also examined for shrinkage. In Table 11 are reported the shrinkage, weight loss and amount of alkali required to neutralize the water-soluble degradation products after 900 hours of exposure. The latter is reported as grams of sodium hydroxide required to neutralize products formed on 100 sq. ft. of exposed surface.

Marked differences were noted between the results of Exposure 1, where light-degraded products were partially dissolved and redeposited on the coatings, and of Exposure 3, where these products were partially removed daily. In Exposure 1, the alkali required to neutralize the light-degraded material formed from the asphalts during 1000 hours of exposure varied from 3 to 9 grams per 100 ft^2; in Exposure 3, 18 to 66 grams were required after 900 hours of exposure. A number of the asphalts showed slight weight gains after 4500 hours under Exposure 1; all showed progressively greater weight losses at the 200, 400, 600 and 900 hour weighing periods of Exposure 3. The relationship of the weight losses in Exposure 3 to the amounts of alkali required to neutralize the water-soluble degradation products indicates strongly that the weight losses were due largely to the removal of water-soluble products.

In another investigation here it has been shown that asphalts exposed to radiation from the carbon arc and washed daily continued to lose weight during an exposure of 4500 hours duration. Similarly, specimens exposed outdoors and weighed at intervals of 6 months, continued to lose weight during a 3-year exposure. Since the formation of water-soluble degradation products is a light-induced reaction, the value of opaque mineral surfacing for any asphalt roof is obvious.

Practically no shrinkage occurred in any of the asphalts during Exposure 3. This is in marked contrast to their behavior in Exposure 1, indicating that the redeposited, water-soluble, degradation products caused the shrinkage. In Exposure 3 the asphalts weathered to a dull, matt surface; in Exposure 1, the surfaces were dull and wrinkled.

As in Exposure 1, the coal-tar pitches in Exposure 3 showed markedly less light-degraded materials formed than the asphalts. The daily removal of the light-degraded materials from the pitches did not increase appreciably the amounts of these materials that were formed. Weight losses of the coal-tar-pitch specimens were confined to the first 200 hours of Exposure 3.

Changes in the component distribution of the asphalts during 900 hours of Exposure 3 are shown in Table 12. These changes paralleled those in Exposure 1, with asphalts 5 and 10, produced from California crudes, showing the largest amounts of matter insoluble in n-pentane at the end of the test. Again, Type I asphalts 6, 8, 9 and 14, produced from Mid-Continent crudes, contained less matter insoluble in n-pentane than the other Type I asphalts. Of all of the asphalts, No. 13, Type II, contained the smallest amount of matter insoluble in n-pentane. The other Type II asphalts, 7 and 12, produced from California crudes, contained less than Type I asphalts 5 and 10, also produced from California crudes.

6. Outdoor Exposures

Changes in Physical Characteristics: In Table 13 are reported the softening points and penetrations of the asphalts before and after 10 months of exposure outdoors.

For the outdoor exposure the specimens were prepared as for Exposure 1. They were exposed in a horizontal position on the roof of the Industrial Building, National Bureau of Standards, during May 1956. The length of the exposure was 10 months.

At the end of the 10-month, outdoor exposure all asphalts showed a marked increase in softening point and a corresponding decrease in penetration values. The increases in softening point ranged from 13° to 21°F and were apparently not related to the initial softening points of the asphalts or to the source of the crudes from which the asphalts were produced. These data indicate that asphalts having softening points near the minimum specification requirement would be preferred to those whose softening points were near the maximum requirement. The coal-tar-pitch specimens also showed an increase in softening point after the 10-month exposure. The maximum increase was 7°F.

TABLE 11. SHRINKAGE, WEIGHT LOSS, AND MATERIALS TITRATABLE WITH ALKALI FORMED AFTER 900 HOURS EXPOSURE TO THE ACCELERATED DURABILITY TEST (EXPOSURE 3).

Sample No.	Source of Crude	Shrinkage From Sides of Dishes	Weight Loss	Sodium Hydroxide Required to Neutralize Water-Soluble Degradation Products
		Inch	Grams/100 ft^2	Grams/100 ft^2
1	Wyo.	None	101	43
2	Wyo.	"	93	36
3	Wyo.	"	93	32
4	Texas	"	162	52
5	Cal.	0.1	195	66
6	Mid. Con.	None	53	30
7	Cal.	"	63	29
8	Mid. Con.	"	137	39
9	Mid. Con.	"	75	32
10	Cal.	"	240	61
11	Ven.	"	340	75
12	Cal.	"	75	34
13	----	0.2	261	46
14	Mid. Con.	None	34	18
2C	----	None	0.210	1.3
3C	----	"	0.093	1.3
4C	----	"	0.180	1.5

TABLE 12. COMPONENT DISTRIBUTION OF ASPHALTS BEFORE AND AFTER 900 HOURS OF EXPOSURE TO ACCELERATED DURABILITY TEST (EXPOSURE 3).

Sample No.	Source of Crude	N-Pentane Insoluble Material		Water White Oils		Dark Oils		Asphaltic Resins	
		A	B	A	B	A	B	A	B
		%	%	%	%	%	%	%	%
1	Wyo.	30.0	37.2	25.0	21.7	32.5	23.9	10.9	15.6
2	Wyo.	29.8	35.0	22.3	16.8	37.4	28.3	11.3	17.0
3	Wyo.	28.5	35.5	19.3	16.0	40.7	30.3	11.3	16.6
4	Texas	34.4	41.9	24.6	20.2	28.3	19.6	11.4	16.8
5	Cal.	35.9	45.7	22.8	18.9	23.8	16.3	17.2	16.0
6	Mid. Con.	24.2	29.6	16.9	16.1	41.5	27.0	16.9	22.9
7	Cal.	29.7	39.0	26.8	14.5	29.2	20.3	14.3	22.8
8	Mid. Con.	30.6	35.5	26.0	22.2	29.5	21.9	14.2	20.0
9	Mid. Con.	27.4	33.8	24.8	23.9	33.2	20.7	14.3	20.9
10	Cal.	41.5	46.8	17.0	15.3	25.4	19.6	14.6	14.5
11	Ven.	31.5	40.8	21.4	17.5	33.7	19.1	13.5	16.1
12	Cal.	29.7	38.3	15.3	13.2	32.0	22.4	22.2	20.0
13	----	24.8	34.1	17.1	15.7	41.8	28.9	14.3	20.5
14	Mid. Con.	27.2	32.2	25.0	19.4	32.0	22.6	15.1	21.1

A = Before Exposure. B = After Exposure.

TABLE 13. SOFTENING POINTS OF ASPHALTS AND COAL-TAR PITCHES AND PENETRATIONS OF ASPHALTS BEFORE AND AFTER 10 MONTHS EXPOSURE OUTDOORS (SPECIMENS EXPOSED MAY 1956)

Sample No.	Source of Crude	Soft. Pt. (R & B)		Penetrations					
				77°F, 100 g, 5 sec.		32°F, 200 g, 60 sec.		115°F, 50 g, 5 sec.	
		A[1/]	B[2/]	A	B	A	B	A	B
		°F	°F	1/10 mm.	1/10 mm.	1/10 mm.	1/10 mm.	1/10 mm.	1/10 mm.
1	Wyo.	145	162	42	29	22	19	117	68
2	Wyo.	148	161	26	19	9	11	90	54
3	Wyo.	146	160	29	20	11	15	108	60
4	Texas	158	174	30	21	17	19	73	47
5	Cal.	144	164	42	25	19	12	132	67
6	Mid. Con.	141	154	31	24	13	12	113	68
7	Cal.	143	157	16	12	5	2	95	52
8	Mid. Con.	149	163	31	23	12	10	89	58
9	Mid. Con.	142	163	46	30	22	16	122	73
10	Cal.	146	160	36	24	18	14	130	77
11	Ven.	148	163	28	19	15	12	98	56
12	Cal.	143	157	16	11	5	1	99	52
13	----	140	154	20	14	7	6	120	56
14	Mid. Con.	148	164	41	31	21	21	105	69
2C		131	138						
3C		132	138						
4C		138	145						

1/ A = Before Exposure.

2/ B = After Exposure.

<u>Shrinkage</u>: Since these exposures were of such short duration, only traces of shrinkage of the asphalt coatings from the sides of the petri dishes occurred. No shrinkage was detected in the coal-tar-pitch specimens.

<u>Self-healing characteristics</u>: To determine self-healing ability the specimens were grooved, as for Exposure 2, before exposure on May 22, 1956. Both asphalt and coal-tar specimens healed within 24 hours. Specimens were grooved again on July 12, 1956, and again all specimens healed within 24 hours. Specimens were grooved again November 8, 1956, and when inspected on December 31, 1956, no self-healing was detected in either the asphalt or coal-tar pitch specimens. Inspection on February 27, 1957, showed evidence of incomplete self-healing in sample 7.

On March 18, 1957, when the exposure was terminated, the asphalt specimens were essentially the same as in the previous inspection. The coal-tar-pitch specimens showed some self-healing. These data indicate that little or no self-healing can be expected from either asphalt or coal-tar pitch during cold weather.

7. Water Absorption Tests

The water absorption tests were made on the asphalts and coal-tar pitches to determine the amount of water absorbed during long-time immersion in distilled water. A further objective was to determine the amount of alkali required to neutralize material leached from the specimens during immersion.

Approximately 25 grams of asphalt and 30 grams of coal-tar pitch were melted, poured into molds, forming disks with a surface area of approximately 0.11 ft^2. Each disk was weighed, placed in a separate petri dish, and covered with distilled water. The disks were removed from the water at intervals, blotted dry, and reweighed. Any gain in weight was taken as the quantity of water absorbed during the period. The total amount of water absorbed per 100 ft^2 of exposed surface in one year is shown in Table 14.

TABLE 14. RESULTS OF WATER-ABSORPTION TESTS.

Sample No.	Source of Crude	H_2O Absorbed	Sodium Hydroxide Required to Neutralize Extracted Materials	Appearance
		grams/100 ft^2/year	grams/100 ft^2/year	
1	Wyo.	223	.11	Glossy Black
2	Wyo.	236	.01	" "
3	Wyo.	263	.03	" "
4	Texas	178	.02	" "
5	Cal.	193	.04	" "
6	Mid. Con.	375	.03	" "
7	Cal.	164	.04	" "
8	Mid. Con.	115	.01	" "
9	Mid. Con.	231	.03	" "
10	Cal.	340	.03	" "
11	Ven.	195	.03	" "
12	Cal.	167	.01	" "
13	----	191	.04	" "
14	Mid. Con.	223	.13	" "
2C		125	.04	" "
3C		141	.02	" "
4C		180	.02	" "

After immersion for one year, the water in which each specimen was immersed was titrated with 0.01N sodium hydroxide solution. The results are reported in Table 14 as grams of sodium hydroxide required to neutralize material leached per 100 ft^2 during one year of immersion.

All specimens were glossy black after one year of immersion.

APPENDIX 3. DISCUSSION OF REQUIREMENTS IN THE PROPOSED SPECIFICATION FOR ASPHALTS FOR LOW-SLOPE ROOFS

1. Conventional Requirements

Softening point: All of the asphalts submitted in this program had softening points within the range of 140°F to 165°F specified in Federal Specification SS-A-666, Type I, Class A, for surfaced built-up roofs, and all but sample 4 (158°F) within the range of 135°F to 150°F specified by A.S.T.M. Specification D312, "Mineral-Surfaced Flat". We believe that a softening point range of 135°F to 145°F will insure, in large measure, asphalts of desirable qualities for low-slope roofs.

Ductility at 77°F: Federal Specification SS-A-666, Type I, Class A, requires a minimum ductility at 77°F of 5 cm. and A.S.T.M. Specification D312, "Mineral-Surfaced Flat", a minimum ductility of 10 cm. While five of the Type I asphalts had a ductility lower than 15 cm. at 77°F, as suggested in the proposed specification, four of these showed softening points higher than the maximum specified. We believe that these asphalts would have met the higher ductility requirement if they had been processed to meet the softening point requirement in the proposed specification.

The high ductility at 77°F of asphalts 7, 12 and 13 indicates that these asphalts have been processed to simulate some of the physical characteristics of coal-tar pitch used in low-slope roofs. The relatively high ductility of asphalts of this type makes mandatory the inclusion of two types of asphalt in the specification.

Penetration tests:

At 32°F: The Federal and A.S.T.M. Specification requirements of 10 and 5, respectively, can be raised safely to 15 for Type I asphalts. This higher value is more likely to insure desirable self-healing qualities. Examination of the three Type II asphalts indicated a minimum penetration requirement at 32°F of 5.

At 77°F: The penetration limits of 25 to 60 suggested are in line with the softening point and the penetration values of the Type I asphalts tested. These limits also provide a means for differentiating between Type I and Type II asphalts. Limits of 15 to 25 are indicated for Type II asphalts from the samples tested.

At 115°F: Federal Specification SS-A-666, Type I, contains no requirement for penetration at 115°F. Since a self-healing material is desired, a minimum requirement of 100, for Type I asphalt, as specified in A.S.T.M. Specification D312, "Mineral-Surfaced Flat", is considered desirable. Tests indicate a minimum of 90 for Type II asphalts.

Weight loss, 5 hrs. at 32°F: Tests of numerous asphalts have indicated that the maximum requirement of 1.5% in the Federal and A.S.T.M. Specifications for this test is entirely unrealistic. The loss on heating of each of the Type I and Type II samples tested in this series was less than 0.1%. Consequently, the proposed maximum was placed at 0.5%.

Penetration at 77°F after heating: Here again the requirement of 60% of the penetration at 77°F before heating, in both Federal and A.S.T.M. Specifications, is known to be unrealistic. Minimums of 85% and 75% for Types I and II asphalts, respectively, are more realistic.

Bitumen soluble in CCl_4: The requirement of 99% bitumen soluble in carbon tetrachloride is standard for unfilled petroleum asphalts.

2. New Requirements

Matter insoluble in n-pentane: The percentage of matter insoluble in n-pentane is known to increase as the blowing operation is continued; consequently, a minimum requirement for the amount of material insoluble in n-pentane could control the extent of blowing. Large percentages of matter insoluble in pentane are characteristic of "cracked residuums", generally regarded as having poor resistance to weathering.

The two asphalts in the series tested that initially contained the largest percentage of matter insoluble in n-pentane also gave positive spot tests, showed the greatest shrinkage under Exposure 1, and were included in the three that required the most alkali to neutralize water-soluble degradation products formed in Exposure 3. It is significant also that the percentage of matter insoluble in n-pentane increased in all asphalts during the accelerated and outdoor weathering tests and the heat tests.

Spot test: The spot test has been used largely in specifications for road asphalts to eliminate cracked residuums. It is believed that it can serve the same purpose for roofing asphalts. Note the reference to the two asphalts that gave positive spot tests, under "Matter insoluble in n-pentane".

Susceptibility factor: The susceptibility factor requires no extra laboratory work since it is calculated from the penetration determinations. Inclusion of minimum requirements for a susceptibility factor serves to distinguish between Type I and Type II asphalts and places numerical values on the "relatively susceptible" and "self-healing" properties described in A.S.T.M. specifications.

Flow test: The flow test, adapted from Federal Specification SS-S-164, "Sealer; Hot-Poured Type, for Joints in Concrete", was included in an attempt to insure asphalts with good self-healing qualities.

Ductility at 40°F, 0.25 cm/min.: The principal purpose served by this requirement is to distinguish between Type I and Type II asphalts. All samples of Type II asphalt and all of the samples of coal-tar pitch tested fractured rather than stretched in this test; all samples of Type I asphalt met the requirement for the test.

U. S. DEPARTMENT OF COMMERCE
Sinclair Weeks, *Secretary*

NATIONAL BUREAU OF STANDARDS
A. V. Astin, *Director*

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