MICHIGAN
STATE HIGHWAY DEPARTMENT
G. Donald Kennedy
State Highway Commissioner

PROPOSED SPECIFICATIONS FOR A SLOW CURING LIQUID ASPHALT

By

J.W. Kushing
E.A. Finney

Research Project 36 C-1

Research Laboratory
Testing and Research Division
Report No. 17
June 15, 1941

CONTENTS

Page

Preface. 1

Introduction. 2

Changes in Characteristics of Slow Curing Asphalts When Subjected to Different Laboratory Treatments. 5

Changes in Characteristics of Slow Curing Asphalts After Weathering Under Service Conditions. 10

Condition Survey of Existent Bituminous Surfaces Using SC-6A Asphaltic Materials as a Binder. 13

Summary of Data Relevant to Proposed Changes in Specification for Slow-Curing Liquid Asphaltic Material. 17

1. Flash Point Temperature. 19
2. Loss on Heating Percentage. 20
3. Viscosity of Residue from Loss on Heating Test. 20
4. Viscosity Range of Original Material. 22
5. Percentage of 100 Penetration Residue. 23
6. Ash Content. 24
7. Emulsion Test. 24
8. Water and Sediment. 24

Summary. 25

Table of Proposed Specification Requirements. 26

PREFACE

The Research and Testing Divisions of the Michigan State Highway Department have collaborated in preparing a new specification for a slow-curing liquid asphalt to be used in oil aggregate construction. A separate report has been prepared by each Division covering pertinent research activities and including their proposed specification. This report constitutes the work and recommendations of the Research Division.

PROPOSED SPECIFICATION
FOR
SLOW-CURING LIQUID ASPHALTIC MATERIALS

Introduction

From the standpoint of Michigan practice the oil aggregate road surface was developed with the idea that, during a certain period of years after construction, it should be possible to correct any irregularities in the road surface by scarifying and remaking the existent material, perhaps with the addition of slight quantities of bituminous material. Therefore, any abnormal hardening of the bituminous mixture either in the road surface or in stock piles established for maintenance purposes, is objectionable.

It is evident that an oil aggregate surface may change gradually from a flexible to a semi-rigid or rigid state over a period of years. The rate of transformation is dependent upon the inherent properties and constituents of the oil aggregate mixture.

Thus, in establishing criteria for judging bituminous materials on the basis of their service behavior, it is evident that the following factors must be taken into consideration, namely, the amount of oily constituents and their rate of dissipation, the cohesive and adhesive properties of the bitumen when aged, the petrogenic characteristics of the mineral aggregates and the inherent properties of the oil aggregate mixture.

In regard to the factors set forth above the last two pertaining to the petrogenic characteristics of the aggregate and the inherent properties of the bituminous mixture can be controlled to a certain extent by construction practice. The characteristics and service behavior of the bituminous material can be controlled only in so far as specifications are applicable and by the willingness of the manufacturer to furnish satisfactory material.

It is necessary from time to time to revise specification limitations in order to obtain more desirable bituminous products. Consequently, with the thought of improving a specification for a slow curing liquid asphalt, an extensive long range field and laboratory research program was initiated in 1936, which included a study of slow curing asphaltic oils and oil aggregate bituminous mixtures. The study on slow curing asphaltic oils has been confined to the viscosity-temperature relationship and to the change in viscosity of bituminous materials before and after weathering. Also, a study was made of the change in consistency of recovered asphaltic oils after incorporation in bituminous mixtures at different ages. This work has been augmented by field studies relative to the service behavior of existing bituminous surfaces constructed with asphaltic materials of the slow curing type.

It is the intent of this report to summarize briefly the scope and results of the bituminous researches on slow curing asphalts to date for the purpose of setting up new specification requirements as a means of obtaining a more uniform product and a product which will have a slow rate of hardening in the road surface.

The report includes such studies as; the changes in characteristics of slow curing asphalts when subjected to different laboratory treatments; changes in characteristics of slow curing asphalts after weathering under service conditions; a condition survey of existent bituminous surfaces using SC-6A bituminous materials and a summary of data relevant to proposed changes in specification for slow curing liquid asphalts.

CHANGES IN CHARACTERISTICS OF SLOW CURING ASPHALTS WHEN SUBJECTED TO DIFFERENT LABORATORY TREATMENTS

The purpose of the laboratory study was to determine if the changes which occur in the characteristics of slow-curing asphaltic materials when subjected to laboratory treatments could be correlated with the changes occuring when the materials are incorporated into bituminous road surfaces.

Sixteen different asphaltic materials of the SC-6 type meeting Michigan State Highway Specifications were subjected to the four following treatments; an oven volatilization test, the A.S.T.M. distillation test; blowing with air, and ultra-violet radiations by a modified weatherometer test. After these treatments the viscosity of the residue was determined at 25°C and 60°C, and the susceptibility computed. The data obtained from these tests are tabulated in table I.

Oven volatilization test: The oven volatilization test was conducted in the following manner; the oil was weighed out in tared tins and then placed into an ordinary laboratory drying oven at a constant temperature for different periods of time; i.e., 50 grs - 163°C. The tins, after removal from the oven were cooled and weighed and the per cent loss was computed.

Four materials, B, D, G, J, of varying degrees of volatility as shown by the A.S.T.M. distillation tests to be 7%, 5%, 12.0% and 1.5% respectively, were chosen to study the effect of time on the loss of volatile matter and consequent change in viscosity of the residue.

The data shown in table 2 and illustrated in figures 1 and 2 shows that there is a marked difference between the rates of hardening of the four materials and especially for the two materials D and G.

In addition to the above oven volatilization test on materials B, D, G, and J, a five hour oven test at 163°C - 50 gram was performed on the balance of the sixteen materials as a matter of comparison between percentage loss of volatile matter and increase in viscosity to determine to what degree the change in consistency of the residues would be comparable to gain in viscosity of a material in service. The data is presented in table 1.

Distillation Test: The sixteen materials were also subjected to the A.S.T.M. Distillation test. The viscosity and susceptibility of the residue was determined upon completion of the test. See table 1.

Air Blowing: The third treatment involved blowing air through a 75 gram sample of each asphaltic material at 180°C for two hours at a rate of 500 cubic centimeters per minute. Air was introduced into the sample through a copper tube similar to that used in the Abson

TABLE NO. 1

VISCOSITY AND SUSCEPTIBILITY OF SLOW-CURING ASPHALTS
AFTER SUBJECTION TO LABORATORY TREATMENTS

Viscosity in Centistokes

	Original Asphaltic Oil			Oven Volatilization Residue		Distillation Residue		Air Blown Residue		Residue Modified Weatherometer	
Oil	Viscosity 25°C	Viscosity 60°C	Susc.	Viscosity 60°C	Susc.	Viscosity 60°C	Susc.	Viscosity 60°C	Susc.	Viscosity 60°C	Susc.
A	120,480	1,003	4.741	3,671	4.898	5,810	4.919	6,884	4.719	22,847	4.987
B	25,720	1,032	3.429	2,316	3.656	4,195	3.664	5,945	3.798	51,274	3.915
C	24,800	909	3.562	2,104	3.640	3,016	3.682	8,335	3.657	30,208	3.362
D	26,290	954	3.551	1,001	3.661	1,060	3.520	1,628	3.584	10,841	3.815
E	41,810	1,179	3.679	2,390	3.956	2,130	3.768	4,535	4.014	28,650	4.451
F	40,280	1,403	3.434	3,392	3.415	4,335	3.497	8,183	3.652	18,851	3.730
G	28,130	1,174	3.343	2,821	3.406	11,440	3.440	22,104	3.476	5,207,400	3.357
H	63,920	976	4.275	3,301	4.329	3,116	4.381	13,344	3.494	314,885	4.234
I	35,390	1,115	3.629	2,821	3.662	2,342	3.789	5,080	3.585	58,059	3.833
J	63,360	1,161	4.043	2,316	4.295	2,209	4.439	2,943	3.901	96,876	4.041
K	25,760	949	3.540	2,182	3.721	2,848	3.843	6,750	3.970	12,250	4.086
L	46,410	1,005	3.952	3,685	4.044	2,590	4.048	12,556	4.030	77,224	3.242
M	51,130	1,386	3.644	2,292	3.583	1,695	3.636	5,475	3.842	50,966	4.252
N	67,070	1,398	3.855	3,298	3.936	3,958	3.772	17,923	3.889	165,620	4.415
O	78,840	900	4.551	1,043	4.289	1,287	4.799	3,652	3.687	10,682	5.691
P	55,110	1,152	3.939	1,403	3.944	1,551	3.917	2,127	4.130	10,276	3.939

TABLE NO. 2

VISCOSITY DETERMINATIONS ON RESIDUES FROM

OVEN VOLATILIZATION TEST

50 grams - at 163°C - Time as given - Viscosity in centistokes

Hrs. in Oven	% % Loss	B Viscosity	D % Loss	D Viscosity	G % Loss	G Viscosity	J % Loss	J Viscosity
Temperature at 25°C								
0	0	25,723	0	26,294	0	28,126	0	63,364
1	2.053	44,420	0.110	28,791	1.836	44,633	0.761	122,462
2	2.348	52,270	0.305	28,936	2.893	66,048	1.392	129,837
3	3.234	63,533	0.395	29,215	4.110	81,343	1.621	136,576
4	4.123	75,526	0.401	30,160	4.185	85,195	1.890	169,944
5	5.074	112,095	0.437	32,022	5.078	108,725	2.308	257,555
25	10.181	791,628	2.520	62,487	13.673	1,940,705	6.082	1,307,108
50	15.500		3.220	118,870	16.805		10.421	
Temperature at 60°C								
0	0	1.031	0	954	0	1,174	0	1,161
1	2.053	1,271	0.110	898	1.836	1,507	0.761	1,268
2	2.348	1,590	0.305	977	2.893	2,020	1.392	1,444
3	3.234	1,646	0.395	1,018	4.110	2,269	1.621	1,573
4	4.123	1,898	0.401	1,034	4.185	2,525	1,890	1,615
5	5.074	2,316	0.437	1,087	5.078	2,821	2.308	1,849
25	10.181	7,546	2.520	1,544	13.673	34,632	6.082	5,159
50	15.500	40,911	3.220	2,613	16.805	277,300	10,421	37,596

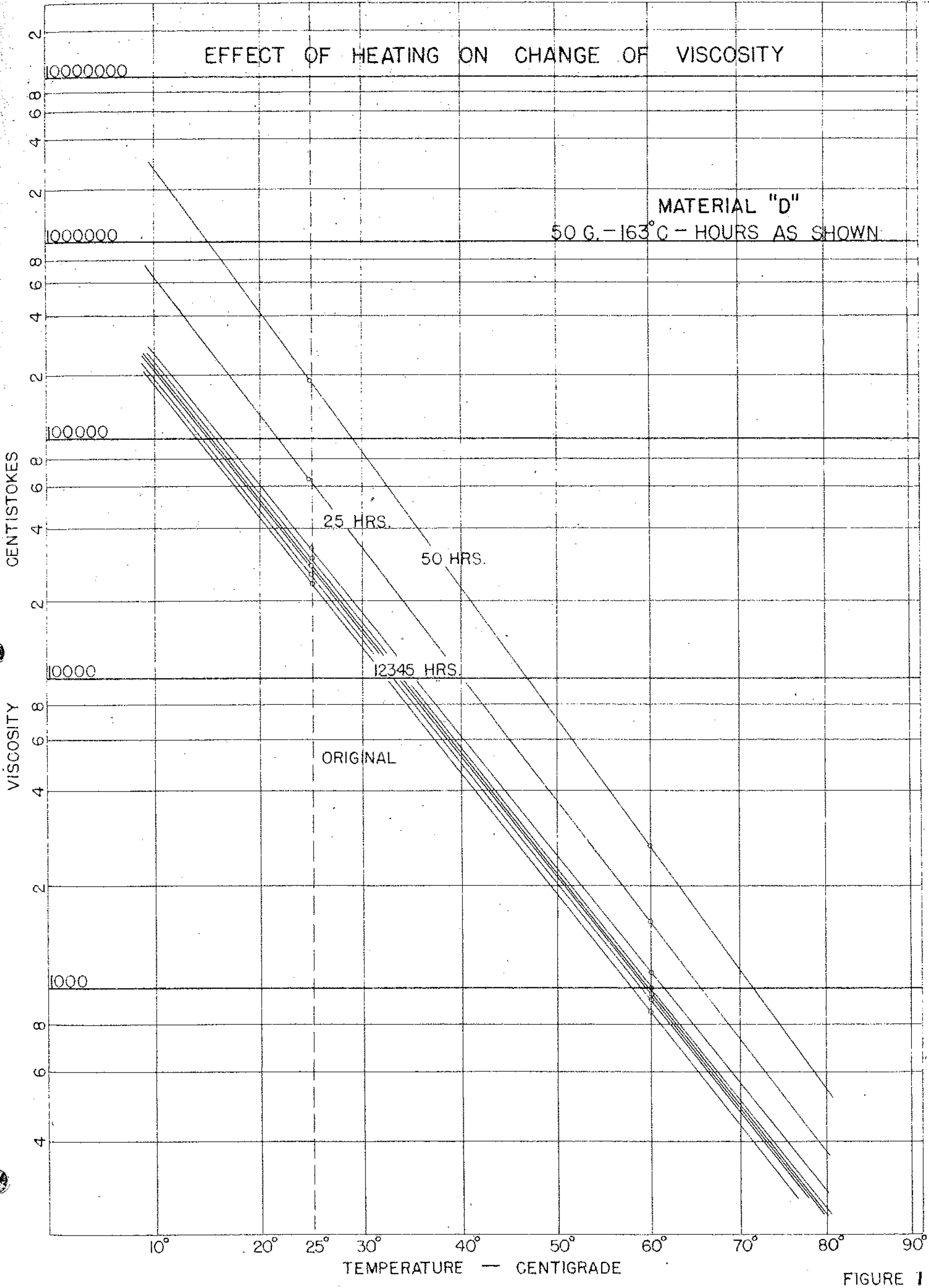

EFFECT OF HEATING ON CHANGE OF VISCOSITY
MATERIAL "D"
50 G. – 163°C – HOURS AS SHOWN
10000000
1000000
100000
10000
1000
VISCOSITY CENTISTOKES
25 HRS.
50 HRS.
12345 HRS.
ORIGINAL
10° 20° 25° 30° 40° 50° 60° 70° 80° 90°
TEMPERATURE — CENTIGRADE

FIGURE 1

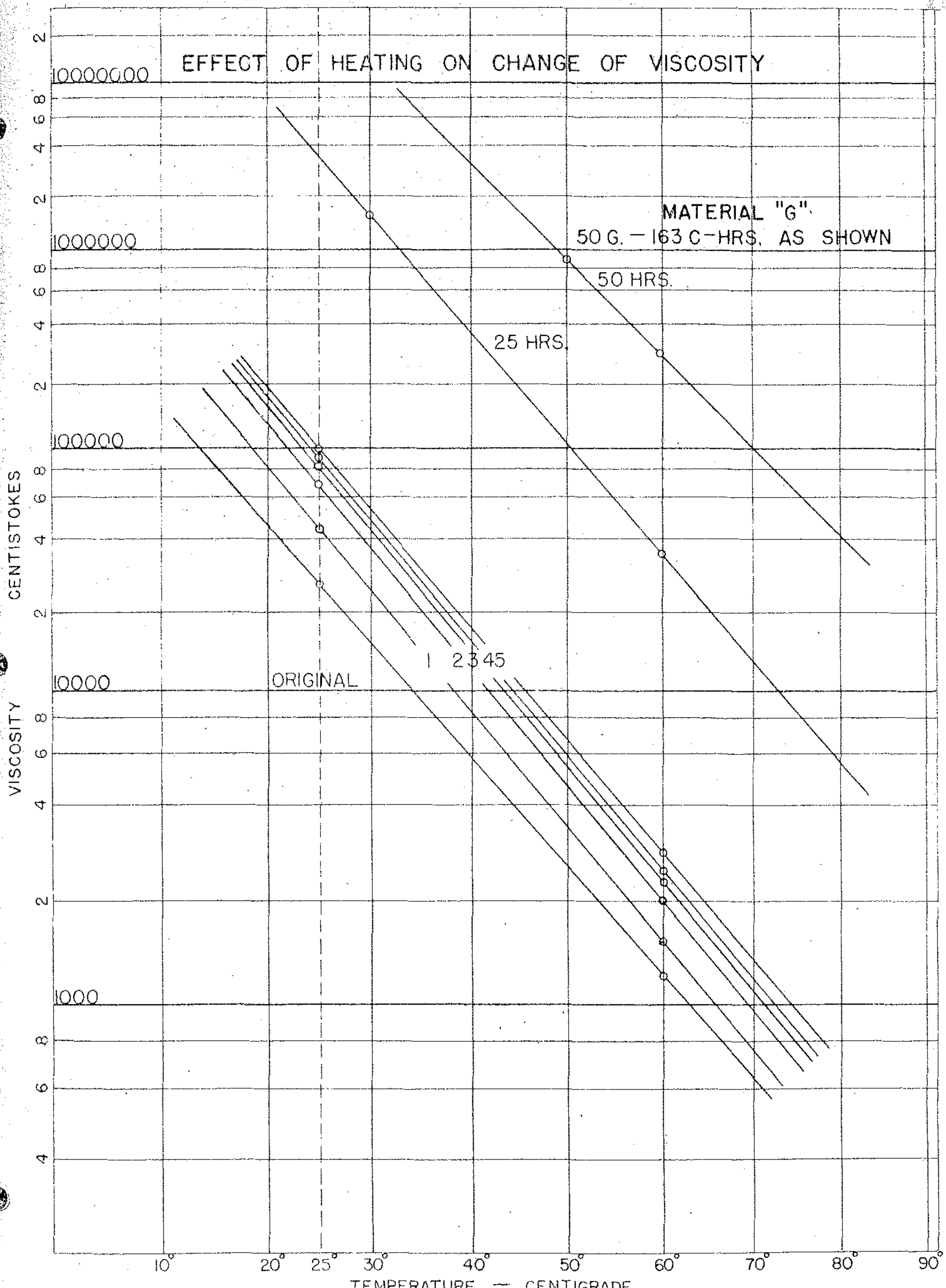

EFFECT OF HEATING ON CHANGE OF VISCOSITY
MATERIAL "G"
50 G. — 163 C—HRS. AS SHOWN
50 HRS.
25 HRS.
1 2 3 4 5
ORIGINAL
10000000
1000000
100000
10000
1000
2
8
6
4
VISCOSITY CENTISTOKES
10°
20°
25°
30°
40°
50°
60°
70°
80°
90°
TEMPERATURE — CENTIGRADE

FIGURE 2

recovery method. The compressed air was bubbled through NaOH to remove carbon dioxide and then H_2SO_4 to remove moisture before passing through a calibrated flowmeter which was regulated to maintain a flow of 500 cubic centimeters per minute. See table 1.

Weatherometer Tests: The fourth treatment involved mixing 15 grams of asphaltic oil with 485 grams of clean 20-30 grade Ottawa sand and exposing the mixture for 168 hours on a weatherometer. The weatherometer was manufactured by the Atlas Electric Device Company, of Chicago, Illinois, and consisted of two arc lamps, one acting from the center and one from the side of the apparatus. The weatherometer has a turntable for supporting the samples. The rotation of the turntable was one revolution in 17 minutes and 35 seconds.

The bituminous mixtures were placed in tins giving an exposed area of 48 square inches. The mixtures were stirred twice daily to insure uniform exposure of all surfaces. The temperature of test was maintained at 140°F by circulating air over the samples. The tests were performed in a dust-free room. The results from the various treatments are presented for comparative study in table 1. Two materials "A and O" were selected as typical samples for representing graphically the Viscosity-Temperature curves. These curves are illustrated in figures 3 and 4.

Effect of continued weathering with weatherometer: Further weathering tests were concluded on material "O" to determine the rate

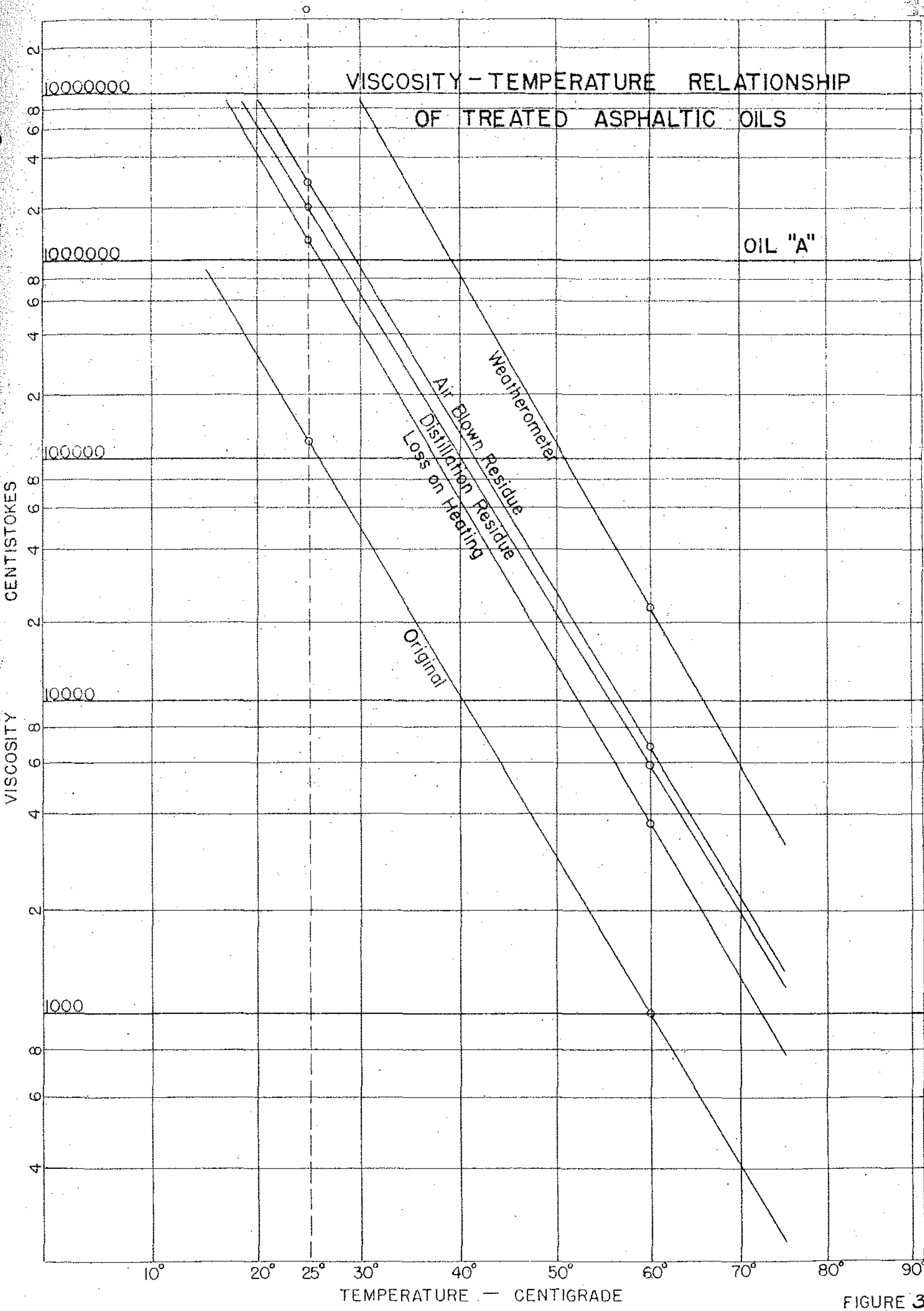

VISCOSITY - TEMPERATURE RELATIONSHIP
OF TREATED ASPHALTIC OILS
OIL "A"
Weatherometer
Air Blown Residue
Distillation Residue
Loss on Heating
Original
VISCOSITY CENTISTOKES
10000000
1000000
100000
10000
1000
TEMPERATURE — CENTIGRADE
10°
20°
25°
30°
40°
50°
60°
70°
80°
90°

FIGURE 3

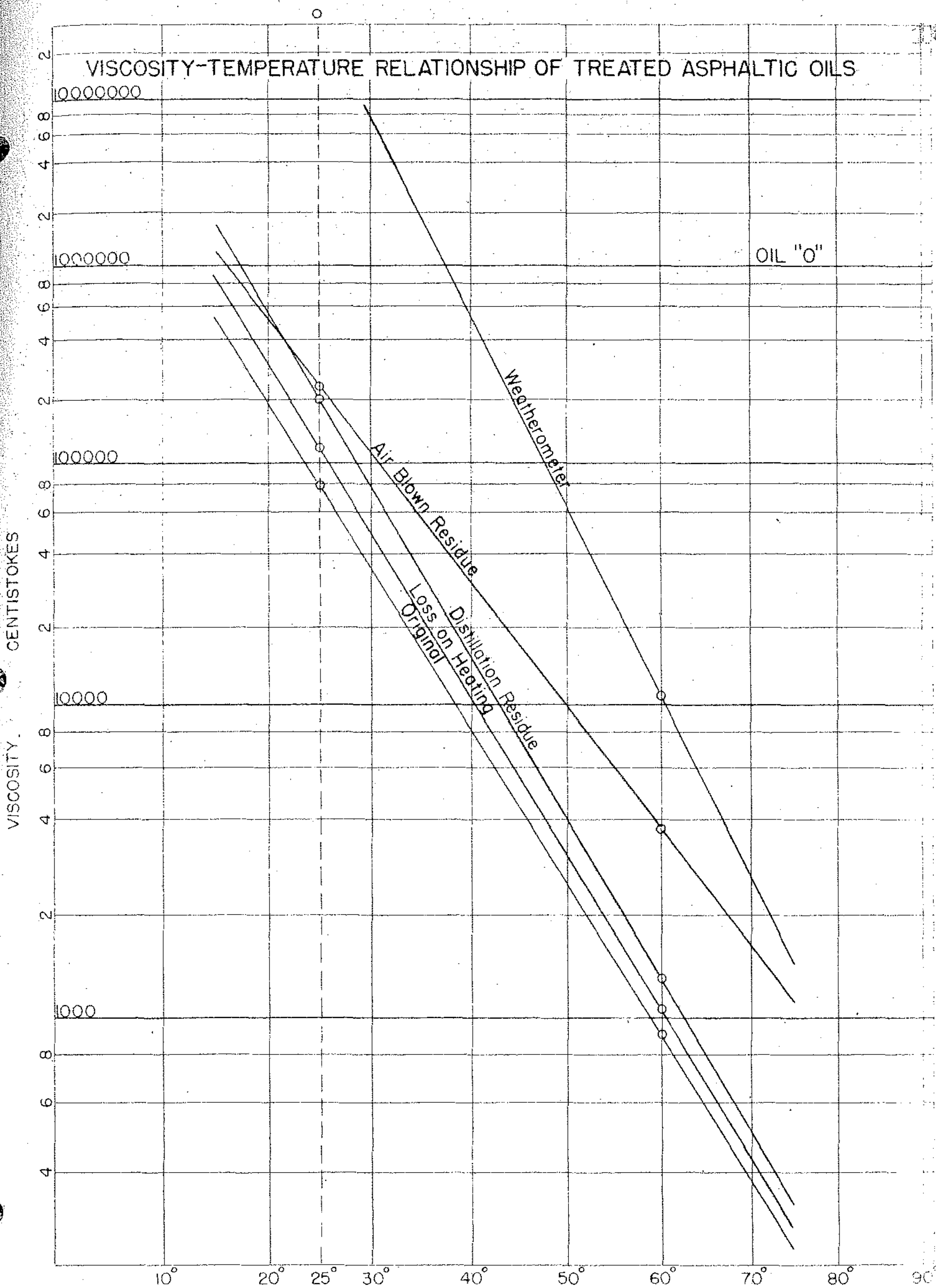

VISCOSITY-TEMPERATURE RELATIONSHIP OF TREATED ASPHALTIC OILS
OIL "O"
10000000
1000000
100000
10000
1000
Weatherometer
Air Blown Residue
Distillation Residue
Loss on Heating
Original
VISCOSITY CENTISTOKES
10° 20° 25° 30° 40° 50° 60° 70° 80° 90°
TEMPERATURE CENTIGRADE

FIGURE 4

of hardening which might be obtained on the weatherometer. "O" was chosen because it appeared to be affected most by the weatherometer test. The material was mixed with Ottawa sand and subjected to same test procedure as defined above for oils "A", "G", and "D", with the exception of time which was varied to give residues at 1 day, 7 days and 12 days. Upon recovery the viscosity and susceptibility of the residue was determined. The results of this study show that the viscosity temperature curves when plotted indicate the rate of hardening of the residue. At the end of 7 days test the residue of material "O" had lost its adhesive properties and appeared to be completely dried out. The viscosity-temperature curves are illustrated in figure 5.

From the data obtained from the oven volatilization test, the distillation test, the air blowing and the weatherometer tests, it may be concluded that the oven volatilization test will give residues comparable to those obtained from the other three tests if the proper length of exposure at 163°C is maintained. The weatherometer test produces the greatest change in consistency of the residue. A presentation of these data* in graph form seems to indicate that the major increase

* For complete information see "Characteristics of Slow Curing Asphaltic Oils", Proceedings of the Association of Asphalt Paving Technologists, Vol. 12, pages 233-275.

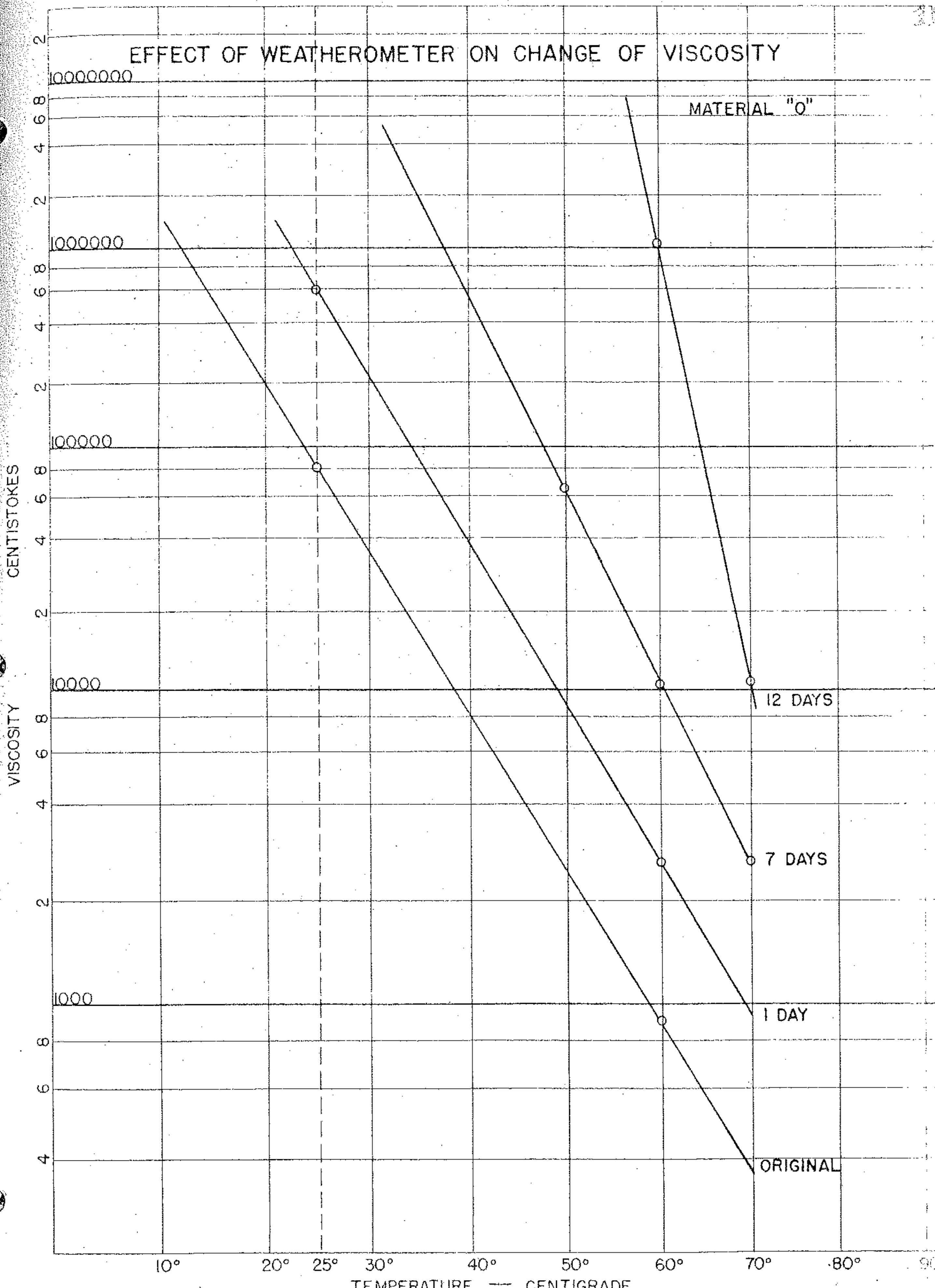

EFFECT OF WEATHEROMETER ON CHANGE OF VISCOSITY
MATERIAL "O"
10000000
1000000
100000
10000
1000
2
8
6
4
VISCOSITY CENTISTOKES
12 DAYS
7 DAYS
1 DAY
ORIGINAL
10°
20°
25°
30°
40°
50°
60°
70°
80°
90°
TEMPERATURE — CENTIGRADE

FIGURE 5

in consistencies of the various residues was caused by evaporation of the volatile materials present in the original asphalt. The slope of the viscosity-temperature curves also indicate that some materials undergo other changes in their characteristics due to conditions imposed by the test.

CHANGE IN CHARACTERISTICS OF SLOW-CURING ASPHALTS AFTER WEATHERING UNDER SERVICE CONDITION

In order to correlate the laboratory studies with changes occurring under service conditions a separate study in the field was made on similar materials. The purpose of this phase of the investigation was to ascertain what changes in viscosity and susceptibility occur in slow-curing asphaltic oils when they are incorporated into a bituminous mixture and subjected to service conditions. It was thought that the results from this study might be helpful in establishing certain definite relationships and to predict the normal rate of hardening of bituminous materials occurring under actual service condition.

To pursue this study, 50 foot test sections were established on newly constructed bituminous surfaces using asphaltic binders of the SC-6 type and from different sources. These test sections were essential in order to facilitate future sampling and to insure that the same source of bituminous material would be obtained throughout the study.

At present there have been 15 test projects established embracing six different types of SC-6 asphaltic oil all meeting Michigan State Highway specifications. Two test projects, No. 1 and No. 2 were established in November 1937, eight test projects were established in July 1939 and five test projects were established in July 1940. The test projects are located throughout the entire state where they will be subjected to varying traffic and climatic conditions.

On each test project a selected sampling area, approximately 50 feet long was definitely located where the source of bituminous material going into the mixture could be ascertained.

Each year samples representing the top half of the road surface will be removed from the sampling area of each test project. The bituminous material will be recovered by a special method* developed for asphaltic materials of the slow curing type. It is contemplated to analyze samples from these test projects over an indefinite period of years.

<u>Study of Changes in Characteristics</u>: At the present time insufficient data has been obtained from the test projects to warrant drawing any definite conclusions. However, the data presented in table 3 for test sections 1 and 2, and illustrated by figures 6 and 7 show very clearly that there is a continuous hardening taking place comparable in some respects to the hardening obtained when the same type of asphaltic materials from similar sources are subjected to the laboratory procedures explained previously. See table 4 for Characteristics of original bituminous materials in test section 1 and 2.

* Changes in Characteristics of Slow Curing Asphaltic Oils, by Finney and Wolczynski, Proceedings of the Association of Asphalt Paving Technologists, Vol. 12, pp. 233-275.

Table No. 3

TEST SECTIONS NO. 1 AND NO. 2
SHOWING THE CHANGE OF VISCOSITY WITH AGING

	Test Section No. 1	Test Section No. 2
Asphaltic Oil in Mix		
Viscosity 25°C	37,786 C.S.	34,529 C.S.
" 60°C	1,135 C.S.	1,074 C.S.
Susceptibility	3.635	3.635
Sp. Gr. 25°C/25°C	.981	.987
Recovered Oil After 1 Year		
Viscosity 25°C	46,631 C.S.	57,927 C.S.
" 60°C	1,539 C.S.	1,703 C.S.
Susceptibility	3.427	3.494
Sp. Gr. 25°C/25°C	.982	.989
Recovered Oil After 2 Years		
Viscosity 25°C	76,212	93,702
" 60°C	1,752	2,393
Susceptibility	3.682	3.477
Sp. Gr. 25°C/25°C	.987	.994
Recovered Oil After 3 Years		
Viscosity 25°C	164,395	129,276
" 60°C	2,928	3,089
Susceptibility	3.679	3.437
Sp. Gr. 25°C/25°C	.990	1.005

Table No. 4

LABORATORY ANALYSIS OF BITUMINOUS MATERIALS
USED IN TEST PROJECTS NO. 1 AND NO. 2

Remarks	Material	Material
Identification	Test Project No. 1	Test Project No. 2
Sp. Gr. 25°C/25°C	.981	.987
Furol Viscosity 60°C	544	531
Flash Point	210°C	160°C
% 100 Pen. Res.	75.0	75.2
Duct. Res. at 25°C	100+	100+
Duct. Res. at 4°C	8.5	9
% Sol. in CCL_4	99.92	99.96
Distillation		
0-225°C	0	0
0-315°C	0	0
0-360°C	Trace	2.5
Loss on Heating		
50 g. 5 hr. 163°C	1.76%	2.93%

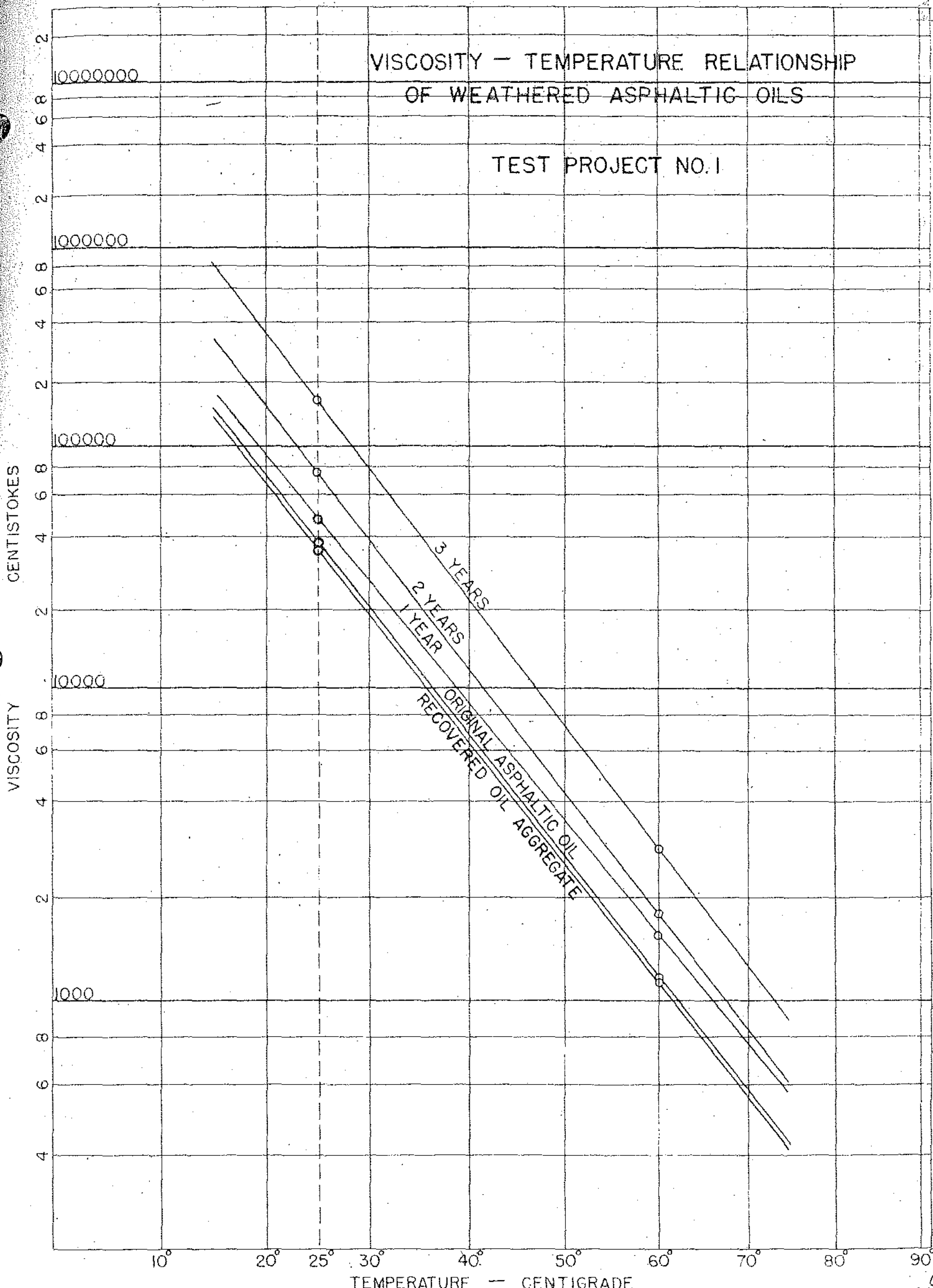

VISCOSITY — TEMPERATURE RELATIONSHIP
OF WEATHERED ASPHALTIC OILS
TEST PROJECT NO. I
10000000
1000000
100000
10000
1000
2
8
6
4
VISCOSITY CENTISTOKES
3 YEARS
2 YEARS
1 YEAR
ORIGINAL ASPHALTIC OIL
RECOVERED OIL AGGREGATE
10°
20°
25°
30°
40°
50°
60°
70°
80°
90°
TEMPERATURE — CENTIGRADE

FIGURE 6

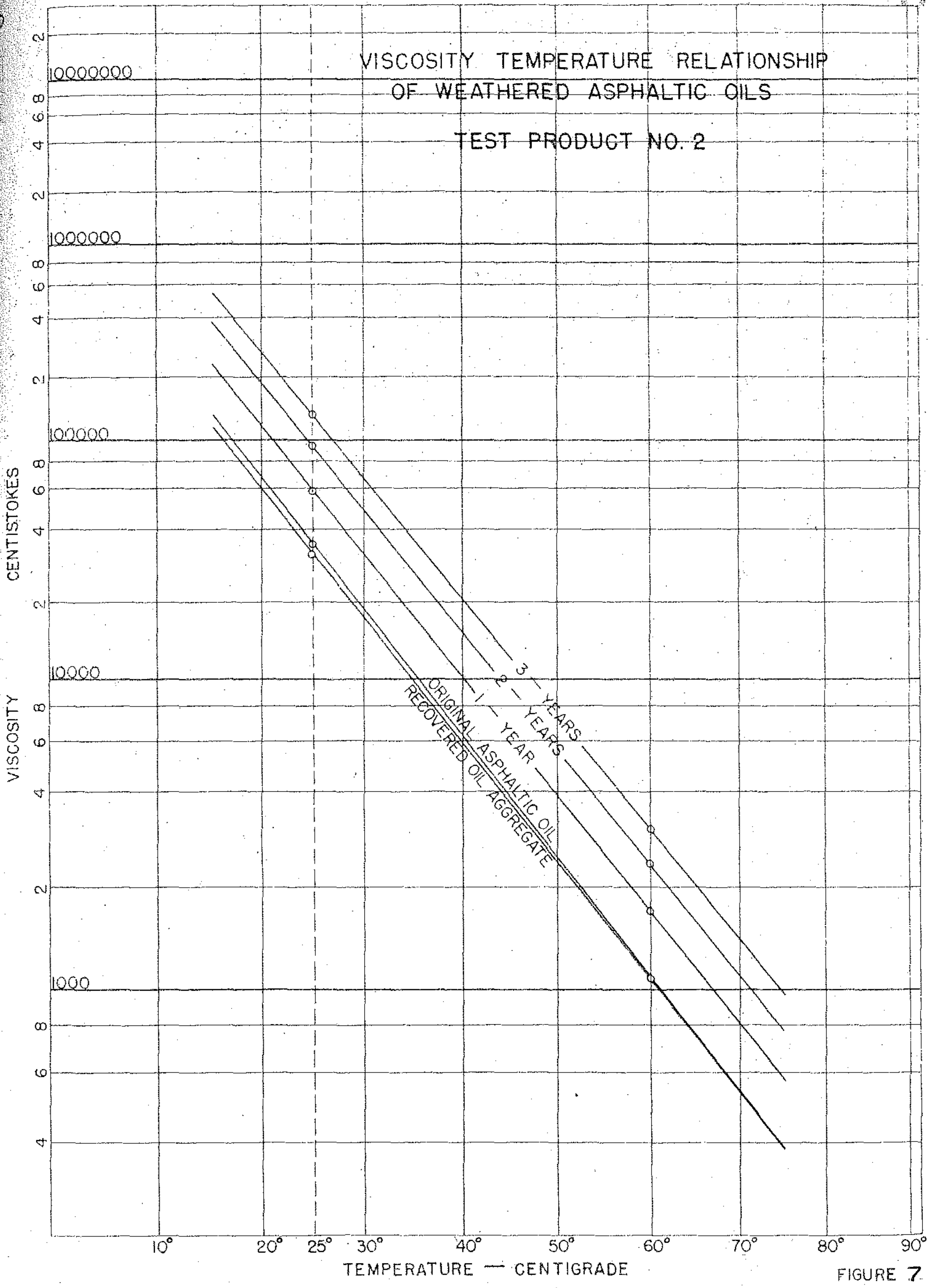

VISCOSITY TEMPERATURE RELATIONSHIP
OF WEATHERED ASPHALTIC OILS
TEST PRODUCT NO. 2
VISCOSITY CENTISTOKES
10000000
1000000
100000
10000
1000
3 — YEARS
2 — YEARS
1 — YEAR
ORIGINAL ASPHALTIC OIL
RECOVERED OIL AGGREGATE
10°
20°
25°
30°
40°
50°
60°
70°
80°
90°
TEMPERATURE — CENTIGRADE

FIGURE 7

On the same materials, a comparative study of the viscosity-temperature curves of residues from the oven volatilization test shown in figure 1 with the viscosity-temperature curves of bitumen recovered from two test projects in figures 6 and 7 shows quite conclusively that the oven volatilization test with certain modifications with respect to time of heating, can be used to evaluate the relative rate of hardening or durability of slow curing asphaltic materials. For instance, in comparing 25 hour curve and 50 hour curve in figure 1 and the 1 year and 2 year curves in figure 6, there is noted a close relationship.

The viscosity-temperature curves in figures 6 and 7 also show that the rate of hardening of the two recovered bituminous materials after three years is practically the same.

Figure 8 illustrates in another manner the rate of hardening of the bituminous materials from the two test projects when the viscosity of the recovered asphalt at 60°C is plotted against time in years.

This study indicates that the hardening of an oil asphalt of this SC-6 type over a period of years may be predicted from laboratory tests such as the loss on heating test.

As determined, the criteria for allowable changes in the oil asphalt by laboratory tests must be established upon the changes which have occurred in pavements laid and subjected to service over a period of years. The characteristics of satisfactory and unsatisfactory materials must be known and limits established.

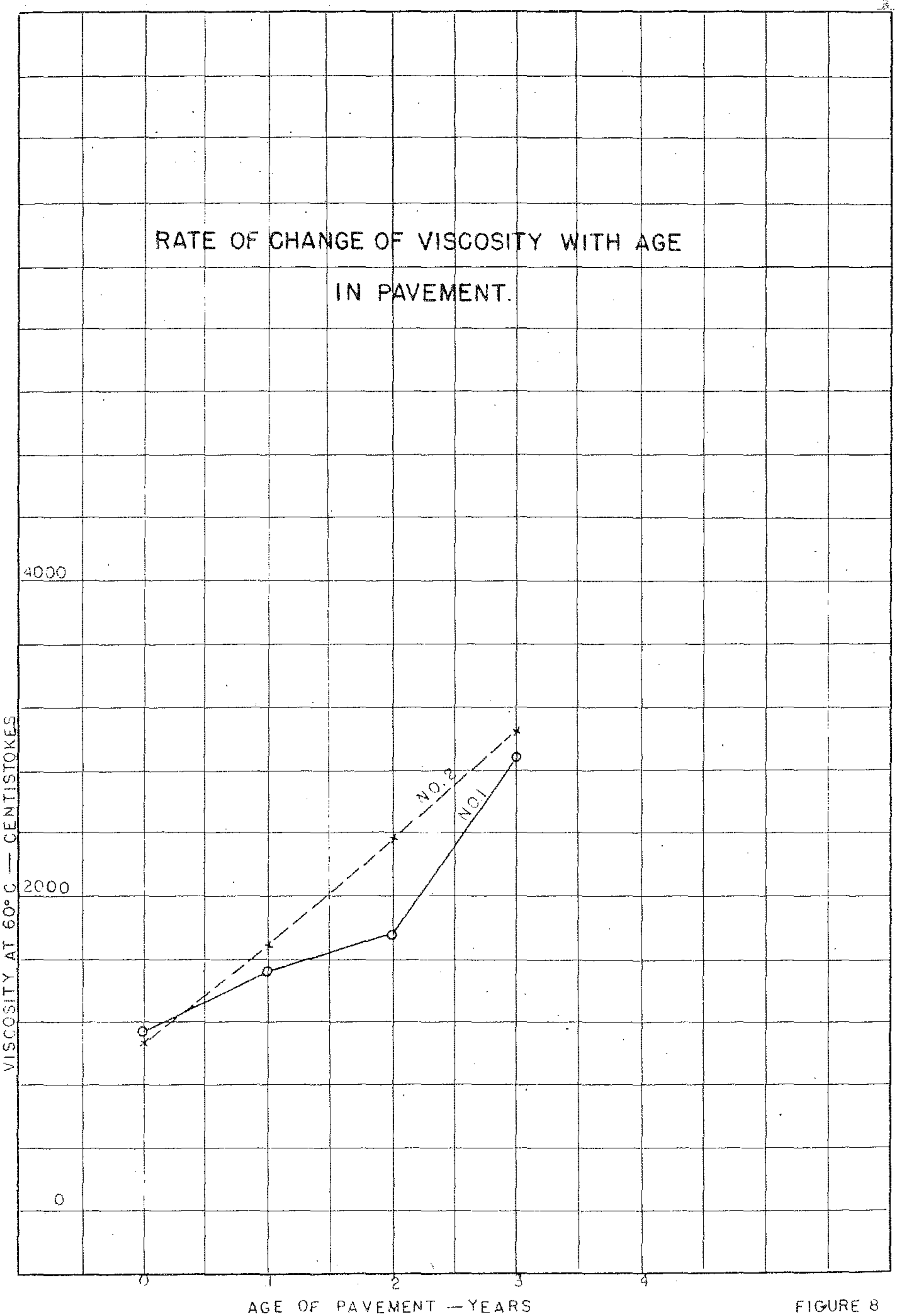

RATE OF CHANGE OF VISCOSITY WITH AGE
IN PAVEMENT.
4000
2000
0
VISCOSITY AT 60° C — CENTISTOKES
NO. 2
NO. 1
0
1
2
3
4
AGE OF PAVEMENT — YEARS

FIGURE 8

CONDITION SURVEY OF EXISTENT BITUMINOUS SURFACES USING SC-6 ASPHALTIC MATERIALS AS BINDER

During the summer of 1940, an extensive survey was made, including a greater portion of the slow-curing bituminous pavements in the state. The purpose of the survey was to study the characteristics of recovered bitumen from surfaces showing normal and abnormal changes typical of aging oil aggregate surfaces. Particular attention was paid to the source of asphalt, age of road, and physical condition of the surface. From this survey, 28 bituminous surfaces were selected for laboratory study. Samples of the mixture were taken from the top course of each project and subjected to laboratory analysis.

In judging the criteria of the complete failure of the bituminous substance in the pavement, the following factors were kept in mind:

1. Conditions must be general, not localized to a few isolated spots or at edges or on hills.
2. That subgrade failures can cause ravelling, alligatoring and cracking of the surface.

This survey revealed that no bituminous surface had failed completely, although a few showed considerable signs of wear and excessive alligatoring, requiring a seal coat or surface treatment to place them in good condition. The alligatoring was caused by poor subgrade conditions which were known to exist prior to laying of the pavement.

However, the most important result of this survey is the realization that many of the surfaces examined have given fair service behavior to date, yet they reveal that abnormal hardening of the bituminous binder has taken place and that the original flexible mat has changed entirely to a rigid type of pavement similar to bituminous concrete. The bituminous mat when once disturbed or broken up cannot be recompacted without additional bituminous material, flux or application of heat. The recovered bitumen from bituminous surfaces of this nature had viscosity values of 4200 centistokes or higher at 60°C, thus indicating a 300 to 400 percent increase over the original value. The pavements possessing abnormal hardness were between three and four years old. The fact that this premature change in bituminous surfaces of the slow curing type is caused by abnormal hardening of the bituminous binder is brought out by further tests which show that the recovered bitumens from a majority of pavements one to five years of age, irrespective of the source of bitumen, had viscosity values at 60°C ranging from 1400 to 3200 centistokes or 700 to 1600 furol seconds respectively. At 60°C the viscosity of slow-during asphaltic materials of the SC-6 type is in the neighborhood of 1000 centistokes or 500 furol seconds. To further illustrate the variation in hardening which might be expected, four pavements were found from which the recovered bitumen showed a viscosity greater than 54,000 centistokes or 27,000 furol seconds at 60°C. Also, it was observed that in some cases the bituminous material derived from the same processing and source, became abnormally hard in one surface while in another it remained soft and flexible over the same period of time. The survey data is presented in table 5.

The degree of hardening throughout the bituminous mat is another factor to be considered. In general, it was found that the bituminous mat consisted of a hard crust at the exposed surface varying in thickness as much as 1 inch and the mixture under the crust was live and workable. It is evident that the progressive hardening of the mat which is characteristic of this type of bituminous road may develop in two ways, either by a slow rate of hardening from the exposed surface of the mat towards the base, or by a gradual and uniform progress throughout the entire mat.

Information shown in table 5 indicates the possibility of other factors affecting the relative rate of hardening of asphaltic materials in bituminous surfaces such as the permeability of the mat surface, the density of the mixture, the composition of the mineral filler and to some extent the petrogenic characteristics of the aggregates. But the laboratory and field control tests would indicate that abnormal hardening is a characteristic of the asphalt.

The studies made in the laboratory and field have contributed important information relative to the changes in characteristics of the SC-6 oil asphalts. It is realized that more data would allow more definite conclusions to be formulated, but it is evident that developmental criteria has been obtained to improve sepcificatíons which may be enumerated as follows: first, that the standard loss on heating test may be modified whereby it can be used as an accelerated serviceability test or rate of hardening test; second, that when the viscosity of the asphaltic material in an oil aggregate surface has increased more than 400 percent, the surface cannot be reworked by ordinary methods and third, assuming that 400 percent increase in viscosity is the limit for a satisfactory product, it is apparent from figure 8 that a good oil aggregate surface will reach an unworkable state in a period of from 7 to 10 years.

SUMMARY OF DATA RELEVANT TO PROPOSED CHANGES
IN SPECIFICATION
FOR SLOW-CURING LIQUID ASPHALTIC MATERIAL

Specifications for bituminous materials include tests which are identification tests as well as those intended to measure durability or serviceability. Further research may indicate that durability is related to some of the laboratory tests discussed herein. But for the present, the Michigan State Highway Department is interested in specifications and tests which will provide uniform material as well as those which determine the ability of the material to meet certain construction and maintenance requirements. This does not mean that we are not interested in the durability of the material, but it is evident that more research work must be done before test limitations can be set up to control this characteristic.

The use of the present specifications for "Slow Curing Asphaltic Oils of the SC-6 Grade" does not give satisfactory and uniform material meeting construction and maintenance requirements.

Therefore, in light of the research work which has been discussed previously it is believed that certain changes in specifications will give an improved uniform and serviceable material.

Such changes should insure better uniformity between tank car shipments from a given source as well as a standard material from different sources. The changes should also provide a product having a slow uniform rate of hardening when incorporated into the road surface.

The non-uniformity in the characteristics of liquid asphaltic materials per tank car shipments for the year 1940 has been shown in graph form in the report by the Testing Division on the Specification Changes. These graphs show very clearly the wide variation in plant control which exists at certain refineries. These variations definitely show that the specification limits in some cases are too wide, yet at the same time show that materials may be obtained from several sources within much narrower ranges.

It is only logical that such wide variations in tank car shipments will in time affect the production of uniform oil aggregate mixtures.

Further, a study of laboratory analysis of tank car shipments, presented in the report by the Testing Division, show a marked variation in similar characteristics of bituminous materials shipped from different refineries. The results from laboratory and field studies illustrated in tables 5 and 6 also show the wide variation in serviceability characteristics which exist in materials from different refineries. Therefore, it is only natural to expect that when products from different sources are not controlled closely, non-uniform conditions will result in the road surface. Also, it is the concensus of opinion that the abnormal hardening of a liquid bituminous material is due primarily to the evaporation of the oily

TABLE NO.5

SURVEY OF EXISTING SLOW-CURING ASPHALTIC PAVEMENTS

Laboratory Designation	Source of Bit. Mat.	Age in Years	Grade of Bit. Mat.	Original Viscosity 60°C Centistokes	Recovered Viscosity 60°C Centistokes	Bitumen Content Recovered Sample	Susc.	Surface Condition	Subgrade Condition	Richness of Surface Mixture	Deterioration	Observation on Removing Sample From Pavement
40 BR-14	V	3	SC-4A	1068	1410	2.69%	3.587	Excellent	Good	Normal	Sound	Live
40 BR-4	V	4	"	892	1465	2.53%	3.348	"	"	"	"	"
40 BR-25	V	1	"	1138	1506	2.62%	3.489	"	"	"	"	"
40 BR-39	VIII	1	"	1112	1856	3.01%	3.358	"	"	"	"	"
40 BR-26	VI	1	"	974	2034	3.47%	3.333	"	"	"	"	"
40 BR-40	VIII	1	"	1112	2116	3.15%	3.391	Bad Spot	Poor	"	Alligatored	"
40 BR-1	II	5	SC-3A	486	2131	2.05%	3.422	Fair	Good	"	Sl. Worn	"
40 BR-15	V	3	SC-4A	1068	2140	3.35%	3.414	Bad Spot	Poor	"	Alligatored	"
40 BR-22	VII	1	"	1112	2156	2.40%	3.464	"	"	"	"	"
40 BR-3	III	5	"	873	2236	3.59%	3.649	Excellent	Good	Sl. Rich	Sound	"
40 BR-23	V	1	SC-7	1780	2309	3.05%	3.585	Fair	"	Normal	"	"
40 BR-10	V	3	SC-4A	984	2323	3.33%	3.558	Excellent	"	"	"	"
40 BR-24	VI	1	"	1289	2365	2.63%	3.440	"	"	"	"	"
40 BR-17	V	3	"	1079	2428	2.65%	3.534	"	"	"	"	"
40 BR-21	VII	1	"	1112	2553	2.49%	3.507	Fair	"	"	"	"
40 BR-12	V	3	"	1062	2708	3.93%	3.504	Bad Spot	Poor	"	Alligatored	"
40 BR-16	I	3	"	1030	2804	2.98%	3.724	Fair	Good	"	Sl. Worn	"
40 BR-18	V	3	"	1065	2928	3.14%	3.679	Fair	"	"	Sound	"
40 BR-19	IV	3	"	1055	3089	2.68%	3.437	"	"	"	"	"
40 BR-2	V	5	"	1062	3161	2.33%	3.493	"	"	"	"	"
40 BR-9	IV	4	"	1190	3352	2.91%	3.566	Excellent	"	"	"	"
40 BR-20	IV	3	"	1055	4268	2.56%	3.810	Bad Spot	Poor	Sl. Dry	Alligatored	Hard Brittle Top
40 BR-8	IV	4	"	985	4540	2.44%	3.583	Excellent	Good	"	Sound	Hard Top-Very Live Base
40 BR-5	V	4	"	942	4594	3.05%	3.676	Fair	"	"	"	Hard Top-Live Base
40 BR-13	V	3	"	1233	4672	2.37%	3.455	Poor	"	"	Worn	Hard Top-Live Base
40 BR-11	V	3	"	1082	54,384	3.70%	3.587	Excellent	"	"	Sound	Hard Top-Live Base
40 BR-7	V	4	"	952	1,059,542	1.77%	4.113	Bad. Cond.	"	"	Badly Worn	Top Brittle-Base Live
40 BR-6	V	4	"	961	1,630,848	3.58%	4.722	Fair	"	Normal	Sl. Cracked	Hard Brittle-Both Course

MICHIGAN
STATE HIGHWAY DEPARTMENT

G. Donald Kennedy
State Highway Commissioner

RESEARCH DIVISION

Table No. 6

CHARACTERISTICS OF VARIOUS SC 4A-6A OILS USED IN MICHIGAN

			VISCOSITY						CONDITION	
Sample Number	Flash Point	Percent Loss on Heating	Original Furol at 60°C	Residue Loss on Heating	Percent Change	Recovered	Percent Change	Age	Appearance	Workability
LION OIL										
D	214	0.43	420	480	14.3					
BR-2	217		492			1580	221	5	Fair	Live
BR-4	184		425			733	72.5	4	Excellent	Live
BR-5	183		438			2297	424	4	Excellent	Hard
BR-10	174		456			1162	155	3	Excellent	Live
BR-11	214		503			27192	532	3	Excellent	Hard Top-Live Base
Br-12	211		492			1354	175	3	Bad Alligatored	Live
BR-13	156		572			2336	309	3	Bad	Intermediate
BR-14	205		494			705	42.7	3	Excellent	Live
BR-15	205		494			1070	116	3	Bad	Live
BR-17	210	1.76	547			1214	122	3	Excellent	Live
BR-18	210		540			1461	170	3	Fair	Live
BR-25	209		528			753	42.6	1	Excellent	Live
BR-38	185		432			91.745	2120	4	Bad	Hard
O-B	215	0.65	575							
	214	0.61	550	645	17	722	31	0		
T-1	210		544			550	1.12	0		
						770	41.6	1		Live
						876	61.0	2		Live
						1464	169	3		Live
T-5	212		540			602	11.5	0		Live
						678	25.5	1		Live
T-6	212		534			535	0.0	0		
						772	46.0	1		Live
T-7	213		534			578	8.2	0		
T-14	215	0.46	566			1360	154	1		Live
T-15	220	0.26	540			571	0.8	0		
						594	10.0	0		
STANDARD NEW JERSEY										
BR-8	160	1.5*	457			2270	397	4	Excellent	Hard Top-Live Base
BR-9	160	1.5*	552			1676	220	4	Excellent	Live
BR-19	154	2.93	489			1544	216	3	Fair	Live
BR-20	154	2.93	489			2134	337	3	Bad	Hard Top
T-4	163		429			933	117	0		
						1059	147	1		
T-2	160	2.93	531			537	1.1	0		Live
						851	60.3	1		
						1197	125	2		
						1544	191	3		
AMERICAN BITUMULS										
BR-16	174		478			1402	214	3	Fair	Intermediate Live
P	390	1.13	507	675	33.2					
TEXAS NORFOLK										
BR-21	140	5.0*	515			1276	148	1	Fair	Live
BR-22	140		515			1078	109	1	Bad	Live
TEXAS LOCKPORT										
BR-24	148	1.0*	597			1182	98.2	1	Excellent	Live
BR-26	161	0.7*	451			1017	125	1	Excellent	Live
O-F	143	5.59	500			1764	253	0		
O-F	140	4.32	509	1178	132					
TEXAS - LAWRENCEVILLE										
BR-39	160	1.0*	517			928	79.5	1	Excellent	Live
BR-40	160	1.0*	517			1058	105	1	Bad	Live
T-8	152		493			566	14.8	0		Live
						844	71.2	1		Live
WYOMING - TEXAS										
T-9	146		603			1250	107.2	0		
						1351	124	1		Live
Wy-I	140	1.1	498	1358	172					
NATIONAL REFINING										
O-H	141	3.97	489			1151	235	0		
O-H	141	5.5	479	1221	157					
MID-CONTINENT										
O-C	218	0.47	555	616	13					
T-3	218		535			584	9.2	0		
PRODUCERS - MICHIGAN										
O-D	162	3.05	466			1070	130	0		
O-D	195	1.83	527	902	42					
SHELL - ILLINOIS										
O-9	150	2.35	524			3045	540	0		
O-9	143	4.12	417	1190	188					
SHELL - LOUISIANA										
G	140	8.90	593	1354	128					
INDIAN REFINING										
OE	160	2.65	493			892	81	0		
OE	170	2.23	517	762	49					
T-11	140	3.08	474			500	5.5	0		
T-12	160	2.58	434			462	6.4	0		
T-13	160	2.58	505			544	7.7	0		

* Percent distillate - 360°C

(BR-2) represents samples from Field Survey
(T-5) represents samples from Test Roads
(A) represents samples from Research Division Laboratory Study
(O-F) represents samples from Testing Division Laboratory Study

Table No. 7

SUMMARY OF VISCOSITY CHANGE OF RESIDUE
FROM LOSS ON HEATING AND RECOVERY TESTS

Material	Loss on Heating Residue % Increase				Recovery Residue % Increase			
	Time	Max.	Min.	Average	Time	Max.	Min.	Average
1. Lion Eldorado	0	17	17	17	5 yr. 4 yr. 3 yr. 2 yr. 1 yr. 0 yr.	221 424 532 61 154 31	221 72.5 42.7 61 41.6 0	221 248 199 61 62 9
2. Standard of N.J.					4 yr. 3 yr. 2 yr. 1 yr. 0 yr.	397 337 125 147 117	220 191 125 60 1.1	308 248 125 103 59
3. American Bitumuls Balt.	0	33.2	33.2	33.2	3 yr.	214	214	214
4. Texas Norfolk					1 yr.	148	109	128
5. Texas Lockport	0	132	132	132	1 yr. 0 yr.	125 253	98.2 253	113 253
6. Texas Lawrenceville	0	14.8	14.8	14.8	1	105	71.2	85.2
7. Texas Wyoming	0	172	172	172	—	—	—	—
8. National Refinery	0	157	157	157	0	235	235	235
9. Mid Continent	0	13	13	13	0	9.2	9.2	9.2
10. Producers Michigan	0	42	42	42	0	130	130	130
11. Shell - Illinois	0	188	188	188	0	548	548	548
12. Shell - Louisiana	0	128	128	128	—	—	—	—
13. Indian Refinery	0	49	49	49	0	81	5.5	43.2

constituents of the bituminous material. Therefore, either a straight run product or a product cut-back with a very low percentage of high flash volatile matter would be desirable for use in Michigan.

Therefore, with respect to the above mentioned conditions, it is believed that the following revisions in current specifications for SC-6 slow curing asphaltic materials relative to flash point, loss on heating, viscosity of loss on heating residue, original viscosity, percentage of 100 penetration residue, as well as the addition of the emulsion test and the ash content test will aid materially to insure delivery of satisfactory products. Each of these tests and recommended limits will be discussed in detail.

Flash Point Temperature:

It is apparent that the present flash point limit of 93.3°C+ is entirely too low. A study of the flash point values plotted in figure 9 based on a seasons supply of liquid asphaltic materials of the 6A Grade, show that the lowest value was 130°C. The flash point values lying between 130°C and 155°C are scattered widely which might indicate lack of proper control at the refinery, thereby resulting in a non-uniform product to the contractor.

The points plotted to the right of 155°C, in figure 9, are fairly well grouped, thereby indicating better control on the part of the manufacturers. Also, the bituminous materials having a flash

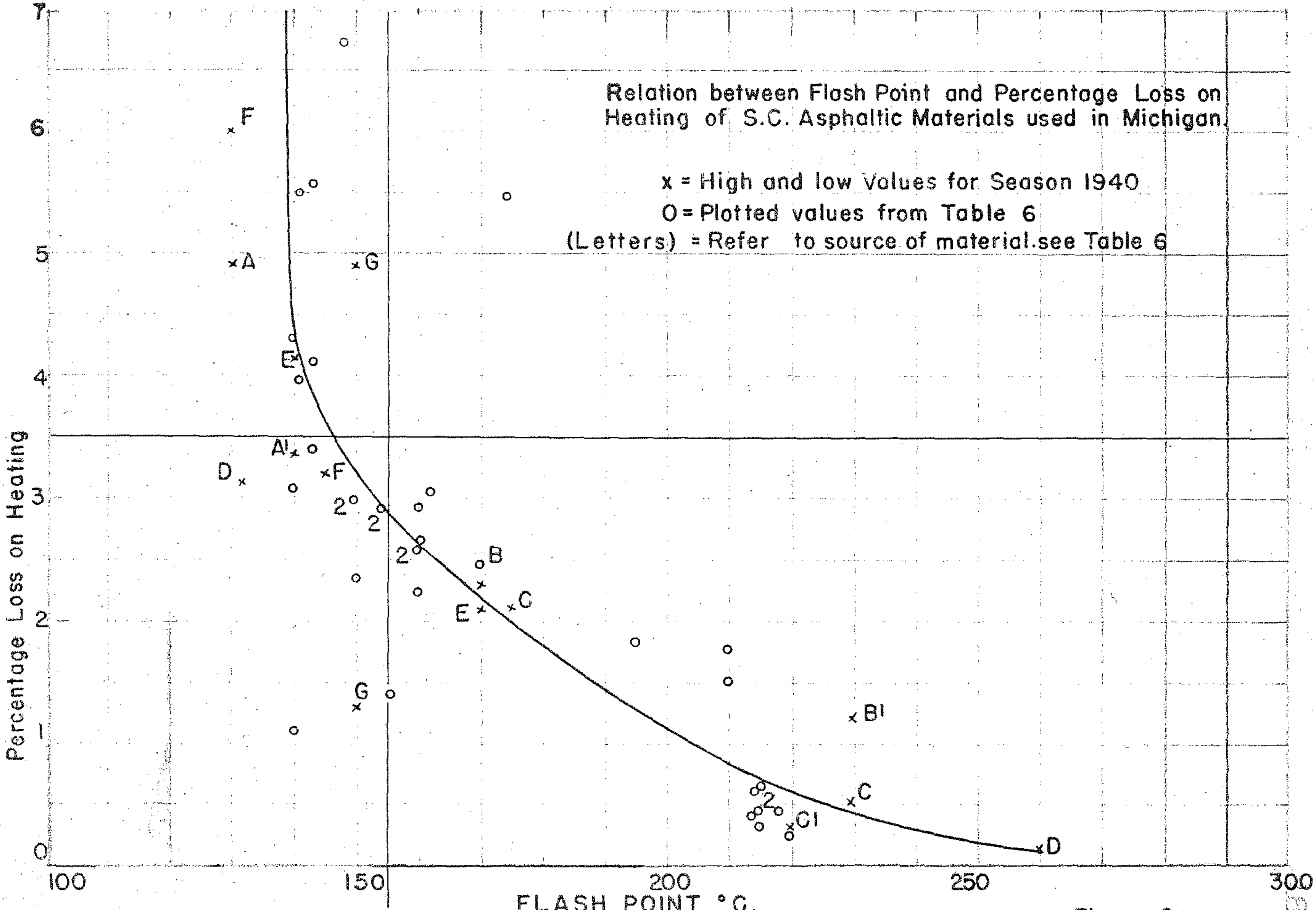

Relation between Flash Point and Percentage Loss on Heating of S.C. Asphaltic Materials used in Michigan.
x = High and low Values for Season 1940
O = Plotted values from Table 6
(Letters) = Refer to source of material. see Table 6
Percentage Loss on Heating
FLASH POINT °C.
0
1
2
3
4
5
6
7
100
150
200
250
300
F
A
G
E
A1
D
F
2
2
2
B
E
C
G
B1
C
2
C1
D

Figure 9

point greater than 155°C, must either be a straight run product or a product cut back with a very small amount of volatile material.

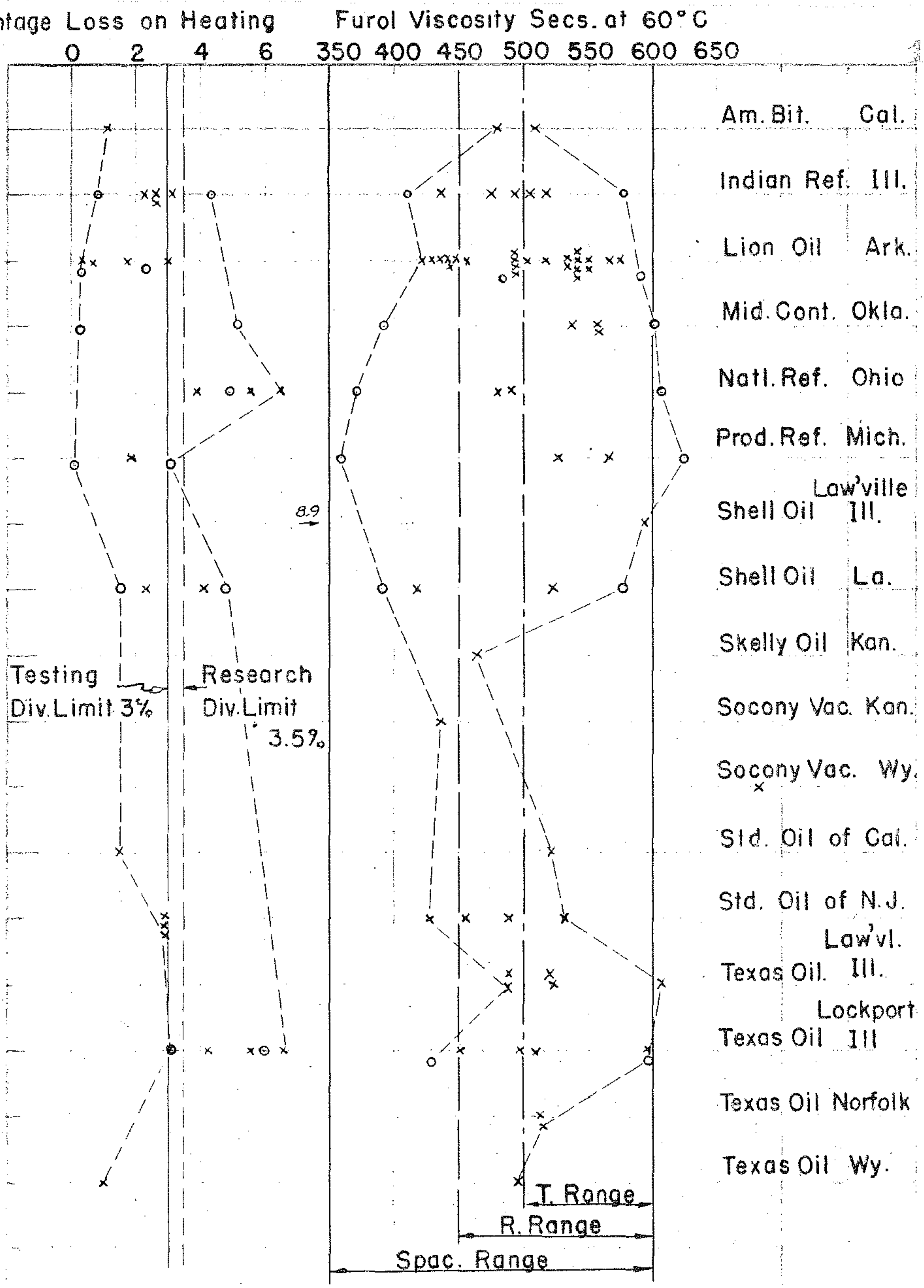
Consistent with these facts it is recommended that the flash point temperature be changed from 93.3°C+ to 155°C+.

Loss on Heating Percentage:

A study of the curve in figure 9 shows that a definite relationship exists between the flash point temperature and the percent of volatile material evaporated off during the loss on heating test. It is evident from the curve in figure 9 that 3.0 percent loss on heating is consistent with a flash point of 155°C. Also, very few of the bituminous materials used during 1940 had a loss on heating greater than 3.5 percent. See figure 10. Therefore, it is recommended that the present loss on heating value of 6 percent be reduced to 3.5 percent to conform with the proposed increase in flash point temperature.

Viscosity of Residue from Loss on Heating Test:

The Public Roads Administration, in their work on asphalts, conclude that a change in consistency of an asphalt after subjection to a 5 hour thin-film oven test offers a means of evaluating its durability when incorporated into a bituminous mixture and after exposure to service conditions. Work done by the Michigan State Highway Department and described previously in this report shows quite conclusively that the oven volatilization test, with certain modification as to quantity of material involved in the test, can be used to evaluate



Relation between Initial Viscosity, Percentage Loss on Heating and Source of Material.

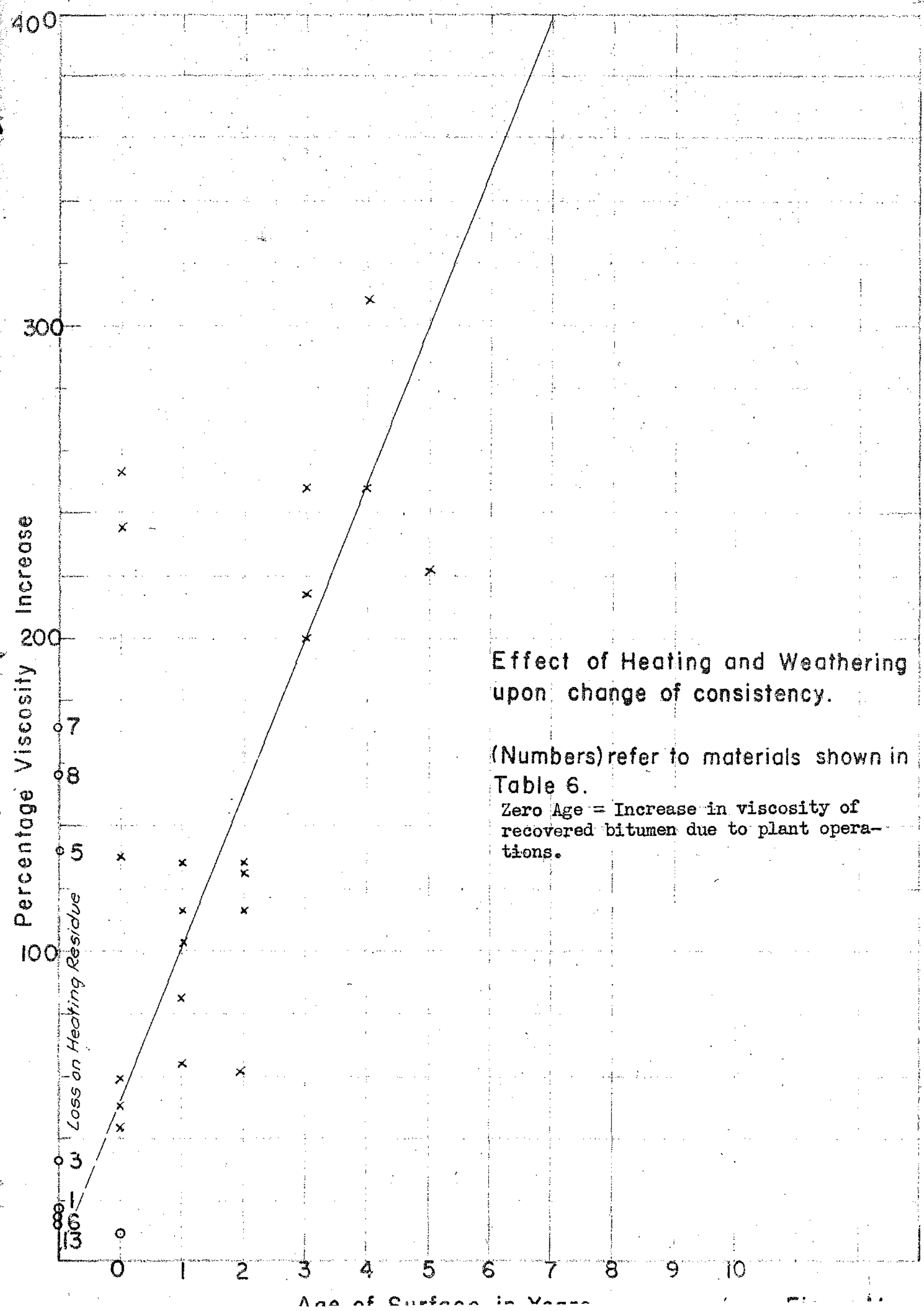
Figure 10

the relative rate of hardening or serviceability of slow curing liquid asphaltic materials.

Further studies relative to the change in consistency of recovered bitumens after definite service periods show that when the viscosity of a slow curing asphalt has undergone a change of 300 - 400 percent, the road surface becomes hard and cannot be reworked by normal methods. The trend of the curves shown in figure 8 indicate that this stage of hardening may occur in approximately 7 to 8 years, especially for the particular materials represented by the curves. Thus, the rate of viscosity increase per year would be approximately 50%. However, the fact that a certain change takes place during mixing operations must be considered. Tests on recovered samples of different bituminous materials taken from the road surface immediately after placing, show a change in viscosity ranging from 9.2 to 253 percent, depending upon the brand of material.

On the basis of the data presented in tables 6 and 7 and illustrated by the graph in figure **11**, it seems logical to require that the viscosity of a recovered bitumen should not increase more than 100% due to mixing operations. This amount of change in viscosity is equivalent to approximately 2 years of service for a good bituminous material.

A requirement placed on the viscosity of the residue of the standard loss on heating test would be more desirable than the Recovery



Effect of Heating and Weathering upon change of consistency.

(Numbers) refer to materials shown in Table 6.

Zero Age = Increase in viscosity of recovered bitumen due to plant operations.

Test because it could be made at the same time as the other laboratory tests and would serve as a check on the material before its use in the road surface. In general, the average percent of viscosity increase of the residue from the loss on heating test is lower than that of the recovery test, and perhaps could be established at a lower figure. However, until such a time when more pertinent data is available, it is felt that a requirement of 100% minus, would not be out of line with the proposed changes in the requirements for the flash point and loss on heating tests. Therefore, it is recommended that a new requirement be embodied in the specification relative to a definite percent increase in the viscosity of the residue of the Standard Loss on Heating Test over that of the original material. The percentage increase shall be not more than 100%. Furol sec. at 60°C.

Viscosity Range of Original Material:

A study of the characteristics of SC-6A asphaltic materials used in Michigan during 1940 and illustrated in figure 10 show the following to be true:

1. That the viscosity values are spread throughout the present specification range of 350 sec. to 600 sec. With such wide viscosity variations in tank car shipments it is only natural to assume that non-uniform bituminous mixtures will result since the contractor has no way of adjusting mixing

operations to conform to erratic changes occurring in tank car shipments of bituminous materials.

2. In all cases the various manufacturers who have produced materials having a viscosity lower than 500 sec. have also furnished materials having viscosities greater than 500 secs.

3. That, in general, the materials with a viscosity of 450 secs. or higher will have a loss on heating percentage lower than 3.5%.

4. That a narrowing of the viscosity range should result in a more uniform product.

Consistent with the above facts, it is recommended that the viscosity limits of the slow curing liquid asphalt considered in this study be changed from 350 secs. to 600 secs. to 450 secs. to 600 secs.

Percentage of 100 Penetration Residue:

The Asphalt Institute, in their Construction Series Bulletin No. 52 on Slow Curing Liquid Asphaltic Road Materials, recommend that the percentage of 100 penetration residue for an SC-6 material should be 80 percent plus. Laboratory analysis of car samples obtained during 1940 reveal that practically all of the materials fell into a range between 75 and 80 percent 100 penetration residue. See figure 12. Only two samples have a value lower than 75 percent.

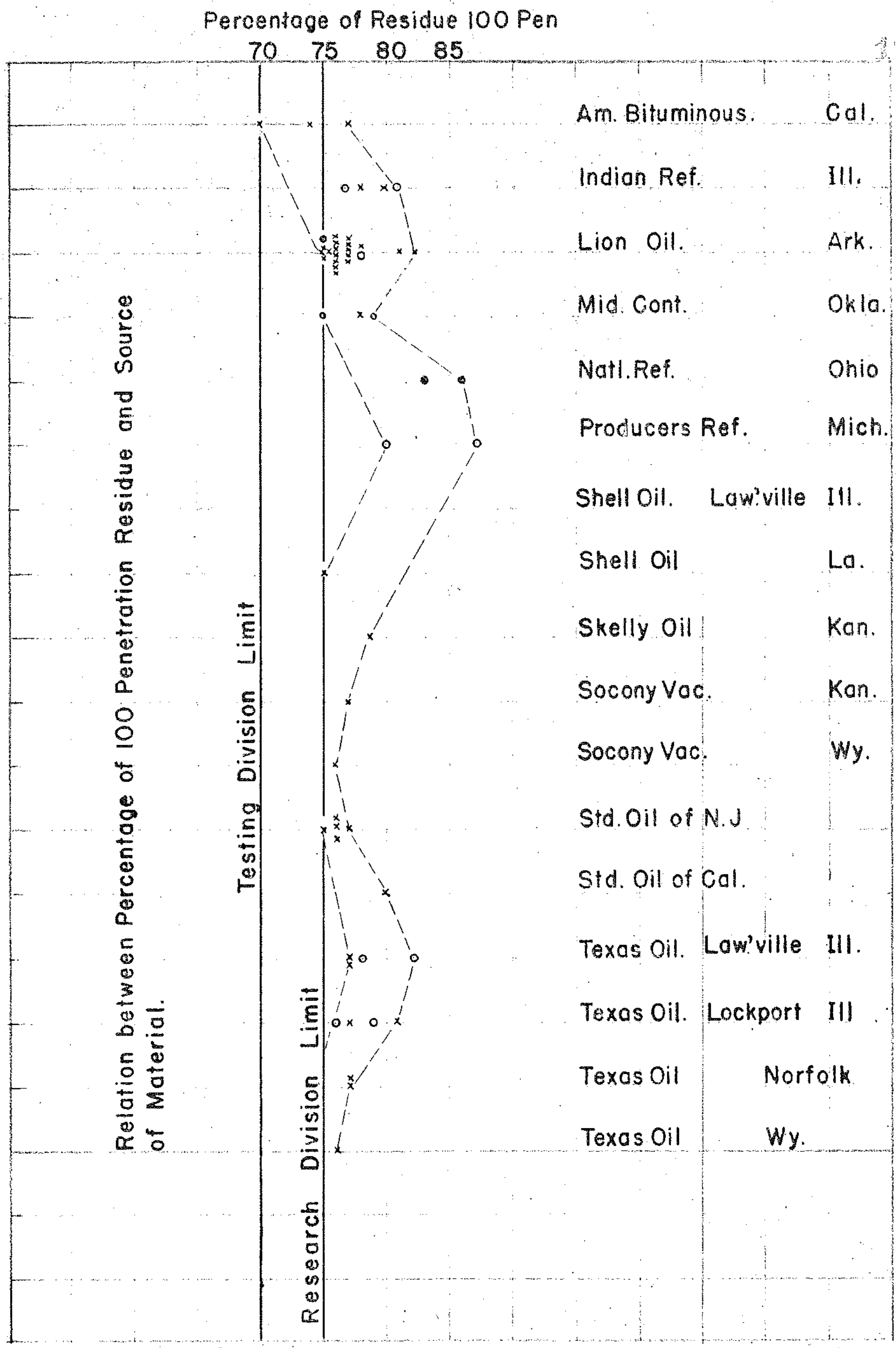

Percentage of Residue 100 Pen
70
75
80
85
Am. Bituminous. Cal.
Indian Ref. Ill.
Lion Oil. Ark.
Mid. Cont. Okla.
Natl. Ref. Ohio
Producers Ref. Mich.
Shell Oil. Law'ville Ill.
Shell Oil La.
Skelly Oil Kan.
Socony Vac. Kan.
Socony Vac. Wy.
Std. Oil of N.J
Std. Oil of Cal.
Texas Oil. Law'ville Ill.
Texas Oil. Lockport Ill
Texas Oil Norfolk
Texas Oil Wy.
Testing Division Limit
Research Division Limit
Relation between Percentage of 100 Penetration Residue and Source of Material.

Figure 12

The present specification requires 70 percent plus 100 penetration residue.

We believe that a change from 70 percent plus to 75 percent plus 100 penetration residue would aid to a certain degree in insuring a uniform product and would not place a hardship on any manufacturer.

Ash Content:

A new requirement, the Ash Content Test, is proposed as a method to detect the presence of fillers or native asphalts which have been introduced into the parent material for the purpose of controlling viscosity. It is recommended that a limit of 0.15 percent maximum be required in the new specification.

Emulsion Test:

The Emulsion Test is another new requirement proposed to eliminate certain bituminous materials which tend to emulsify on contact with water during the mixing operations. It is recommended that the bituminous material must pass the Emulsion Test before acceptance.

Water and Sediment:

It is recommended that the water and sediment requirement be reduced from 0.5 minus to zero. This is consistent with the Asphalt Institute recommended specifications for such materials of May 14, 1940.

SUMMARY

It is believed that the proposed specification for a "Slow Curing Liquid Asphalt", as summarized in table 8 and the test limits as described herein, will improve the uniformity of the product and reduce materially the abnormal hardening of the bitumen during construction and after placement in the road surface. The proposed bituminous material has been designated as a "Slow Curing Liquid Asphalt" in order that it will not be confused with the ordinary SC grade materials.

Table No. 8

PROPOSED SPECIFICATION
FOR
SLOW CURING LIQUID ASPHALT

	1940 MSHD Spec.	Testing	Research
Water and Sediment	0.5-	None	None
Flash Point, open cup °C	93.3+	175.0+	155+
Loss on Heating, 163°C, 50 g., 5 hr.	6- %	3.0-%	3.5-%
Loss on Heating Residue Viscosity at 60°C	Not required	Not required	Not more than 100% of original viscosity
Viscosity, Saybolt Furol, Sec., 60°C	350-600	500-600	450-600
Solubility in CCl_4	99.5	99.5	99.5
Ash, content	Not required	0.15%	0.15%
Oliensis Spot Test	Negative	Negative	Negative
Emulsion Test	Not required	Pass	Pass
Residue of 100 Penetration			
Residue percent	70+	70+	75+
Ductility at 25°C, cm.	100+	100+	100+
Ductility at 4°C., cm.	7+	Delete by request of P.R.A.	Not required