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# **NATIONAL BUREAU OF STANDARDS REPORT**

6256

A RAPID METHOD FOR PREDICTING THE DURABILITY  
OF COATING-GRADE ROOFING ASPHALTS

by

John P. Falzone

and

George D. McDonald



**U. S. DEPARTMENT OF COMMERCE**  
**NATIONAL BUREAU OF STANDARDS**

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# NATIONAL BUREAU OF STANDARDS REPORT

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and  
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## ABSTRACT

Twelve coating-grade asphalts, representative of those commercially available in the manufacture of roofing were studied to determine whether the rates of filtration, changes in constituents or losses in weight could be correlated with their durability.

Rates of filtration determined by the method described distinguished between the better and the poorer weatherers, but a quantitative correlation was poor although the method proved simple and reproducible.

Average rate of changes of total oils, dark oils, asphaltenes or asphaltenes plus loss in weight showed excellent correlations, but were inapplicable to early predictions of durabilities since their rates of change were not comparable throughout the exposure cycle.

Average rate of loss in weight was related to durability. In addition, the weight change for each asphalt was nearly linear with time and was comparable in early periods of exposure. The loss at 300 hours is consequently recommended as a rapid, reliable criterion for predicting the durability of a blown, coating-grade asphalt.

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## 1. INTRODUCTION

This investigation is part of the long established asphalt research project jointly sponsored by the National Bureau of Standards and the Asphalt Roofing Industry Bureau.

It has been known for some time that other factors being equal, the durability of roll roofings or shingles depends upon the nature of the asphalt employed in its manufacture. In turn, the nature of the asphalt is principally a function of the source of the petroleum crude from which it is derived and, to a lesser extent, the processing used to convert the residuum into the coating-grade product having the necessary physical characteristics. Because of the relatively long life of even the poorest roofing, a rapid, reliable evaluation of the weathering properties of an asphalt has long been an objective in the research project. The inherent chemical and rheological complexity of an asphaltic system has impeded the attainment of a simple satisfactory solution to this problem.

Early investigators concentrated their efforts towards developing methods for accelerated weathering. Since the effects of light, heat and oxygen were recognized as factors contributing to the degradation of the coating (1, 2)<sup>1/</sup>, the carbon arc lamp was soon introduced to asphalt studies (3). Later developments led to the periodic use of a water spray. Subsequent work on the effects of current and voltage (4), exposure temperature (5), accelerated cycle used (6), film preparation (7), inspection procedure and end-point determinations (8, 9) ultimately led to some recommended practices in accelerated testing of bituminous products.

Unfortunately, the quantitative relationship between accelerated and natural weathering is as yet not satisfactorily resolved. However, there is sufficient evidence to indicate that the relative order of failure for asphalts having significant differences in weather resistance is the same for both weatherometer and roof exposures (6).

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<sup>1/</sup> Figures in parentheses indicate literature references at the end of this report.



The purpose of this investigation was to evaluate several proposed criteria for predicting durability employing accelerated tests as primary standards, namely:

- (1) The filtration rate of n-pentane solubles through the insolubles of an asphalt.
- (2) The rate of changes in components.
- (3) The rate of losses in weight.

## 2. EXPERIMENTAL PROCEDURES

### 2.1 Asphalts

Twelve samples, representative of commercially available asphalts, were submitted by several members of the Research Committee of the Asphalt Roofing Industry Bureau, four from California, four from Midcontinent, and three from Venezuelan crudes. An additional Midcontinent sample, blown in a laboratory still to a significantly lower softening point, was included. The lower softening point of this sample rendered difficult the determination of its durability.

### 2.2 Sampling

Enough material was taken from a 5-gallon sample and at least one inch below the surface so that every determination that was made for each asphalt was done on a portion poured from the same melt. This precaution was taken because of the recognized susceptibility of asphalts to both reversible and irreversible changes even at room temperatures. The surface of samples in storage for three years, for example, has been shown to change as much as 8% in pentane insoluble (asphaltene) content (10).

### 2.3 Softening Points

Two softening point determinations were made for each asphalt by the Ring and Ball method as prescribed in A.S.T.M. Procedure D36-26.



The first specimen was poured immediately prior to casting the films necessary for accelerated testing, the second immediately after. The results presented in Table 1 are averages of the two.

## 2.4 Penetrations

Penetrations at 32°, 77° and 115°F were determined for each asphalt employing A.S.T.M. Procedure D5-49. The specimens for these determinations were poured immediately after half of the necessary films for determining durability were cast. The results are shown in Table 1. The susceptibility factor is defined as the ratio of the penetration at 115°F minus the penetration at 32°F to that at 77°F for a given asphalt.

## 2.5 Filtration Rate

In the primary separation of an asphalt into asphaltenes, white oils, dark oils and resins (11), n-pentane is normally added to a sample in order to precipitate the asphaltenes. Subsequent removal is effected by filtering through a tared Gooch crucible, the bottom of which is covered by an asbestos mat. During the separation of 21 asphalts (12), Kleinschmidt observed that he could generally predict the relative order of failure of these samples in an accelerated cycle by arranging them according to the time necessary for filtration, those taking the least time failing most rapidly.

A quantitative method to measure this filtration rate was developed with the aid of a pressure filter designed by the Johns-Manville Research Laboratories. The instrument was essentially an all metal cylinder having a fitted, removable stopcock assembly at the bottom so that a Whatman #50 paper filter disc could be inserted and a fitted cap having a lead-in tube so that a suitable inert gas could be introduced for increasing the pressure. The latter was regulated by a simple manostat in series with a manometer.



The general procedure employed was as follows:

Ten ml. of a benzene solution having the desired weight concentration of asphalt was pipetted into 90 ml. of n-pentane previously measured into a suitable glass bottle. After swirling, the bottle was stoppered and the system was allowed to stand for 20 hours at room temperature.

The mixture was again swirled. Timed with a stopwatch, the ensuing 90 seconds were used to transfer the sample into the pressure filter tube, fitting the cap into place and elevating the pressure to one p.s.i. using nitrogen gas for this purpose.

The stopcock was opened allowing the filtrate to flow into a suitable graduated cylinder. The times required for successive 5 ml. volumes to flow were recorded.

The filtration rate was obtained as follows:

The flow rate was found to follow the expression  $V^2 = KT$  fairly closely, where  $V$  = volume of filtrate in ml. and  $T$  = time in minutes. Taking the logarithm of both sides and rearrangement of the result yielded

$$\ln V = 1/2 \ln T + 1/2 \ln K.$$

Consequently, volume was plotted against time on logarithmic graph paper. A line having the slope =  $1/2$  was fitted to the experimental points and extrapolated to  $T = 1$ . The value of  $V$  thus obtained, when squared, evaluated  $K$  according to the above expression.

Two values of  $K$  were obtained for each asphalt. The first was derived employing a 10% solution of each asphalt in benzene. A second filtration rate, designated  $K_s$ , was determined in which the amount of asphaltenes precipitated was calculated to be the same for all samples. The level arbitrarily chosen was that amount of asphaltenes obtained when an aliquot of the 10% benzene solution of the asphalt having the lowest initial asphaltene concentration was used.



## 2.6 Durability by Accelerated Weathering

Since the primary standard employed in this study was the weatherometer test, every reasonable precaution was taken to obtain reliable ratings. Five replicate specimens of each asphalt were cast in uniform films of 25 mils  $\pm 1$  mil on 6- x 2-3/4-in. aluminum panels (7). Essentially these were exposed to accelerated conditions as proposed by A.S.T.M. Method D529-39T. To offset possible differences in the carbon arc machines, two specimens of each asphalt were exposed in one weatherometer and the remaining three in another.

The 51-9C cycle, consisting of 51 minutes of dry light and heat per hour followed by 9 minutes of light and cold (40°F.) water spray, was employed.

A maximum panel temperature, 144°  $\pm 2^\circ$ F., was attained in each machine. The variation in coating temperatures during each cycle was comparable.

Failure determinations were made by obtaining spark photographs using the high voltage crack detector technique (8, 9) The coating was removed as a final failure when the photograph showed that at least 50% of the specimen's area had cracks.

The specimens were inspected regularly twice each week, more frequently when failure appeared to be imminent. The inspections were standardized as follows:

At the end of 51 minutes of dry light and heat, the specimens were removed, permitted to cool to room temperature and inspected preliminarily. Those coatings which showed pronounced sparking were put aside for further evaluation. Twenty minutes after they were removed from the machine, spark photographs of these specimens were made. The failed coatings were removed and the time to failure was recorded.



## 2.7 Rate of Change of Components

A primary separation of the asphalts was obtained by chromatographic analysis (11). This consisted of filtering off the n-pentane insolubles (asphaltenes), and further separating the solubles (maltenes) into white oils, dark oils and resins by absorbing them on activated Fuller's earth in a chromatographic column and eluting with increasingly polar solvents, namely, n-pentane, methylene chloride and methyl ethyl ketone.

Two modifications of the procedure cited were made:

- (1) The Fuller's earth was activated at  $140^{\circ} \pm 5^{\circ}\text{C}$ . for 18-20 hours.
- (2) A volume of 250 ml. of each solvent was employed to obtain successive fractions.

An individual specimen of each asphalt was fractionated before exposure, at 200 hours, at a second estimated intermediate point and at final failure. Thus, changes in every component with time could be followed. The average rate of change of each desired constituent was obtained by dividing the total change by the time of exposure.

## 2.8 Loss in Weight

Data for this study were obtained by weighing the replicate specimens of each asphalt at approximately 200-hr. intervals and computing their average. Since this is a surface phenomenon, actual losses in weight and not percentages were compared since the surface area of each specimen was the same. Similar to the constituent change study, loss in weight for each asphalt was plotted against time of exposure. Average rate of loss in weight was computed by dividing the total loss by its durability.



### 3. RESULTS AND DISCUSSION

#### 3.1 Rate of Filtration

The softening points, penetrations, rates of filtration at two concentrations and the durabilities for the 12 asphalts are presented in Table 1. For ease of comparison the listing appears in the order of increasing durability. It is immediately evident that, except for a general trend, a good correlation does not exist between the rate of filtration at either concentration and the durability of the asphalt. It should be borne in mind that the conditions for this determination were selected on the basis of a limited study of the effects of varying the concentration of the benzene solution of the asphalt and settling period of the precipitate after transfer to the pressure filter. They constitute a compromise designed to minimize erratic results and compression of differences among the asphalts induced by lower concentrations while at the same time offsetting inordinate running times and similar compressive effects brought about by longer settling periods. It is conceivable that an extended study of these variables would indicate other conditions to be more favorable for the measurement of this property. The method is simple while the reproducibility of duplicate determinations is  $\pm 3\%$ .

However, changes in constituents and losses in weight studies were favored because of the promising nature of the results obtained.

#### 3.2 Changes in Components

The fractions obtained with the separation employed were asphaltenes, while oils, dark oils and resins. As each asphalt degraded during exposure, the overall pattern was quite similar; asphaltenes and resins increased and losses in weight became evident as the dark and white oils were depleted. Thus, it might be expected that the rate of change of one or more of these fractions would be related to durability. A previous report on 20 asphalts had



TABLE 1.

Asphalt	S. P. R & B °F	Penetration, $\frac{\text{mm}}{10}$			Susc. Factor	K	K <sub>s</sub>	Av. Durab. (days)
		115°F	32°F	77°F				
Ambit II (Cat.)	232	34	14	17	1.18	49.0	57.7	27
Shell	218	33	14	18	1.06	60.0	54.8	30
Ambit I	227	34	12	18	1.22	51.8	54.0	35
Union	241	32	13	19	1.00	74.8	75.6	36
Talco	224	35	15	21	0.95	39.1	49.0	38
Lag. I	233	27	12	17	0.88	44.2	47.6	39
Tia Juana	223	35	15	21	0.95	33.6	31.9	42
Lag. II	222	32	12	19	1.05	44.2	34.8	50
Socony	222	32	12	21	0.95	35.4	38.4	59
Kansas	223	33	19	23	0.61	39.1	37.2	72
Louisiana	207	34	17	21	0.81	43.5	43.5	90
Oklahoma	220	36	18	23	0.78	47.6	29.6	95



shown a good correlation to exist between accelerated weathering and (1) the average rate of change of asphaltenes, (2) the average of rate of change of total oils (white plus dark oils), (3) the average rate of change of asphaltenes plus weight loss (10). These conclusions have been reaffirmed in the present study. Figures 1, 2 and 3 show the relationships. In addition, the average rate of change in dark oils also correlated well with weather resistance (Figure 4).

In order to determine whether these data could be translated into a practical, reliable method for predicting durability, it was necessary to establish whether the same relative order of change was maintained in each asphalt during the early periods of exposure. The data compiled for this purpose are shown in Table 2. Thus, the change in asphaltenes, white oils, dark oils, resins and weight loss of each asphalt was followed in the manner illustrated by Figures 5, 6 and 7 for three of the samples. A direct graphical comparison of all asphaltene component changes with time established that the same relative order of change was not maintained throughout the exposure (Figure 8). A similar study of total oil change during exposure for each asphalt likewise showed that an interchange of relative position took place which was different from that obtained by comparing the calculated average rates of change. Consequently, predictions based on early rates of change of these constituents could lead to serious discrepancies.

### 3.3 Loss in Weight

Losses in weight data were analyzed in an identical manner to those involving changes in components. Thus Figure 9 showed a good correlation with durability by accelerated weathering. Additionally, the loss for each asphalt during the early periods was nearly linear with time. Figure 10 indicates that the relative position assumed by each sample could be attributed to the length of time elapsed before it actually started to show a loss, i.e., its induction period, in addition to its difference in slope (rate). The loss in weight of each asphalt after 300



AMBIT II - 27 Days

TABLE 2.

% Component	Asphaltene	White Oils	Dark Oils	Resins	Cleanup	Wt. Loss	Recovery
0 Hrs.	41.3	19.8	30.2	9.0	0.1	0	100.4
220 Hrs.	49.8	16.1	19.6	10.3	0.1	3.7	99.6
458 Hrs.	52.2	13.5	16.4	9.1	0.0	8.1	99.3
585 Hrs.	51.4	13.0	14.8	11.3	0.0	9.3	99.8
594 Hrs.*	52.1	12.5	14.6	11.3	---	9.4	99.9
% Change	10.8	7.3	15.6	2.3	---	9.4	---
% Change/Day	.400	.270	.579	---	---	.344	---

SHELL - 30 Days

% Component	Asphaltene	White Oils	Dark Oils	Resins	Cleanup	Wt. Loss	Recovery
0 Hrs.	39.7	25.7	26.0	8.1	0.2	0	99.7
199 Hrs.	47.2	22.6	17.4	9.6	0.0	2.7	99.7
570 Hrs.	48.2	20.2	13.9	9.8	0.0	8.1	100.2
657 Hrs.	48.6	18.2	13.9	9.9	0.1	9.0	99.7
660 Hrs.*	48.6	18.4	13.7	9.8	---	9.0	99.5
% Change	8.9	7.3	12.3	1.7	---	9.0	---
% Change/Day	.297	.243	.410	---	---	.300	---

AMBIT I - 35 Days

% Component	Asphaltene	White Oils	Dark Oils	Resins	Cleanup	Wt. Loss	Recovery
0 Hrs.	41.2	21.7	27.3	9.0	0.1	0	99.3
220 Hrs.	48.6	19.0	18.7	10.9	0.1	2.8	100.1
458 Hrs.	49.7	16.1	15.9	11.1	0.0	7.1	99.9
751 Hrs.	50.3	13.4	14.0	11.2	0.1	10.5	99.5
770 Hrs.*	50.2	13.2	14.0	11.3	---	10.8	---
% Change	9.0	8.5	13.3	2.3	---	10.8	---
% Change/Day	.257	.242	.380	---	---	.309	---

\*Composition at final failure; obtained from graphical analysis.



TABLE 2. (CONTINUED) - 2

UNION - 36 Days									
% Component	Asphaltene	White Oils	Dark Oils	Resins	Cleanup	Wt. Loss	Recovery		
0 Hrs.	40.8	24.1	26.2	8.3	0.1	0	99.5		
199 Hrs.	46.9	19.9	19.5	11.0	0.1	2.8	100.2		
572 Hrs.	48.0	17.3	15.1	10.8	0.1	8.9	100.2		
725 Hrs.	48.8	14.9	14.6	10.6	0.1	10.7	99.7		
792 Hrs.*	49.6	14.7	13.6	10.5	---	11.6	100.0		
% Change	8.8	9.4	12.6	2.2	---	11.6	---		
% Change/Day	.244	.261	.350	---	---	.322	---		
TALCO - 38 Days									
% Component	Asphaltene	White Oils	Dark Oils	Resins	Cleanup	Wt. Loss	Recovery		
0 Hrs.	41.4	21.8	30.8	5.5	0.4	0	99.8		
200 Hrs.	47.4	17.8	23.3	8.2	0.2	2.4	99.3		
471 Hrs.	47.5	17.9	19.2	7.9	0.2	6.6	99.3		
875 Hrs.	48.0	14.9	17.9	7.6	0.1	11.4	99.9		
836 Hrs.*	47.9	15.1	18.2	7.5	---	11.1	99.8		
% Change	6.5	6.7	12.6	2.0	---	11.1	---		
% Change/Day	.171	.176	.332	---	---	.292	---		
LAGUNILLAS I - 39 Days									
% Component	Asphaltene	White Oils	Dark Oils	Resins	Cleanup	Wt. Loss	Recovery		
0 Hrs.	39.4	24.7	28.0	7.2	0.3	0	99.6		
199 Hrs.	42.8	24.0	22.1	9.2	0.0	1.9	100.0		
745 Hrs.	45.2	19.5	16.3	10.2	0.2	8.4	99.8		
769 Hrs.	45.5	20.1	15.9	9.8	0.1	8.7	100.1		
854 Hrs.*	45.6	18.6	15.2	9.8	---	9.7	98.9		
% Change	6.2	6.1	12.8	2.6	---	9.7	---		
% Change/Day	.159	.157	.328	---	---	.249	---		

\*Composition at final failure; obtained from graphical analysis.



TABLE 2. (CONTINUED) - 3

## TIA JUANA - 42 Days

% Component	Asphaltene	White Oils	Dark Oils	Resins	Cleanup	Wt. Loss	Recovery
0 Hrs.	38.1	23.7	30.8	7.1	0.4	0	100.1
202 Hrs.	44.3	19.8	22.1	9.1	0.4	1.9	97.6
666 Hrs.	45.1	16.4	20.7	9.1	0.3	8.2	99.6
859 Hrs.	44.5	17.4	17.6	10.3	0.2	9.7	99.7
924 Hrs.*	44.8	16.4	18.4	10.1	---	10.0	99.7
% Change	6.7	7.3	12.4	3.0	---	10.0	---
% Change/Day	.160	.174	.296	---	---	.238	---

## LAGUNILLAS II - 50 Days

% Component	Asphaltene	White Oils	Dark Oils	Resins	Cleanup	Wt. Loss	Recovery
0 Hrs.	37.0	19.7	32.6	8.6	0.7	0	98.6
200 Hrs.	41.8	21.0	25.0	10.0	0.5	1.3	99.6
742 Hrs.	44.2	16.2	19.0	11.2	0.3	8.6	99.5
1039 Hrs.	44.9	15.0	17.8	11.3	0.4	10.1	99.5
1100 Hrs.*	44.8	15.0	17.5	11.5	---	10.5	99.3
% Change	7.8	4.7	15.1	2.9	---	10.5	---
% Change/Day	.156	.094	.302	---	---	.210	---

## SOCONY - 59 Days

% Component	Asphaltene	White Oils	Dark Oils	Resins	Cleanup	Wt. Loss	Recovery
0 Hrs.	35.9	22.0	31.4	8.5	0.8	0	98.6
200 Hrs.	41.8	22.8	22.6	10.7	0.6	0.6	99.1
670 Hrs.	43.6	19.6	21.3	9.7	0.7	4.9	99.8
1356 Hrs.	43.4	15.5	18.2	10.8	0.5	11.0	99.4
1298 Hrs.*	43.0	15.8	18.0	11.3	---	10.3	98.4
% Change	7.1	6.2	13.4	2.8	---	10.3	---
% Change/Day	.120	.105	.227	---	---	.175	---

\*Composition at final failure; obtained from graphical analysis.



TABLE 2. (CONTINUED) - 4

## KANSAS - 72 Days

% Component	Asphaltene	White Oils	Dark Oils	Resins	Cleanup	Wt. Loss	Recovery
0 Hrs.	35.2	25.9	28.7	8.3	0.4	0	98.5
184 Hrs.	41.6	22.7	23.9	10.4	0.5	0.3	99.4
911 Hrs.	43.3	20.1	19.7	8.8	0.2	7.2	99.3
1674 Hrs.	43.0	16.3	15.8	9.5	0.4	14.2	99.2
1584 Hrs.*	43.4	16.7	16.0	9.7	---	13.4	99.2
% Change	7.8	9.2	12.7	1.4	---	14.2	---
% Change/Day	.108	.128	.176	---	---	.186	---

## LOUISIANA - 90 Days

% Component	Asphaltene	White Oils	Dark Oils	Resins	Cleanup	Wt. Loss	Recovery
0 Hrs.	33.6	25.6	32.2	7.2	0.6	0	99.2
202 Hrs.	39.2	23.8	26.4	8.5	0.7	0.0	98.6
666 Hrs.	40.4	21.5	23.9	8.8	0.6	3.8	99.7
2071 Hrs.	39.5	16.8	18.2	10.4	0.4	14.7	100.0
1980 Hrs.*	40.1	17.2	18.5	9.5	---	13.8	99.1
% Change	6.5	8.4	13.7	2.3	---	13.8	---
% Change/Day	.072	.093	.152	---	---	.153	---

## OKLAHOMA - 95 Days

% Component	Asphaltene	White Oils	Dark Oils	Resins	Cleanup	Wt. Loss	Recovery
0 Hrs.	35.8	24.4	29.7	8.0	0.9	0	98.8
204 Hrs.	39.5	23.6	24.4	9.2	0.8	0.2	97.7
1072 Hrs.	41.6	18.2	22.1	8.3	0.7	7.4	98.3
1946 Hrs.	41.3	14.8	16.8	11.0	0.7	14.9	99.5
2090 Hrs.*	41.6	14.3	16.2	10.9	---	16.0	99.0
% Change	5.8	10.1	13.5	2.9	---	16.0	---
% Change/Day	.061	.106	.142	---	---	.169	---

\*Composition at final failure; obtained from graphical analysis.



hours of weathering was obtained from this graph and plotted against its durability. The result is shown in Figure 11 which may now be employed as a calibration curve to predict durabilities of other asphalts if the loss at 300 hours is known.

This development is entirely consistent with the facts known about the general degradative pattern in asphalt and to a limited extent elucidates the role of oils and asphaltenes with respect to durability. Specifically, asphalt degrades under the influence of heat and ultra-violet light, the former leading predominantly to an increase in asphaltenes, the latter to oxygenated, water extractable and volatile degradation products. Both of these general reactions result in the depletion of the total oils. The data in Table 1 show that the asphaltene increase had either ceased or tapered to a nominal value well before the coating had weathered to final failure. Evidently, then, the controlling reaction was the light-induced one which continued to deplete the available oils to a level sufficiently low so that the cracked coating could no longer heal itself under thermal influence. Hence, the faster the rate of this reaction, the more rapidly did the coating attain final failure. It appears that the weight loss obtained after 200-300 hours of exposure was an effective measure of the relative rates of this reaction for the asphalts studied.

Those asphalts having the greater durability had the longer induction period, the slower rate of loss in weight and lost weight linearly throughout the exposure cycle. The less durable asphalts had extremely short induction periods, the greater rate of loss in weight and evidenced more deviation from linear loss during the determination of their durability. The latter effect was apparently a result of such a rapid depletion of the reactants that the amount made available by the coating for continuation of this surface phenomena diminished with time.



The length of the induction period was not determined quantitatively for each asphalt. However, one of the less durable asphalts required only forty hours of exposure before it lost weight while a more durable coating required 160 hours under the same conditions. Further work on this phase should prove of great interest.

#### 4. CONCLUSIONS

The rate of filtration as determined under the conditions described in this report did not correlate strictly with durability although it did distinguish between the less durable and the more durable asphalts.

The average rate of change of total oils or dark oils or asphaltenes correlated well with accelerated durability. However, these constituent changes could not be employed as criteria for prediction of durability because they did not change comparably in every asphalt during exposure.

The average rate of loss in weight correlated well with durability by accelerated weathering. Moreover, the loss in weight was nearly linear with time during the first several hundred hours of exposure. Determination of the loss in weight at 300 hours appears to afford a simple, reliable method for the prediction of durability of a coating grade asphalt.

An induction period, during which no loss in weight occurs, apparently furnishes a means for determining the relative positions of the loss in weight curves for each asphalt in a series. Further study of this phenomenon is indicated.



## 5. REFERENCES

- (1) V. Vojtech, "Light Action on Asphalts", J. Soc. Chem. Ind., 654 (1906).
- (2) Errera, J., "Light Sensitivity of Asphalts", Trans. Farad. Soc., 314-17 (1923).
- (3) Strieter, O. G., "Accelerated Tests of Asphalt", J. Res. NBS 5, 247-253 (Aug. 1930), RP197.
- (4) Kolb, W. C., "The Characteristics of the Carbon Arc. How the Properties Vary with Voltage and Current", Electrical Engineering 53, 1173-9 (1934), 56, 319-24 (1937).
- (5) Weetman, B., "Accelerated Weathering of Bituminous Materials - Operating Variables", Proc. A.S.T.M. 43, 1154-64 (1943).
- (6) Kleinschmidt, L. R. and Greenfield, S. H., "Influence of Exposure Conditions on the Accelerated Durability Testing of Asphalt", A.S.T.M. Bulletin No. 213 (April 1956).
- (7) Greenfield, S. H., "A Method of Preparing Uniform Films of Bituminous Materials", A.S.T.M. Bulletin No. 193, 50-2 (Oct. 1953).
- (8) "The Design and Application of a Spark-Gap Instrument for Detecting Crack Failures of Asphalt Coatings During Weathering Tests", (issued as A.S.T.M. STP No. 94) Symp. on Accelerated Durability Testing of Bituminous Materials, American Society for Testing Materials, p. 153 (1949).
- (9) Hunter, J. B., Gzemski, F. C. and Laskaris, L., "A New Method for Evaluating Failure of Bituminous Materials Due to Weathering", Symp. on Acc. Durability Testing of Bit. Materials, American Society for Testing Materials, p. 144 (1949) (Issued as A.S.T.M. STP No. 94).

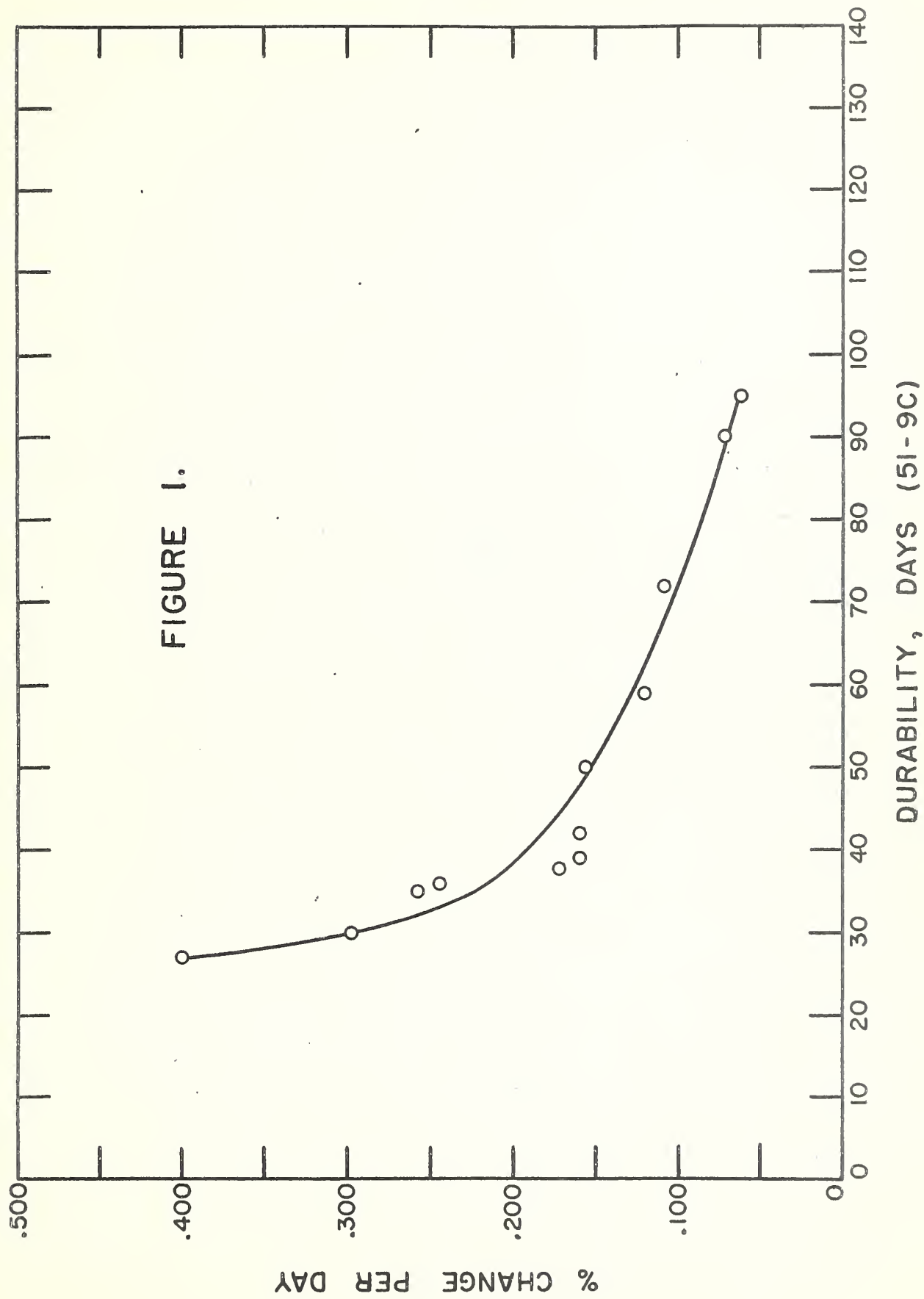


- (10) Falzone, J. P., "Relation of Rate of Component Changes in Asphalts to Accelerated Durability", N.B.S. Report No. 5935 (June 20, 1958).
- (11) Kleinschmidt, L. R., "Chromatographic Method for the Fractionation of Asphalts Into Distinctive Groups of Components", J. Res. NBS 54, 163-166 (1955).
- (12) Greenfield, S. H., "Report on the Evaluation of 21 Coating-Grade Roofing Asphalts", N.B.S. Report No. 4732 (July 1, 1956).



AVERAGE RATE OF ASPHALTENE CHANGE VS. ACCELERATED DURABILITY

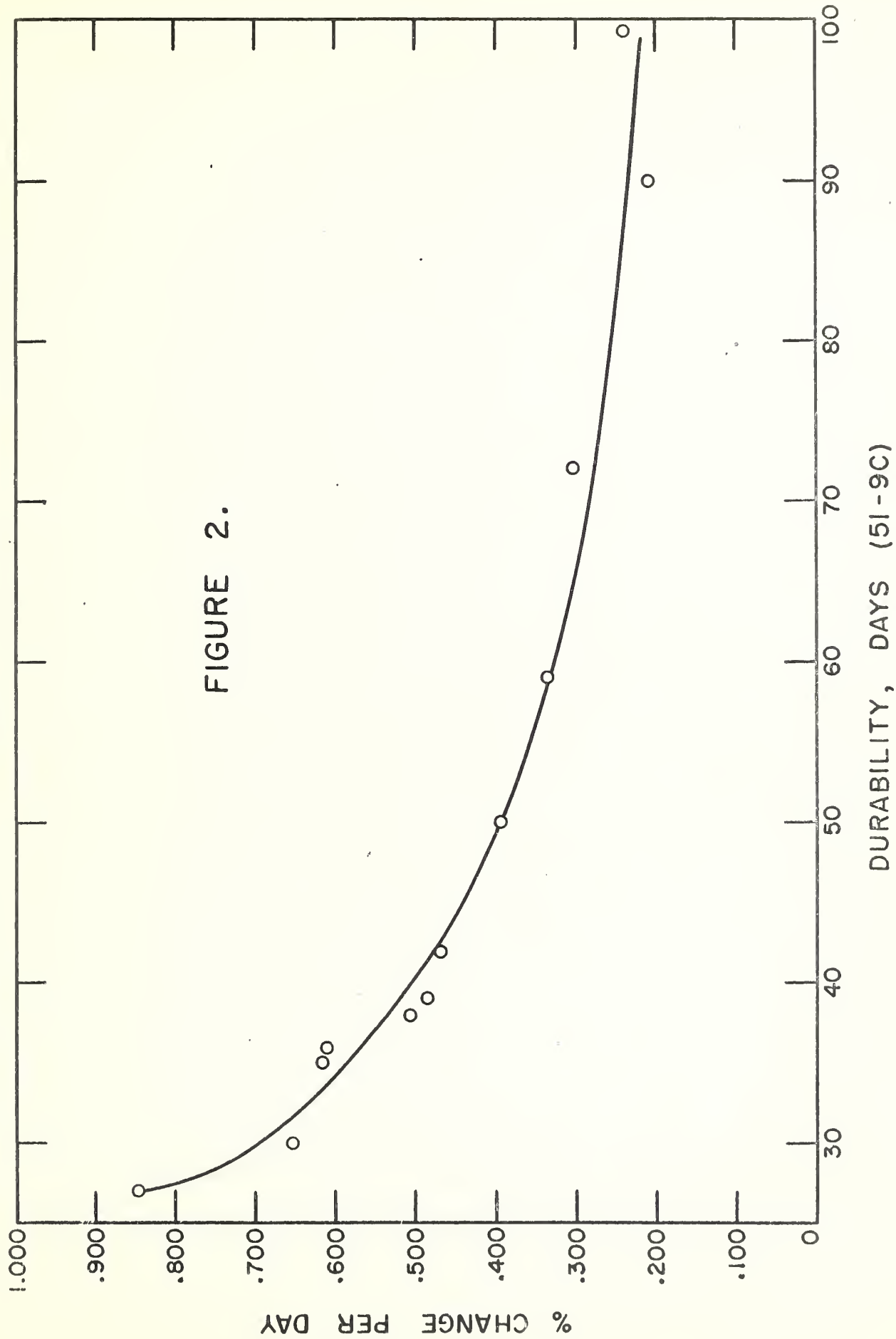
FIGURE 1.





AVERAGE RATE OF CHANGE OF TOTAL OILS VS. DURABILITY

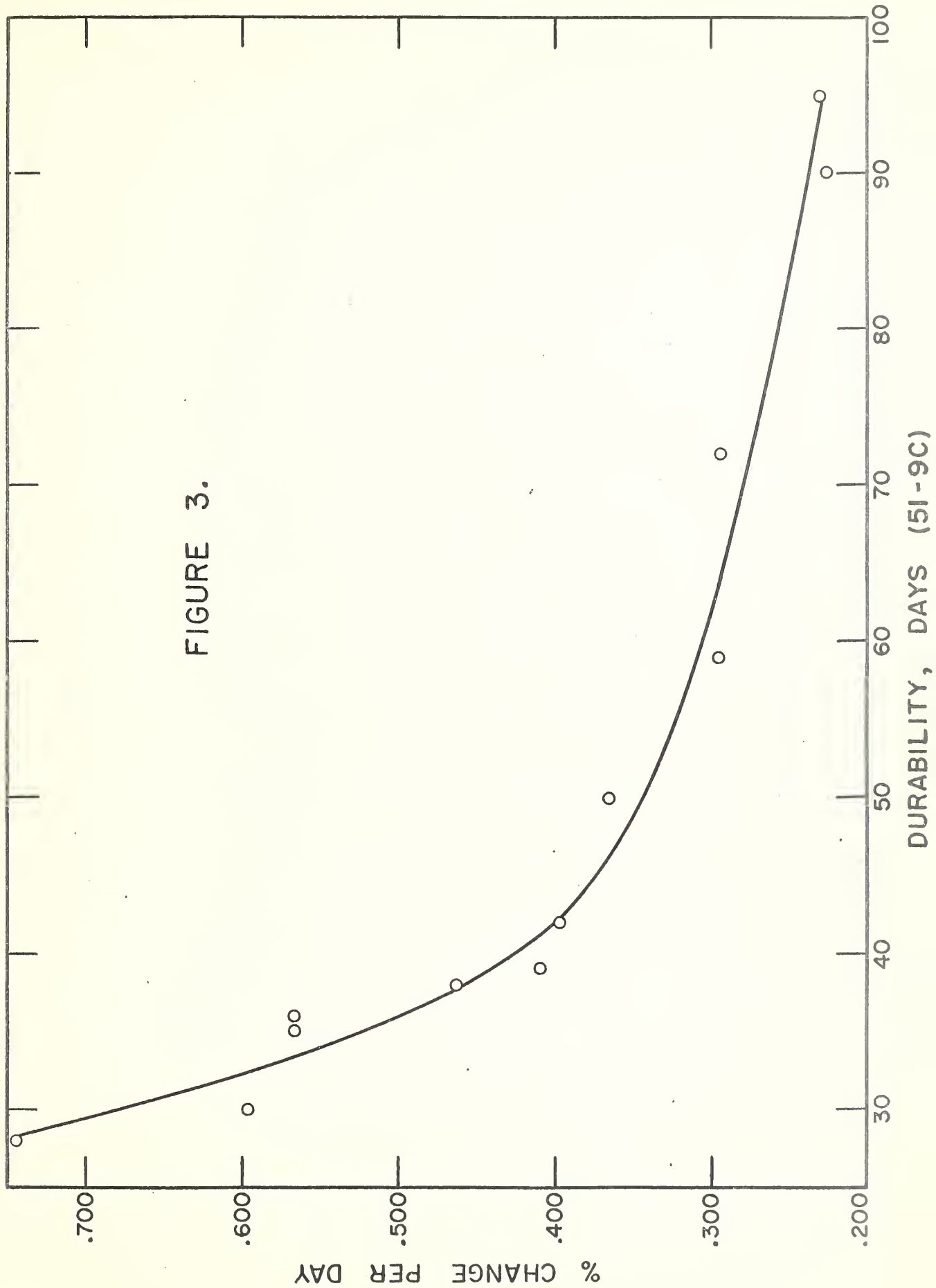
FIGURE 2.





AVERAGE RATE OF CHANGE OF ASPHALTENES + LOSS IN WEIGHT VS. DURABILITY

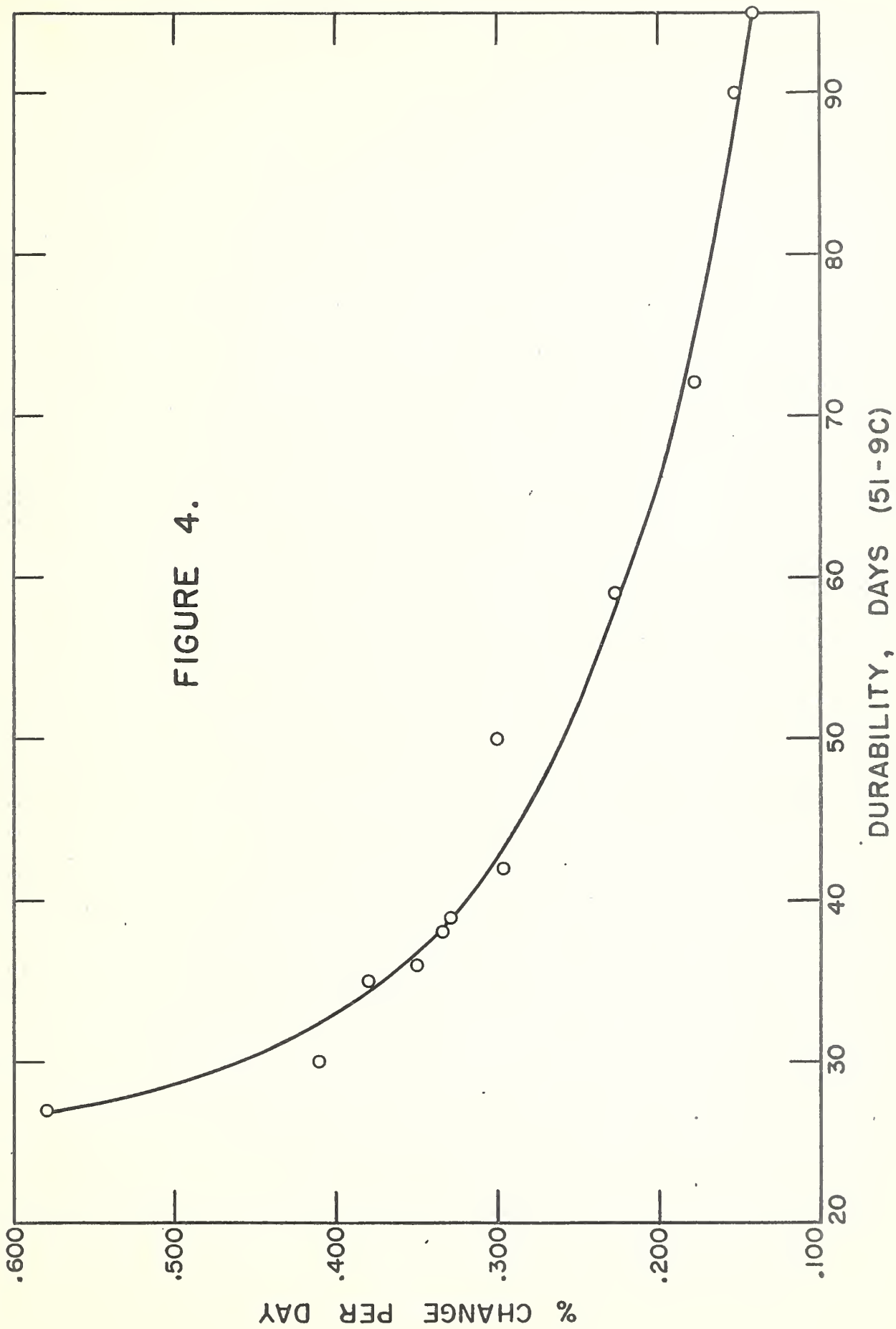
FIGURE 3.





AVERAGE RATE OF CHANGE OF DARK OILS VS. DURABILITY

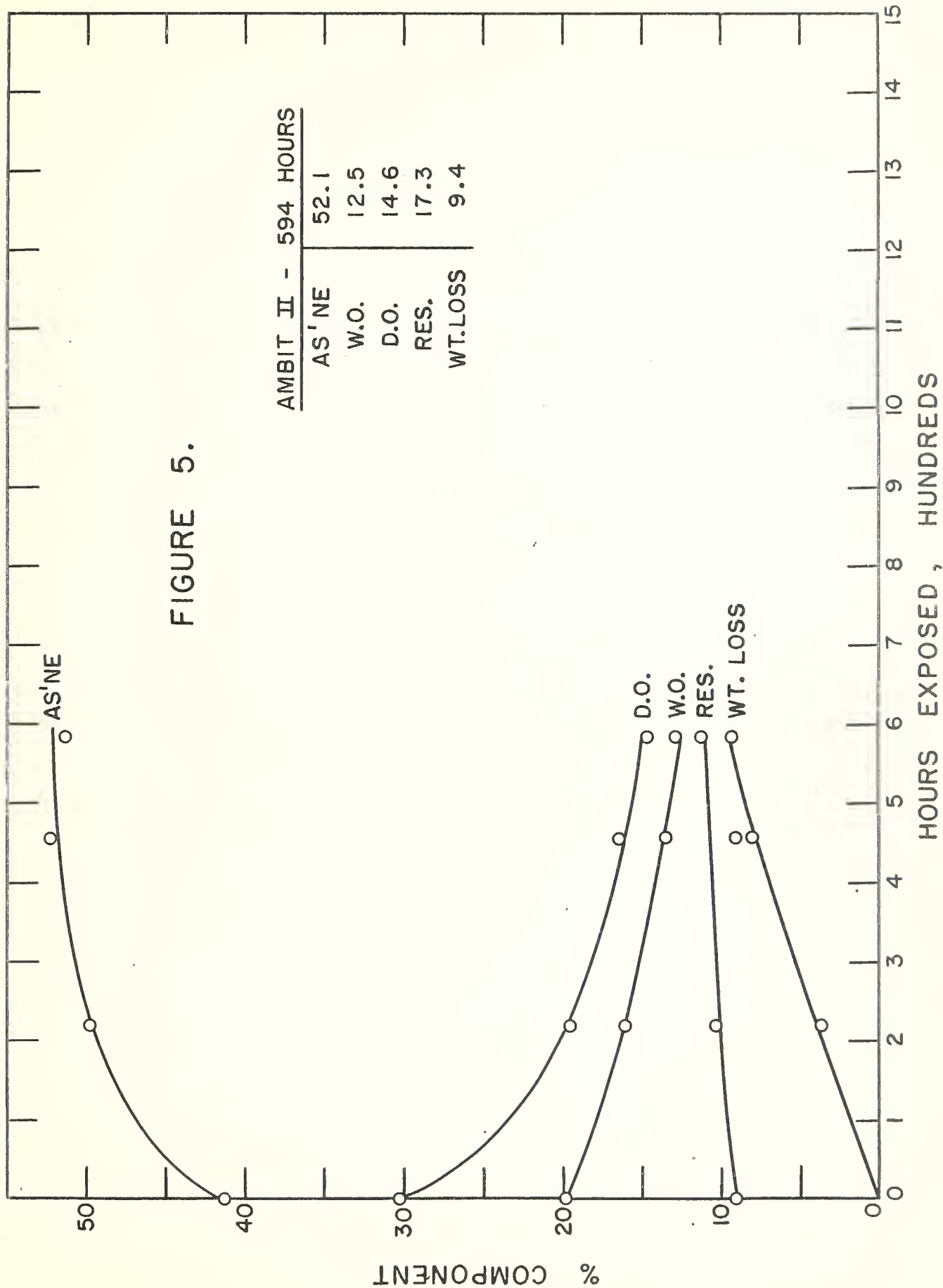
FIGURE 4.





# AMBIT II - AVERAGE COMPOSITION AT FINAL FAILURE

FIGURE 5.





# LAG I - AVERAGE COMPOSITION AT FINAL FAILURE

FIGURE 6.

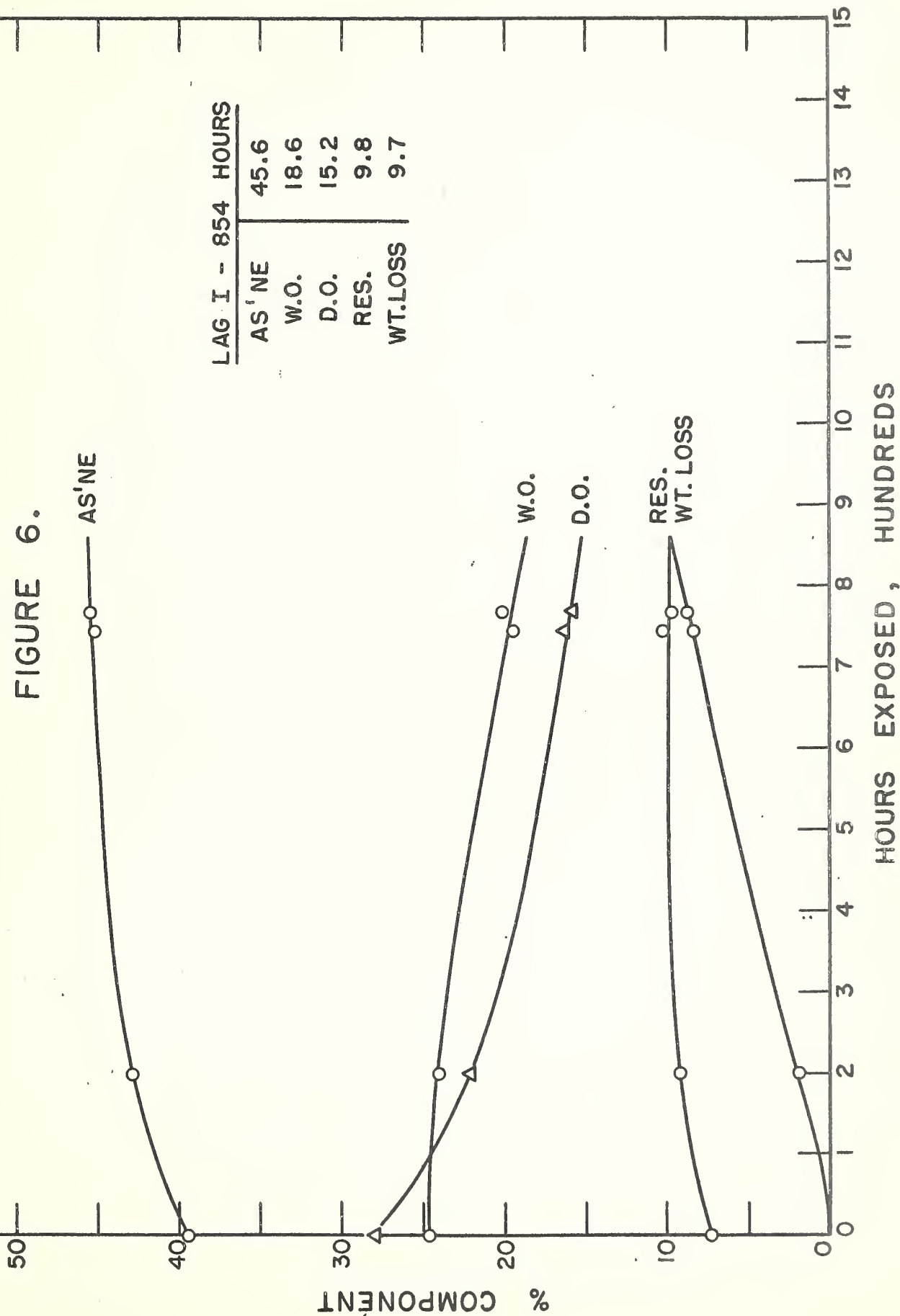
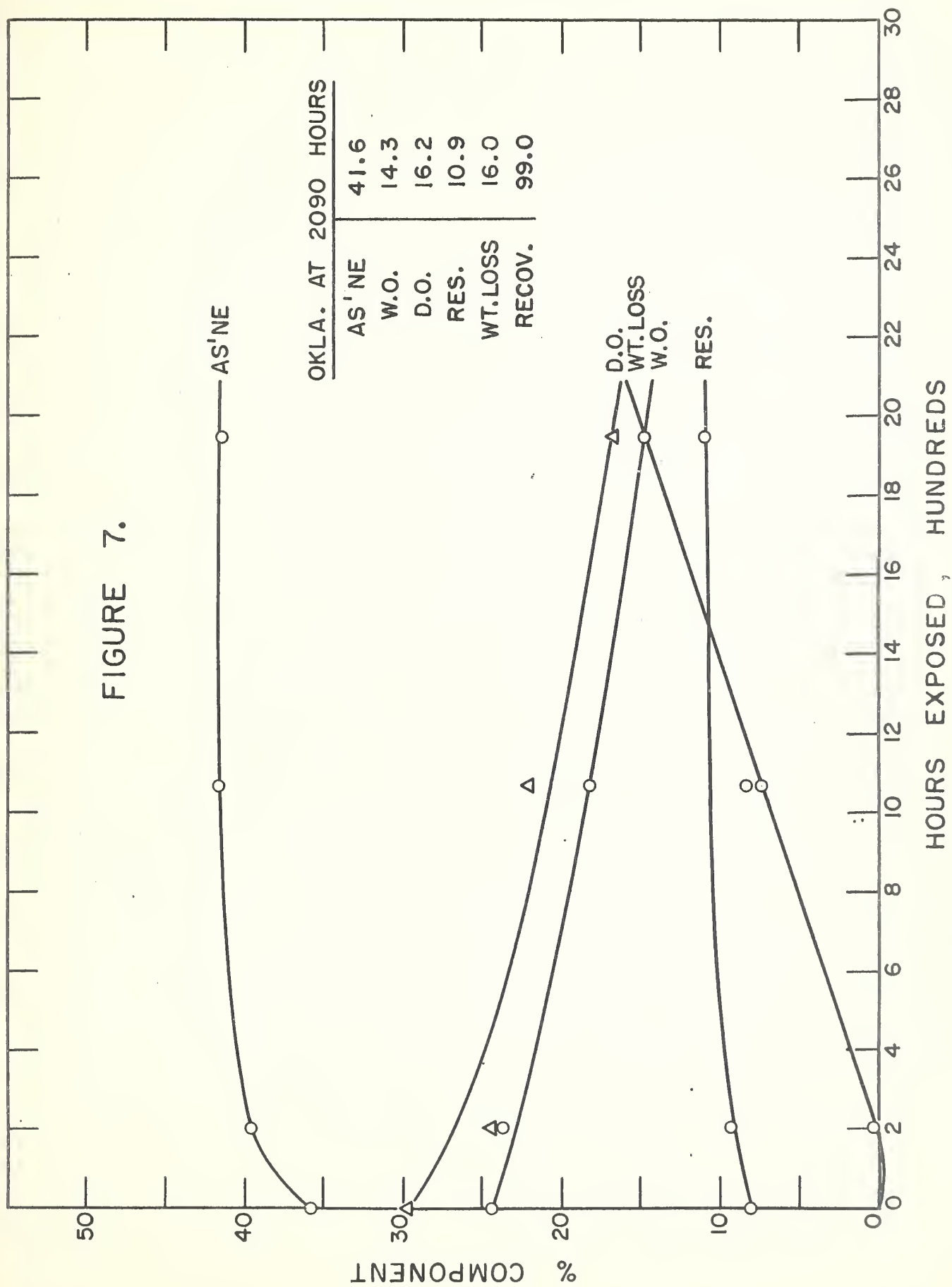


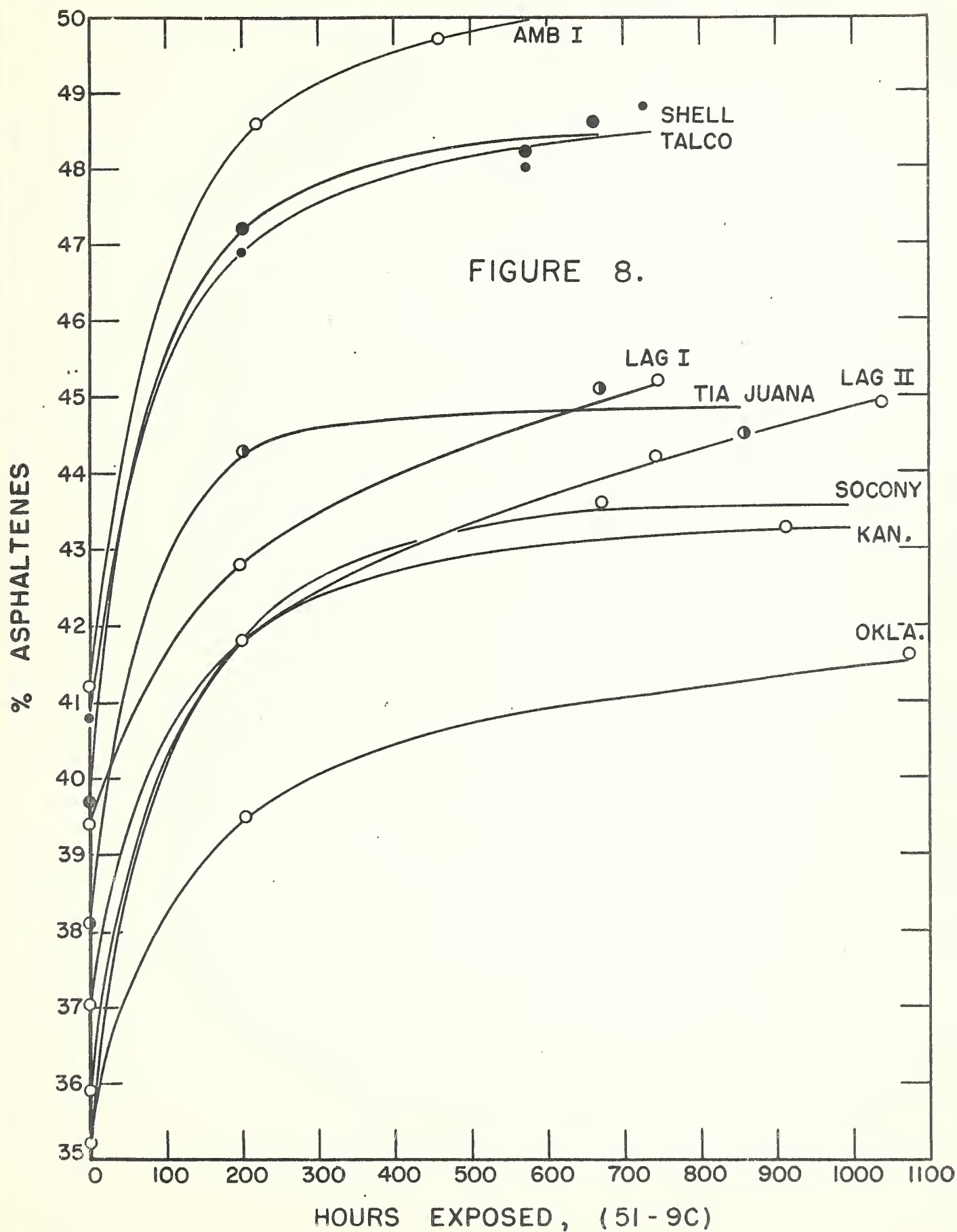


FIGURE 7.





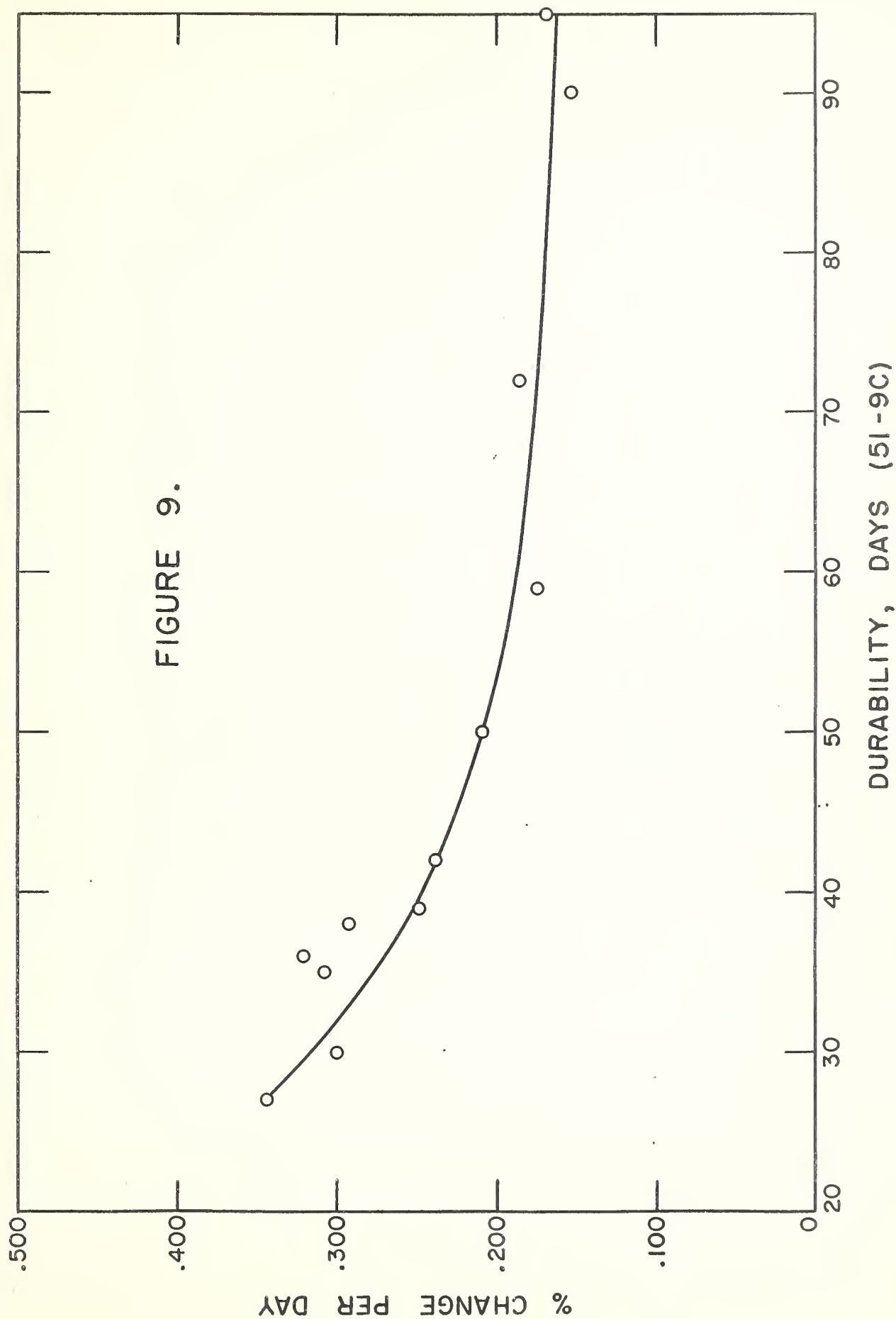
# % CHANGE IN ASPHALTENES DURING EXPOSURE





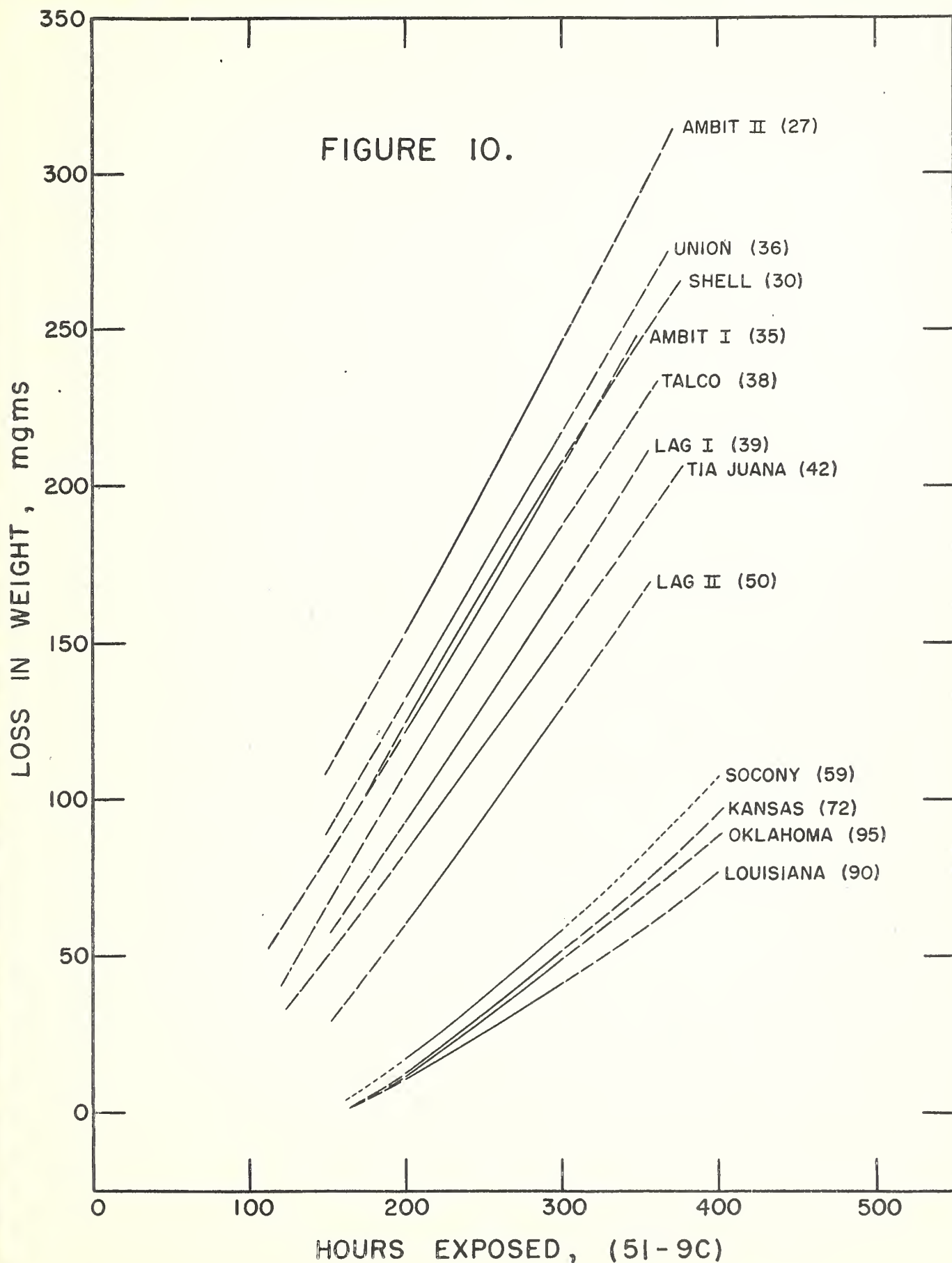
AVERAGE RATE OF LOSS IN WEIGHT VS. DURABILITY

FIGURE 9.





# LOSS IN WEIGHT BETWEEN 200-300 HOURS OF WEATHERING

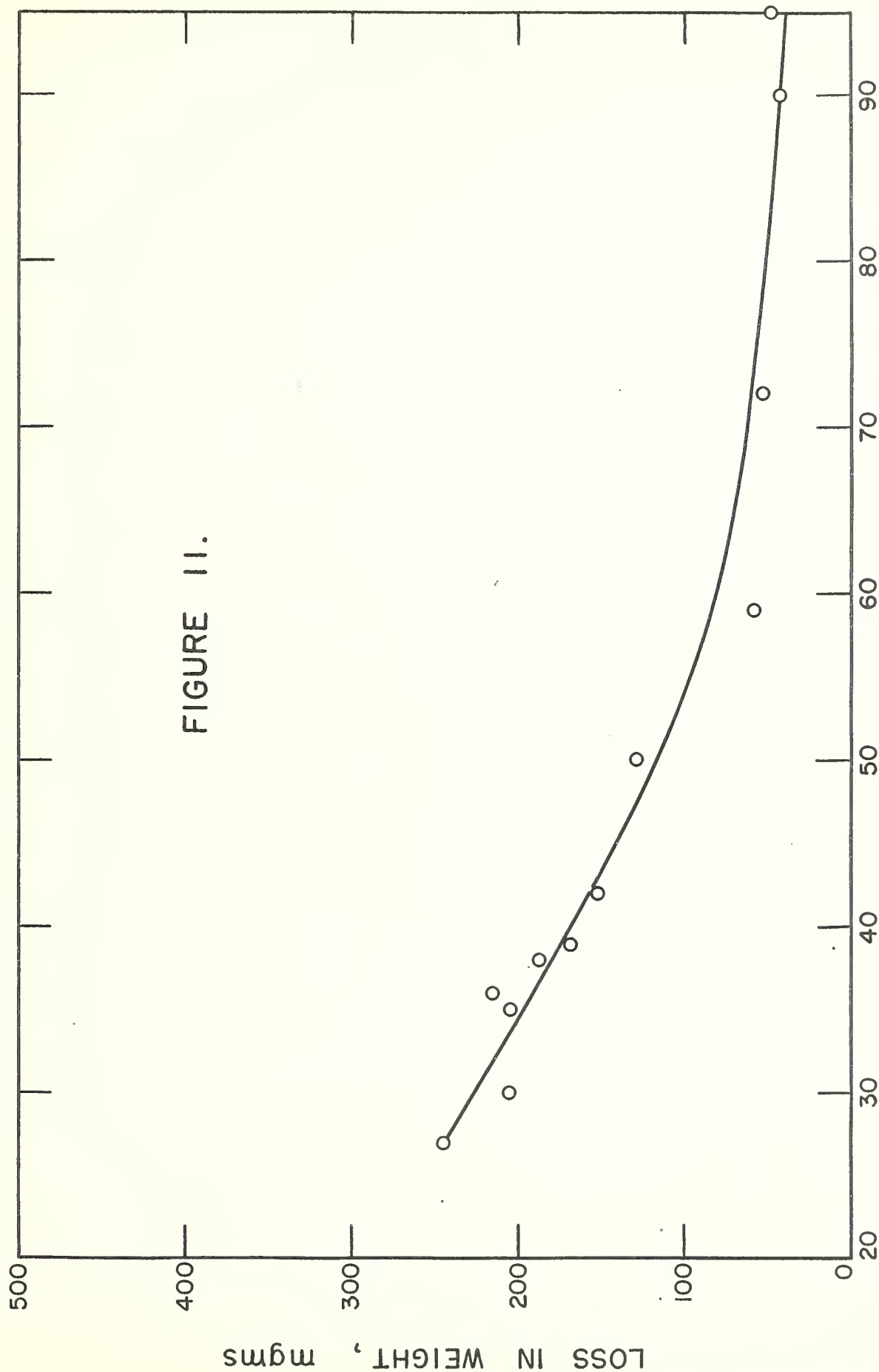




LOSS IN WEIGHT AT 300 HOURS VS. DURABILITY

FIGURE II.

DURABILITY, DAYS (51-9C)





# U. S. DEPARTMENT OF COMMERCE

Lewis L. Strauss, *Secretary*

## NATIONAL BUREAU OF STANDARDS

A. V. Astin, *Director*



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**Radio Propagation Engineering.** Data Reduction Instrumentation. Modulation Systems. Navigation Systems. Radio Noise. Tropospheric Measurements. Tropospheric Analysis. Radio Systems Application Engineering. Radio-Meteorology.

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