

EFFECTS OF ASPHALT CEMENT REJUVENATING AGENTS

FINAL REPORT

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Witco Chemical Company, Golden Bear Division

## METRIC CONVERSION FACTORS\*

| <u>To Convert from</u>     | <u>To</u>                                 | <u>Multiply by</u>           |
|----------------------------|---|------------------------------|
| <u>Length</u>              |   |                              |
| foot                       | meter (m)                                 | 0.3048                       |
| inch                       | millimeter (mm)                           | 25.4                         |
| yard                       | meter (m)                                 | 0.9144                       |
| mile (statute)             | kilometer (km)                            | 1.609                        |
| <u>Area</u>                |   |                              |
| square foot                | square meter (m <sup>2</sup> )            | 0.0929                       |
| square inch                | square centimeter (cm <sup>2</sup> )      | 6.451                        |
| square yard                | square meter (m <sup>2</sup> )            | 0.8361                       |
| <u>Volume (Capacity)</u>   |   |                              |
| cubic foot                 | cubic meter (m <sup>3</sup> )             | 0.02832                      |
| gallon (U.S. liquid)**     | cubic meter (m <sup>3</sup> )             | 0.003785                     |
| gallon (Can. liquid)**     | cubic meter (m <sup>3</sup> )             | 0.004546                     |
| ounce (U.S. liquid)        | cubic centimeter (cm <sup>3</sup> )       | 29.57                        |
| <u>Mass</u>                |   |                              |
| ounce-mass (avdp)          | gram (g)                                  | 28.35                        |
| pound-mass (avdp)          | kilogram (kg)                             | 0.4536                       |
| ton (metric)               | kilogram (kg)                             | 1000                         |
| ton (short, 2000 lbm)      | kilogram (kg)                             | 907.2                        |
| <u>Mass per Volume</u>     |   |                              |
| pound-mass/cubic foot      | kilogram/cubic meter (kg/m <sup>3</sup> ) | 16.02                        |
| pound-mass/cubic yard      | kilogram/cubic meter (kg/m <sup>3</sup> ) | 0.5933                       |
| pound-mass/gallon (U.S.)** | kilogram/cubic meter (kg/m <sup>3</sup> ) | 119.8                        |
| pound-mass/gallon (Can.)** | kilogram/cubic meter (kg/m <sup>3</sup> ) | 99.78                        |
| <u>Temperature</u>         |   |                              |
| deg Celsius (C)            | kelvin (K)                                | $t_k = (t_c + 273.15)$       |
| deg Fahrenheit (F)         | kelvin (K)                                | $t_k = (t_f + 459.67) / 1.8$ |
| deg Fahrenheit (F)         | deg Celsius (C)                           | $t_c = (t_f - 32) / 1.8$     |

\*The reference source for information on SI units and more exact conversion factors is "Metric Practice Guide" ASTM E 380.

\*\*One U.S. gallon equals 0.8327 Canadian gallon.

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## ABSTRACT

Louisiana's initial work in the recycling of asphaltic concrete pavements has demonstrated the need to obtain a base of knowledge in the area of rejuvenating age-hardened reclaimed asphalt cement. In this report, eight rejuvenating agents are examined with regard to the rejuvenator's effect, at various levels of addition, upon age-hardened asphalts; the subsequent aging characteristics of the rejuvenated asphalts; and the uniformity of mixing of the rejuvenating agents with asphaltic concrete mixes. Prediction equations yielding the proper quantity of rejuvenator to be added to an oxidized asphalt were examined. The rejuvenated asphalts followed anticipated patterns when tested for penetration at 77°F, viscosity at 140°F and 275°F, and ductility at 77°F. Generally, the rejuvenated asphalts demonstrated acceptable viscosity indices when subjected to the Thin Film Oven Test. Testing for uniformity of mixing in asphaltic concrete mixes proved inconclusive.

## IMPLEMENTATION STATEMENT

The recommendations of this report call for the Department to utilize the results of this study in the development of the Department's recycling program. A direct application of the prediction equation confirmed in this report will provide the necessary information for recycle mix design. The viscosity indices examined will provide a means to approve rejuvenating agents for a qualified products list.



## INTRODUCTION

The present-day awareness of the need to conserve available raw materials and energy, along with the current economics of highway construction, has evolved recycling of asphaltic concrete pavements. Innovations in milling equipment for the removal of existing pavements efficiently and economically have created an accessible supply of materials for these recycling efforts. Most of the recovered paving mixtures, however, have an asphalt binder that has hardened due to aging. A logical step to be taken when reworking the mixture is to add a softening or rejuvenating agent to increase the penetration and lower the viscosity of the aged binder. Such rejuvenation should not only increase the workability of the recycled mix at the time of reconstruction, but should also yield added service life to the resulting asphalt binder.

This study is concerned with acquiring a basic familiarity with several asphalt cement rejuvenating agents in anticipation of their future use in Louisiana recycling operations. Of primary importance is the development of a consistent relationship between aged asphalt cement and a rejuvenating agent which would provide an approximation of when and in what quantities these additives may be incorporated in recycled mixtures. The effect of rejuvenating agents on the subsequent aging characteristics of the binder and the uniformity of mixing of the rejuvenating agents in asphaltic concrete mixes is examined.

## SCOPE

This laboratory study was conducted in three phases:

Phase I consisted of the determination of the rejuvenating agents effect, at various levels of addition, upon age-hardened asphalts from different sources. Eight rejuvenators, each at four different levels of addition, were combined with each of two asphalt cements which had been laboratory aged to two different states of oxidation. The combined binder was then characterized by viscosities at 140°F and 275°F, penetration at 77°F, and ductility at 77°F.

Phase II investigated the subsequent aging characteristic of the rejuvenated asphalts. The Thin Film Oven Test was used to determine the aging index of the two original asphalts and of the rejuvenated asphalts.

Phase III included the testing for uniformity of mixing of the rejuvenating agents with asphaltic concrete mixes. Marshall test properties were used for this evaluation.

The intent of this study was not to evaluate one manufacturer against another, but rather to obtain basic knowledge for subsequent decision making.

## METHODOLOGY

A request was made to various companies in regard to the use of their rejuvenating agents under the auspices of this study and the scope presented herein. In response, ten rejuvenators from six manufacturers were forwarded. Two of these rejuvenators were eliminated from consideration: the first was an emulsion and thus deemed inappropriate as a softening agent for a hot plant recycling process; the second had a low flash point (below 350°F).

Sufficient supplies of two AC-30 grade asphalt cements--Exxon and Lion--were obtained. Originally, it was thought that these asphalts could be aged in a prolonged thin film oven exposure. The logistics of aging enough asphalt for the study negated this idea. It was then decided to place an amount of the Exxon asphalt in a shallow aggregate sample pan and subject this binder to continuous 400°F oven heating. The material was agitated twice daily to prevent a crust from forming on the surface and to ensure that the material in the pan was being oxidized uniformly. Each morning a sample was taken and the viscosity at 140°F was tested. The following schedule was obtained:

| <u>Hours of Exposure</u> | <u>Viscosity (Poise)</u> |
|--------------------------|--------------------------|
| 24                       | 6,525                    |
| 48                       | 13,328                   |
| 72                       | 24,245                   |
| 96                       | 44,881                   |
| 120                      | 92,132                   |

Based on this data, it was assumed that the viscosity would approximately double during each twenty-four-hour period. As a representative sampling of oxidized pavement materials to be recycled might typically yield viscosities of 50,000 and 100,000 poises, it was decided to choose aging periods of 96 and 120 hours.

## Phase I

The asphalt cements from the two different sources were aged to two different states of oxidation according to the above plan. These oxidized states were classified by viscosity at 140°F and 275°F, penetration at 77°F, and ductility at 77°F (Table 1).\* Original viscosities of each of the rejuvenating agents were then determined (Table 2). At that point, it was necessary to choose the four levels of rejuvenator addition.

Previous work published by various authors\*\* has examined the relationship between a blended oxidized asphalt cement and a rejuvenating agent. This relationship has been defined in the form of three equations which can be used to predict the rejuvenator content necessary to produce a desired viscosity:

$$\begin{aligned}\log(V) &= a + bp && (a) \\ \log-\log(V) &= a + bp && (b) \\ \log-\log(V) &= a + b(\log p) && (c)\end{aligned}$$

where:

V = viscosity of the blend (measured at 140°F  
in centipoises)

p = volume percent rejuvenator in blend

a and b = constants to be determined for each asphalt-  
rejuvenator-blend

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\*All tables may be found in Appendix A (page 13).

\*\*"Evaluation of Selected Recycling Modifiers," Holmgreen, R. J., and Epps, J. A., prepared for National Cooperative Highway Research Program.

As equation (b) was more familiar to the authors, it was used to determine the preliminary relationship between the rejuvenators obtained for this study and each of the two oxidized states of the two source asphalts. Axes were plotted on semi-log paper with the viscosity axis ranging from  $10^1$  to  $10^8$  centipoises and the percent rejuvenator axis ranging from 0 to 100 percent. One end point was determined from the viscosity of the oxidized asphalt (0 percent rejuvenator); the other end point was determined from the viscosity of the rejuvenator (100 percent rejuvenator). An example of this procedure is shown in Figure 1.\* Based on the relationship determined in this manner, the four levels of addition were chosen for each rejuvenator. One level for each rejuvenator was selected to bring the oxidized asphalt back to its original (pre-oxidized) state. The other levels were chosen to be both reasonable (with respect to quantity) and to provide a good spread along the curve. Table 3 gives the four addition levels selected for further testing for each of the rejuvenators.

Quantities of the oxidized asphalts were combined with the selected addition levels of each rejuvenator and were placed in separate containers. The matrix for testing purposes for each rejuvenator is represented by that for Rejuvenator A (Figure 2). Each of the blends was classified by testing for viscosity at 140°F and 275°F, penetration at 77°F, and ductility at 77°F.

## Phase II

The Thin Film Oven Test was used to determine the aging index of each of the original asphalt cements. This index is simply the oven-aged viscosity divided by the original viscosity and is intended to classify asphalts by characteristic hardening susceptibilities.

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\*All figures may be found in Appendix B (page 29).

The corresponding aging indices for the rejuvenated asphalts were also determined. For each rejuvenator, the resulting rejuvenated asphalt whose viscosity most closely matched that of the original asphalt was chosen for testing. The index was found by dividing the viscosity of the thin film oven-aged rejuvenated asphalt by the viscosity of the rejuvenated asphalt.

### Phase III

In this phase, Marshall test properties were determined and compared for the following identical mixes, with the same mixing and compaction temperature being used for all mixes:

- (1) briquettes made with new asphalt;
- (2) briquettes made with a rejuvenated binder; the binder was composed of oxidized asphalt, pre-blended with the appropriate amount of rejuvenator to obtain the same viscosity as the new asphalt (as determined by the method described in Phase I); and
- (3) briquettes made with a rejuvenated binder; in this case, the appropriate amount of rejuvenator was added to a mixture of oxidized asphalt and aggregate.

This matrix was examined five times with three different rejuvenating agents being blended with the first oxidation level of Exxon AC-30 and two different rejuvenators being blended with the second oxidation level of Lion AC-30. The five rejuvenators were chosen to represent the full viscosity range of the rejuvenators indicated in Table 2 (page 16).

## DISCUSSION OF RESULTS

### Phase I

Tables 4 through 7 (pages 18-21) present the results determined from the classification testing accomplished on the rejuvenated asphalts. The results show the anticipated increase in penetration and decrease in viscosity with greater levels of rejuvenator concentration. Ductilities were not run on the lowest and highest levels of concentration; these levels were chosen to obtain a good spread of viscosity data for curve fitting and would not be utilized as viable blends in hot mix design. It should be noted that the viscosities associated with blends F1, G1 and H1 in Table 7 (251032, 245537 and 292929 poises, respectively) exhibit values greater than the oxidized asphalt cement itself (195313 poises from Table 1). The absolute viscosities were tested according to ASTM D 2171, which states its applicability to materials having viscosities less than 200,000 poises. It is possible that the accuracy in testing near this limit may be such that the value of the viscosity of the oxidized asphalt cement was actually outside the test limit, thereby allowing the inconsistently high values observed in Table 7.

The viscosity at 140°F generated in this phase was used to examine the relationship between viscosity and percent rejuvenator addition as described in the Methodology section. Each prediction equation was examined to determine which equation would more closely approximate the data. The original asphalt cement and rejuvenator viscosity were plotted on axes correlated to equation (a) and equation (b) (page 4). Viscosity data was plotted on the corresponding graphs. Examples are presented in Figures 3 and 4 (pages 33 and 34). Equation (c),  $\log\text{-}\log(V) = a + b(\log p)$ , was not considered as the  $\log(p)$  is not defined when no rejuvenator is considered ( $p = 0$ ).

Due to the different axis scales and the difficulty of graphically determining whether a given data point was closer to the prediction curve from equation (a) or (b), the difference between actual and predicted viscosities from each equation was numerically examined. A smaller absolute value of this difference was considered indicative of the better relationship. Table 8 (page 22) presents a step-by-step example of this procedure. The data generated by this analysis is shown in Table 9. Ninety percent of the blends examined in this phase demonstrated equation (b) to be better able to predict the observed viscosity.

### Phase II

The viscosity indices (V.I.) for the original Exxon and Lion asphalt cements were 1.80 and 1.90, respectively. Figures 5 through 8 (pages 35-38) present the viscosity indices of the rejuvenated asphalts for each state of artificial oxidation. The solid bar represents the viscosity of that rejuvenated asphalt which most closely matches, for each rejuvenator, the viscosity of the original asphalt cement. The clear portion of each bar shows the viscosity of the rejuvenated asphalt after thin film oven testing.

A viscosity index of 4.0 has been established as the maximum allowable for use in Louisiana. Rejuvenator H exceeds this limit in Figure 8 and approaches this limit in the other three cases. While rejuvenators F and G do not exceed the maximum allowable index, they do approach the limit especially for the highly oxidized original asphalts (second state of oxidation). The viscosity indices for the remaining rejuvenated asphalts were acceptable, and these rejuvenators would be expected to perform similarly to new asphalt cements.

### Phase III

Marshall briquettes were formed according to the design indicated in the methodology and were tested for stability and flow. Mean Marshall properties are presented in Table 10. As shown, the



briquettes made with the pre-blended binder have slightly higher stabilities than those briquettes where the rejuvenator was added to the mixture of aggregate and oxidized asphalt. Although it is possible that the rejuvenating agents, in the case where they were added separately to the mix, had not fully blended with the oxidized asphalt at the time of testing, it is felt that the slight difference in stabilities between the pre-blended binder and the separately added rejuvenator can be attributed to normal testing error.

No discernible differences in mix properties were observed between the tested rejuvenator types.

## CONCLUSIONS

The following conclusions are drawn from the data generated in this study and, as such, are confined to the source and grade of asphalt cement examined.

1. The prediction equation,  $\log\text{-}\log(V) = a + bp$ , can be used as a first approximation for the determination of rejuvenating agent requirements.
2. The addition of an appropriate quantity of rejuvenating agent to an oxidized asphalt can provide a binder whose properties closely match those of an original asphalt.
3. All of the rejuvenating agents with the exception of rejuvenator H displayed acceptable hardening characteristics when subjected to the Thin Film Oven Test.
4. Testing for uniformity of mixing of the rejuvenating agents with asphaltic concrete mixes was inconclusive.

## RECOMMENDATIONS

The properties of the aged asphalt cement in the reclaimed hot mix will, for the majority of cases, be in need of some type of rejuvenation in order to be considered as an effective binder in a recycled mix. With this in mind and recognizing the conclusions drawn from this study, the following recommendations are made:

1. As shown in this study, a standardized viscosity age-hardening index (V.I.) should be utilized for the qualification of rejuvenating agents.
2. The  $\log\text{-}\log(V) = a + bp$  prediction equation should be used as a first approximation to determine the quantity of rejuvenating agent necessary in recycled mixes.
3. The uniformity of rejuvenator mixing with the oxidized asphalt cement in reclaimed hot mix should be examined further. It is recommended that samples be taken and analyzed from a recycling project in which the rejuvenating agent is introduced to the mix by different methods.

APPENDIX A

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TABLE 1  
ORIGINAL AND OXIDIZED ASPHALT CEMENT PROPERTIES

| <u>Asphalt<br/>Cement</u> | <u>Oxidation<br/>State</u> | <u>Penetration<br/>(77°F)</u> | <u>Viscosity<br/>(Poise @<br/>140°F)</u> | <u>Viscosity<br/>(Saybolt Furol<br/>Seconds @<br/>275°F)</u> | <u>Ductility<br/>(77°F)</u> |
|---------------------------|----------------------------|-------------------------------|--|--|-----------------------------|
| Exxon                     | New                        | 60                            | 3,151                                    | 302  | 100+                        |
| Exxon                     | 1st                        | 19                            | 57,872                                   | 1,134  | 15                          |
| Exxon                     | 2nd                        | 10                            | 125,555                                  | 1,626  | .6                          |
| Lion                      | New                        | 54                            | 2,673                                    | 252  | 100+                        |
| Lion                      | 1st                        | 15                            | 64,736                                   | 1,017  | 10                          |
| Lion                      | 2nd                        | 10                            | 195,313                                  | *  | 4                           |

\*This sample too hard to test.

TABLE 2

## REJUVENATOR VISCOSITIES

| <u>Rejuvenator</u> | <u>Viscosity<br/>(Poise @ 140°F)</u> |
|--------------------|--------------------------------------|
| A                  | 1.67                                 |
| B                  | 110.67                               |
| C                  | 1.27                                 |
| D                  | 25.02                                |
| E                  | 85.0                                 |
| F                  | 0.19                                 |
| G                  | 0.30                                 |
| H                  | 0.12                                 |

TABLE 3  
REJUVENATOR ADDITION LEVELS

| <u>Rejuvenator<br/>/Level</u> | <u>Percent<br/>Addition</u> | <u>Rejuvenator<br/>/Level</u> | <u>Percent<br/>Addition</u> |
|-------------------------------|-----------------------------|-------------------------------|-----------------------------|
| A1                            | 5                           | E1                            | 15                          |
| A2                            | 13                          | E2                            | 30                          |
| A3                            | 21                          | E3                            | 45                          |
| A4                            | 29                          | E4                            | 60                          |
| B1                            | 20                          | F1                            | 3                           |
| B2                            | 35                          | F2                            | 9                           |
| B3                            | 50                          | F3                            | 15                          |
| B4                            | 65                          | F4                            | 21                          |
| C1                            | 5                           | G1                            | 3                           |
| C2                            | 13                          | G2                            | 9                           |
| C3                            | 21                          | G3                            | 15                          |
| C4                            | 29                          | G4                            | 21                          |
| D1                            | 15                          | H1                            | 3                           |
| D2                            | 25                          | H2                            | 9                           |
| D3                            | 35                          | H3                            | 15                          |
| D4                            | 45                          | H4                            | 21                          |

TABLE 4  
 PROPERTIES OF REJUVENATED EXXON -  
 FIRST OXIDATION STATE

| <u>Rejuvenated Asphalt</u> | <u>Penetration (77°F)</u> | <u>Viscosity (Poise @ 140°F)</u> | <u>Viscosity (Saybolt Furol Seconds @ 275°F)</u> | <u>Ductility (77°F)</u> |
|----------------------------|---------------------------|----------------------------------|--|-------------------------|
| A1                         | 27                        | 17,447                           | 655  | -                       |
| A2                         | 53                        | 4,390                            | 342  | 100+                    |
| A3                         | 119                       | 1,236                            | 188  | 100+                    |
| A4                         | 238                       | 433                              | 110  | -                       |
| B1                         | 31                        | 12,094                           | 522  | -                       |
| B2                         | 51                        | 4,239                            | 325  | 100+                    |
| B3                         | 86                        | 1,533                            | 205  | 100+                    |
| B4                         | 150                       | 602                              | 135  | -                       |
| C1                         | 26                        | 21,527                           | 709  | -                       |
| C2                         | 58                        | 4,162                            | 327  | 100+                    |
| C3                         | 119                       | 1,175                            | 176  | 100+                    |
| C4                         | 247                       | 398                              | 102  | -                       |
| D1                         | 35                        | 8,846                            | 449  | -                       |
| D2                         | 67                        | 2,941                            | 260  | 100+                    |
| D3                         | 108                       | 1,258                            | 165  | 100+                    |
| D4                         | 190                       | 533                              | 108  | -                       |
| E1                         | 31                        | 12,686                           | 518  | -                       |
| E2                         | 56                        | 3,575                            | 266  | 100+                    |
| E3                         | 104                       | 1,171                            | 148  | 100+                    |
| E4                         | 184                       | 494                              | 94   | -                       |
| F1                         | 25                        | 24,436                           | 795  | -                       |
| F2                         | 52                        | 5,462                            | 405  | 100+                    |
| F3                         | 118                       | 1,460                            | 221  | 100+                    |
| F4                         | 258                       | 418                              | 129  | -                       |
| G1                         | 22                        | 29,186                           | 829  | -                       |
| G2                         | 44                        | 6,685                            | 423  | 100+                    |
| G3                         | 98                        | 1,654                            | 217  | 100+                    |
| G4                         | 180                       | 672                              | 137  | -                       |
| H1                         | 27                        | 26,607                           | 842  | -                       |
| H2                         | 60                        | 4,276                            | 373  | 100+                    |
| H3                         | 142                       | 1,056                            | 198  | 100+                    |
| H4                         | 266+*                     | 304                              | 114  | -                       |

\*The penetration needle hit the bottom of the sample cup.



TABLE 5  
 PROPERTIES OF REJUVENATED EXXON -  
 SECOND OXIDATION STATE

| <u>Rejuvenated<br/>Asphalt</u> | <u>Penetration<br/>(77°F)</u> | <u>Viscosity<br/>(Poise @ 140°F)</u> | <u>Viscosity<br/>(Saybolt Furol<br/>Seconds @ 275°F)</u> | <u>Ductility<br/>(77°F)</u> |
|--------------------------------|-------------------------------|--------------------------------------|--|-----------------------------|
| A1                             | 19                            | 39,916                               | 957  | -                           |
| A2                             | 41                            | 7,547                                | 455  | 100+                        |
| A3                             | 79                            | 2,092                                | 231  | 100+                        |
| A4                             | 192                           | 573                                  | 135  | -                           |
| B1                             | 23                            | 18,489                               | 710  | -                           |
| B2                             | 38                            | 7,185                                | 393  | 100+                        |
| B3                             | 73                            | 1,900                                | 235  | 100+                        |
| B4                             | 127                           | 720                                  | 149  | -                           |
| C1                             | 17                            | 35,194                               | 910  | -                           |
| C2                             | 44                            | 7,026                                | 431  | 100+                        |
| C3                             | 95                            | 1,739                                | 218  | 100+                        |
| C4                             | 217                           | 492                                  | 115  | -                           |
| D1                             | 27                            | 13,466                               | 589  | -                           |
| D2                             | 48                            | 4,821                                | 334  | 100+                        |
| D3                             | 98                            | 1,590                                | 198  | 100+                        |
| D4                             | 227                           | 660                                  | 126  | -                           |
| E1                             | 24                            | 18,384                               | 700  | -                           |
| E2                             | 45                            | 4,870                                | 330  | 100+                        |
| E3                             | 84                            | 1,608                                | 178  | 100+                        |
| E4                             | 170                           | 490                                  | 98   | -                           |
| F1                             | 16                            | 41,675                               | 1,074  | -                           |
| F2                             | 45                            | 7,817                                | 467  | 100+                        |
| F3                             | 98                            | 1,917                                | 234  | 100+                        |
| F4                             | 218                           | 556                                  | 135  | -                           |
| G1                             | 16                            | 58,433                               | 1,131  | -                           |
| G2                             | 39                            | 10,386                               | 527  | 100+                        |
| G3                             | 79                            | 2,730                                | 290  | 100+                        |
| G4                             | 167                           | 825                                  | 155  | -                           |
| H1                             | 16                            | 69,133                               | 1,249  | -                           |
| H2                             | 46                            | 7,162                                | 475  | 100+                        |
| H3                             | 99                            | 1,967                                | 260  | 100+                        |
| H4                             | 245                           | 452                                  | 129  | -                           |

TABLE 6  
 PROPERTIES OF REJUVENATED LION -  
 FIRST OXIDATION STATE

| Rejuvenated<br>Asphalt | Penetration<br>(77°F) | Viscosity<br>(Poise @ 140°F) | Viscosity<br>(Saybolt Furol<br>Seconds @ 275°F) | Ductility<br>(77°F) |
|------------------------|-----------------------|------------------------------|---|---------------------|
| A1                     | 24                    | 21,880                       | 619   | -                   |
| A2                     | 46                    | 4,957                        | 312   | 100+                |
| A3                     | 100                   | 1,343                        | 170   | 100+                |
| A4                     | 206                   | 444                          | 104   | -                   |
| B1                     | 30                    | 14,771                       | 508   | -                   |
| B2                     | 45                    | 5,000                        | 308   | 100+                |
| B3                     | 77                    | 1,689                        | 195   | 100+                |
| B4                     | 137                   | 384                          | 129   | -                   |
| C1                     | 22                    | 20,661                       | 605   | -                   |
| C2                     | 53                    | 4,448                        | 291   | 100+                |
| C3                     | 115                   | 1,086                        | 151   | 100+                |
| C4                     | 244                   | 364                          | 88  | -                   |
| D1                     | 33                    | 10,636                       | 421   | -                   |
| D2                     | 50                    | 3,491                        | 361   | 100+                |
| D3                     | 95                    | 1,382                        | 246   | 100+                |
| D4                     | 185                   | 511                          | 103   | -                   |
| E1                     | 30                    | 11,382                       | 432   | -                   |
| E2                     | 51                    | 3,568                        | 240   | 100+                |
| E3                     | 113                   | 1,030                        | 130   | 100+                |
| E4                     | 187                   | 464                          | 84  | -                   |
| F1                     | 23                    | 31,563                       | 727   | -                   |
| F2                     | 55                    | 4,907                        | 333   | 100+                |
| F3                     | 114                   | 1,256                        | 180   | 100+                |
| F4                     | 255+*                 | 341                          | 98  | -                   |
| G1                     | 21                    | 27,398                       | 670   | -                   |
| G2                     | 46                    | 5,648                        | 338   | 100+                |
| G3                     | 94                    | 1,585                        | 191   | 100+                |
| G4                     | 195                   | 519                          | 113   | -                   |
| H1                     | 25                    | 26,491                       | 680   | -                   |
| H2                     | 58                    | 3,778                        | 290   | 100+                |
| H3                     | 125                   | 1,080                        | 166   | 100+                |
| H4                     | 255+*                 | 280                          | 87  | -                   |

\*The penetration needle hit the bottom of the sample cup.

TABLE 7  
 PROPERTIES OF REJUVENATED LION -  
 SECOND OXIDATION STATE

| Rejuvenated<br>Asphalt | Penetration<br>(77°F) | Viscosity<br>(Poise @ 140°F) | Viscosity<br>(Saybolt Furol<br>Seconds @ 275°F) | Ductility<br>(77°F) |
|------------------------|-----------------------|------------------------------|---|---------------------|
| A1                     | 15                    | 143,760                      | 1,446   | -                   |
| A2                     | 29                    | 17,306                       | 569   | 100+                |
| A3                     | 56                    | 3,916                        | 278   | 100+                |
| A4                     | 118                   | 1,013                        | 150   | -                   |
| B1                     | 18                    | 58,752                       | 916   | -                   |
| B2                     | 31                    | 11,487                       | 478   | 100+                |
| B3                     | 52                    | 3,041                        | 260   | 100+                |
| B4                     | 105                   | 850                          | 155   | -                   |
| C1                     | 15                    | 128,552                      | 1,390   | -                   |
| C2                     | 27                    | 19,628                       | 572   | 100+                |
| C3                     | 63                    | 3,388                        | 254   | 100+                |
| C4                     | 135                   | 904                          | 136   | -                   |
| D1                     | 20                    | 55,274                       | 886   | -                   |
| D2                     | 31                    | 13,443                       | 455   | 100+                |
| D3                     | 58                    | 3,520                        | 242   | 100+                |
| D4                     | 107                   | 1,136                        | 139   | -                   |
| E1                     | 19                    | 76,117                       | 1,033   | -                   |
| E2                     | 32                    | 11,318                       | 422   | 100+                |
| E3                     | 65                    | 2,481                        | 195   | 100+                |
| E4                     | 139                   | 710                          | 106   | -                   |
| F1                     | 15                    | 251,032                      | *   | -                   |
| F2                     | 30                    | 29,430                       | 851   | 40                  |
| F3                     | 52                    | 6,352                        | 399   | 100+                |
| F4                     | 117                   | 1,464                        | 193   | -                   |
| G1                     | 13                    | 245,537                      | *   | -                   |
| G2                     | 24                    | 38,371                       | 906   | 48                  |
| G3                     | 48                    | 5,966                        | 362   | 100+                |
| G4                     | 88                    | 1,872                        | 211   | -                   |
| H1                     | 14                    | 292,929                      | *   | -                   |
| H2                     | 27                    | 22,584                       | 948   | 90                  |
| H3                     | 62                    | 3,748                        | 303   | 100+                |
| H4                     | 148                   | 914                          | 159   | -                   |

\*These samples were too hard to test.

TABLE 8

## PREDICTION EQUATION ANALYSIS

Observed Viscosity ( $V_O$ ) = 423,900 cp @ p = 35

Oxidized Asphalt Viscosity ( $V_A$ ) = 5,787,200 cp @ p = 0

Rejuvenator Viscosity ( $V_R$ ) = 11,067 cp @ p = 100

Determination of CoefficientsEquation (a)

$$\log(V_A) = a + b(0) = 6.76$$

$$a = 6.76$$

$$\log(V_R) = 6.76 + b(100) = 4.04$$

$$b = -.027$$

Equation (b)

$$\log-\log(V_A) = a + b(0) = .830$$

$$a = .830$$

$$\log-\log(V_R) = .830 + b(100)$$

$$b = -.0022$$

Prediction of Viscosity ( $V_p$ ) @ p = 35Equation (a)

$$\log(V_p) = 6.76 + (-.027)(35)$$

$$V_p = 6.53 \times 10^5 \text{ cp}$$

Equation (b)

$$\log-\log(V_p) = .830 + (-.0022)(35)$$

$$V_p = 4.6 \times 10^5 \text{ cp}$$

Difference Between Observed and Predicted ViscositiesEquation (a)

$$|V_O - V_p| = 2.29 \times 10^5 \text{ cp}$$

$$= 2,290 \text{ poise}$$

Equation (b)

$$|V_O - V_p| = 3.57 \times 10^4 \text{ cp}$$

$$= 357 \text{ poise}$$

TABLE 9

## COMPARISON OF OBSERVED AND PREDICTED VISCOSITIES\*

## a. REJUVENATED EXXON - FIRST OXIDATION STATE

| Rejuve-<br>nator | Addition<br>Level<br>(%) | Viscosity<br>Observed<br>(V <sub>o</sub> ) | Viscosity<br>Eq. (a)<br>(V <sub>a</sub> ) | Viscosity<br>Eq. (b)<br>(V <sub>b</sub> ) | V <sub>o</sub> -V <sub>a</sub> | V <sub>o</sub> -V <sub>b</sub> |
|------------------|--------------------------|--|---|---|--------------------------------|--------------------------------|
| A1               | 5                        | 17,446                                     | 34,300                                    | 25,000                                    | 16,800                         | 7,520                          |
| A2               | 13                       | 4,390                                      | 15,000                                    | 7,180                                     | 10,600                         | 2,790                          |
| A3               | 21                       | 1,236                                      | 6,530                                     | 2,290                                     | 5,300                          | 1,060                          |
| A4               | 29                       | 433  | 2,850                                     | 807                                       | 2,420                          | 374                            |
| B1               | 20                       | 12,094                                     | 16,600                                    | 12,900                                    | 4,500                          | 771                            |
| B2               | 35                       | 4,239                                      | 6,530                                     | 4,600                                     | 2,290                          | 357                            |
| B3               | 50                       | 1,533                                      | 2,570                                     | 1,770                                     | 1,040                          | 237                            |
| B4               | 65                       | 602  | 1,010                                     | 731                                       | 408                            | 129                            |
| C1               | 5                        | 21,527                                     | 33,500                                    | 23,700                                    | 12,000                         | 2,200                          |
| C2               | 13                       | 4,162                                      | 14,100                                    | 6,360                                     | 9,940                          | 2,200                          |
| C3               | 21                       | 1,175                                      | 5,930                                     | 1,920                                     | 4,750                          | 745                            |
| C4               | 29                       | 398  | 2,490                                     | 645                                       | 2,100                          | 247                            |
| D1               | 15                       | 8,846                                      | 17,800                                    | 12,500                                    | 8,940                          | 3,610                          |
| D2               | 25                       | 2,941                                      | 8,130                                     | 4,880                                     | 5,190                          | 1,940                          |
| D3               | 35                       | 1,258                                      | 3,720                                     | 2,040                                     | 2,460                          | 778                            |
| D4               | 45                       | 533  | 1,700                                     | 901                                       | 1,170                          | 368                            |
| E1               | 15                       | 12,686                                     | 21,900                                    | 16,700                                    | 9,190                          | 4,030                          |
| E2               | 30                       | 3,575                                      | 8,320                                     | 5,350                                     | 4,740                          | 1,770                          |
| E3               | 45                       | 1,171                                      | 3,160                                     | 1,870                                     | 1,990                          | 701                            |
| E4               | 60                       | 494  | 1,200                                     | 713                                       | 708                            | 219                            |
| F1               | 3                        | 24,436                                     | 39,400                                    | 27,100                                    | 14,900                         | 2,650                          |
| F2               | 9                        | 5,462                                      | 18,400                                    | 6,660                                     | 12,900                         | 1,200                          |
| F3               | 15                       | 1,460                                      | 8,610                                     | 1,870                                     | 7,150                          | 412                            |
| F4               | 21                       | 418  | 4,030                                     | 593                                       | 3,610                          | 175                            |
| G1               | 3                        | 29,186                                     | 39,900                                    | 28,800                                    | 10,700                         | 378                            |
| G2               | 9                        | 6,685                                      | 19,200                                    | 7,880                                     | 12,500                         | 1,200                          |
| G3               | 15                       | 1,654                                      | 9,230                                     | 2,410                                     | 7,570                          | 760                            |
| G4               | 21                       | 672  | 4,440                                     | 820                                       | 3,760                          | 148                            |
| H1               | 3                        | 26,607                                     | 38,800                                    | 25,000                                    | 12,200                         | 1,640                          |
| H2               | 9                        | 4,276                                      | 17,700                                    | 5,350                                     | 13,400                         | 1,070                          |
| H3               | 15                       | 1,056                                      | 8,040                                     | 1,340                                     | 6,980                          | 289                            |
| H4               | 21                       | 304  | 3,660                                     | 391                                       | 3,350                          | 87                             |

\*All viscosities are presented in poises. All viscosities have been rounded to three significant digits with the exception of the observed viscosity.

TABLE 9 (CONTINUED)

## COMPARISON OF OBSERVED AND PREDICTED VISCOSITIES

## b. REJUVENATED EXXON - SECOND OXIDATION STATE

| Rejuve-<br>nator | Addition<br>Level<br>(%) | Viscosity<br>Observed<br>( $V_o$ ) | Viscosity<br>Eq. (a)<br>( $V_a$ ) | Viscosity<br>Eq. (b)<br>( $V_b$ ) | $ V_o - V_a $ | $ V_o - V_b $ |
|------------------|--------------------------|------------------------------------|-----------------------------------|-----------------------------------|---------------|---------------|
| A1               | 5                        | 39,916                             | 71,600                            | 50,000                            | 31,700        | 10,100        |
| A2               | 13                       | 7,547                              | 29,000                            | 12,900                            | 21,500        | 5,320         |
| A3               | 21                       | 2,092                              | 11,800                            | 3,730                             | 9,710         | 1,640         |
| A4               | 29                       | 573                                | 4,780                             | 1,210                             | 4,200         | 634           |
| B1               | 20                       | 18,489                             | 30,200                            | 22,600                            | 11,700        | 4,070         |
| B2               | 35                       | 7,185                              | 10,400                            | 7,050                             | 3,170         | 140           |
| B3               | 50                       | 1,900                              | 3,550                             | 2,410                             | 1,650         | 510           |
| B4               | 65                       | 720                                | 1,220                             | 901                               | 496           | 181           |
| C1               | 5                        | 35,194                             | 70,800                            | 47,400                            | 35,600        | 12,200        |
| C2               | 13                       | 7,026                              | 28,200                            | 11,300                            | 21,200        | 4,320         |
| C3               | 21                       | 1,739                              | 11,200                            | 3,100                             | 9,480         | 1,360         |
| C4               | 29                       | 492                                | 4,470                             | 957                               | 3,970         | 465           |
| D1               | 15                       | 13,466                             | 35,100                            | 22,600                            | 21,600        | 9,090         |
| D2               | 25                       | 4,821                              | 15,000                            | 7,980                             | 10,100        | 3,160         |
| D3               | 35                       | 1,590                              | 6,380                             | 3,040                             | 4,790         | 1,450         |
| D4               | 45                       | 660                                | 2,720                             | 1,240                             | 2,060         | 580           |
| E1               | 15                       | 18,384                             | 41,700                            | 30,600                            | 23,300        | 12,300        |
| E2               | 30                       | 4,870                              | 13,800                            | 8,500                             | 8,930         | 3,630         |
| E3               | 45                       | 1,608                              | 4,570                             | 2,630                             | 2,960         | 1,020         |
| E4               | 60                       | 490                                | 1,510                             | 901                               | 1,020         | 411           |
| F1               | 3                        | 41,675                             | 84,300                            | 55,200                            | 42,700        | 13,600        |
| F2               | 9                        | 7,817                              | 37,800                            | 12,200                            | 30,000        | 4,400         |
| F3               | 15                       | 1,917                              | 17,000                            | 3,130                             | 15,100        | 1,210         |
| F4               | 21                       | 556                                | 7,620                             | 915                               | 7,060         | 359           |
| G1               | 3                        | 58,433                             | 85,500                            | 58,900                            | 27,100        | 475           |
| G2               | 9                        | 10,386                             | 39,400                            | 14,600                            | 29,100        | 4,170         |
| G3               | 15                       | 2,730                              | 18,200                            | 4,080                             | 15,500        | 1,350         |
| G4               | 21                       | 825                                | 8,390                             | 1,280                             | 7,570         | 456           |
| H1               | 3                        | 69,133                             | 83,200                            | 50,700                            | 14,000        | 18,400        |
| H2               | 9                        | 7,162                              | 36,300                            | 9,700                             | 29,100        | 2,540         |
| H3               | 15                       | 1,967                              | 15,800                            | 2,220                             | 13,900        | 249           |
| H4               | 21                       | 452                                | 6,920                             | 593                               | 6,470         | 141           |

TABLE 9 (CONTINUED)

COMPARISON OF OBSERVED AND PREDICTED VISCOSITIES  
 c. REJUVENATED LION - FIRST OXIDATION STATE

| Rejuve-<br>nator | Addition<br>Level<br>(%) | Viscosity<br>Observed<br>( $V_o$ ) | Viscosity<br>Eq. (a)<br>( $V_a$ ) | Viscosity<br>Eq. (b)<br>( $V_b$ ) | $ V_o - V_a $ | $ V_o - V_b $ |
|------------------|--------------------------|------------------------------------|-----------------------------------|-----------------------------------|---------------|---------------|
| A1               | 5                        | 21,800                             | 38,000                            | 27,200                            | 16,100        | 5,300         |
| A2               | 13                       | 4,957                              | 16,300                            | 7,570                             | 11,300        | 2,610         |
| A3               | 21                       | 1,343                              | 6,980                             | 2,350                             | 5,640         | 1,010         |
| A4               | 29                       | 444                                | 2,990                             | 809                               | 2,550         | 365           |
| B1               | 20                       | 14,771                             | 17,800                            | 13,300                            | 3,010         | 1,480         |
| B2               | 35                       | 5,000                              | 6,760                             | 4,530                             | 1,760         | 472           |
| B3               | 50                       | 1,689                              | 2,570                             | 1,670                             | 881           | 19            |
| B4               | 65                       | 384                                | 977                               | 668                               | 593           | 284           |
| C1               | 5                        | 20,661                             | 37,600                            | 26,300                            | 16,900        | 5,610         |
| C2               | 13                       | 4,448                              | 15,800                            | 6,980                             | 11,400        | 2,530         |
| C3               | 21                       | 1,086                              | 6,650                             | 2,090                             | 5,570         | 1,000         |
| C4               | 29                       | 364                                | 2,800                             | 696                               | 2,430         | 332           |
| D1               | 15                       | 10,636                             | 20,000                            | 13,700                            | 9,320         | 3,090         |
| D2               | 25                       | 3,491                              | 9,120                             | 5,350                             | 5,630         | 1,850         |
| D3               | 35                       | 1,382                              | 4,170                             | 2,220                             | 2,790         | 834           |
| D4               | 45                       | 511                                | 1,910                             | 975                               | 1,390         | 464           |
| E1               | 15                       | 11,382                             | 23,700                            | 18,500                            | 12,300        | 7,070         |
| E2               | 30                       | 3,568                              | 8,710                             | 5,860                             | 5,140         | 2,290         |
| E3               | 45                       | 1,030                              | 3,200                             | 2,040                             | 2,170         | 1,010         |
| E4               | 60                       | 464                                | 1,170                             | 770                               | 711           | 306           |
| F1               | 3                        | 31,563                             | 44,200                            | 29,700                            | 12,600        | 1,850         |
| F2               | 9                        | 4,907                              | 20,700                            | 7,110                             | 15,700        | 2,200         |
| F3               | 15                       | 1,256                              | 9,660                             | 1,950                             | 8,400         | 696           |
| F4               | 21                       | 341                                | 4,520                             | 607                               | 4,180         | 266           |
| G1               | 3                        | 27,398                             | 44,800                            | 31,900                            | 17,400        | 4,540         |
| G2               | 9                        | 5,648                              | 21,500                            | 8,660                             | 15,900        | 3,010         |
| G3               | 15                       | 1,585                              | 10,400                            | 2,630                             | 8,770         | 1,050         |
| G4               | 21                       | 519                                | 4,980                             | 886                               | 4,460         | 367           |
| H1               | 3                        | 26,491                             | 43,600                            | 27,600                            | 17,100        | 1,160         |
| H2               | 9                        | 3,778                              | 19,800                            | 5,860                             | 16,000        | 2,080         |
| H3               | 15                       | 1,080                              | 9,020                             | 1,460                             | 7,940         | 379           |
| H4               | 21                       | 280                                | 4,100                             | 421                               | 3,820         | 141           |

TABLE 9 (CONTINUED)

COMPARISON OF OBSERVED AND PREDICTED VISCOSITIES  
d. REJUVENATED LION - SECOND OXIDATION STATE

| Rejuve-<br>nator | Addition<br>Level<br>(%) | Viscosity<br>Observed<br>( $V_o$ ) | Viscosity<br>Eq. (a)<br>( $V_a$ ) | Viscosity<br>Eq. (b)<br>( $V_b$ ) | $ V_o - V_a $ | $ V_o - V_b $ |
|------------------|--------------------------|------------------------------------|-----------------------------------|-----------------------------------|---------------|---------------|
| A1               | 5                        | 143,760                            | 180,000                           | 74,200                            | 35,400        | 69,500        |
| A2               | 13                       | 17,306                             | 42,400                            | 17,500                            | 25,100        | 196           |
| A3               | 21                       | 3,916                              | 16,600                            | 4,710                             | 12,700        | 792           |
| A4               | 29                       | 1,013                              | 6,470                             | 1,430                             | 5,460         | 415           |
| B1               | 20                       | 58,752                             | 42,700                            | 29,600                            | 16,100        | 29,100        |
| B2               | 35                       | 11,487                             | 13,600                            | 8,230                             | 2,160         | 3,250         |
| B3               | 50                       | 3,041                              | 4,370                             | 2,560                             | 1,320         | 485           |
| B4               | 65                       | 850                                | 1,400                             | 877                               | 546           | 27            |
| C1               | 5                        | 128,552                            | 107,000                           | 71,600                            | 21,400        | 57,000        |
| C2               | 13                       | 19,628                             | 41,100                            | 16,100                            | 21,500        | 3,560         |
| C3               | 21                       | 3,388                              | 15,800                            | 4,150                             | 12,400        | 764           |
| C4               | 29                       | 1,136                              | 6,050                             | 1,220                             | 4,920         | 84            |
| D1               | 15                       | 55,274                             | 50,700                            | 32,300                            | 4,570         | 23,000        |
| D2               | 25                       | 13,443                             | 20,700                            | 10,800                            | 7,210         | 2,670         |
| D3               | 35                       | 3,520                              | 8,410                             | 3,900                             | 4,890         | 381           |
| D4               | 45                       | 1,136                              | 3,430                             | 1,520                             | 2,290         | 385           |
| E1               | 15                       | 76,117                             | 60,300                            | 44,200                            | 15,900        | 32,000        |
| E2               | 30                       | 11,318                             | 18,600                            | 11,300                            | 7,300         | 18            |
| E3               | 45                       | 2,481                              | 5,750                             | 3,270                             | 3,270         | 788           |
| E4               | 60                       | 710                                | 1,780                             | 1,060                             | 1,070         | 345           |
| F1               | 3                        | 251,032                            | 129,000                           | 83,500                            | 122,000       | 168,000       |
| F2               | 9                        | 29,430                             | 56,200                            | 17,000                            | 26,800        | 12,400        |
| F3               | 15                       | 6,352                              | 24,500                            | 4,080                             | 18,200        | 2,270         |
| F4               | 21                       | 1,464                              | 10,700                            | 1,130                             | 9,250         | 339           |
| G1               | 3                        | 245,537                            | 131,000                           | 90,200                            | 115,000       | 155,000       |
| G2               | 9                        | 38,371                             | 58,600                            | 21,000                            | 20,200        | 17,300        |
| G3               | 15                       | 5,966                              | 26,300                            | 5,590                             | 20,300        | 371           |
| G4               | 21                       | 1,872                              | 11,800                            | 1,680                             | 9,930         | 193           |
| H1               | 3                        | 292,929                            | 127,000                           | 77,300                            | 166,000       | 216,000       |
| H2               | 9                        | 22,584                             | 54,000                            | 13,900                            | 31,400        | 8,720         |
| H3               | 15                       | 3,748                              | 22,900                            | 3,000                             | 19,200        | 753           |
| H4               | 21                       | 914                                | 9,730                             | 764                               | 8,810         | 150           |



TABLE 10

MEAN MARSHALL PROPERTIES FOR REJUVENATED ASPHALT MIXES

|             | <u>Binder Composition</u> |                                       |                                       |                                       |                          |                                      |                                      |
|-------------|---------------------------|---------------------------------------|---------------------------------------|---------------------------------------|--------------------------|--------------------------------------|--------------------------------------|
|             | <u>Original<br/>Exxon</u> | <u>Oxidized<br/>Exxon<br/>+ REJ G</u> | <u>Oxidized<br/>Exxon<br/>+ REJ C</u> | <u>Oxidized<br/>Exxon<br/>+ REJ D</u> | <u>Original<br/>Lion</u> | <u>Oxidized<br/>Lion<br/>+ REJ F</u> | <u>Oxidized<br/>Lion<br/>+ REJ A</u> |
| Stability   | 1,495                     |                                       |                                       |                                       | 1,414                    |                                      |                                      |
| Flow        | 9                         |                                       |                                       |                                       | 9                        |                                      |                                      |
| Stability*  |                           | 1,452                                 | 1,381                                 | 1,406                                 |                          | 1,534                                | 1,576                                |
| Flow        |                           | 11                                    | 10                                    | 11                                    |                          | 10                                   | 9                                    |
| Stability** |                           | 1,242                                 | 1,283                                 | 1,331                                 |                          | 1,394                                | 1,405                                |
| Flow        |                           | 10                                    | 13                                    | 10                                    |                          | 9                                    | 8                                    |

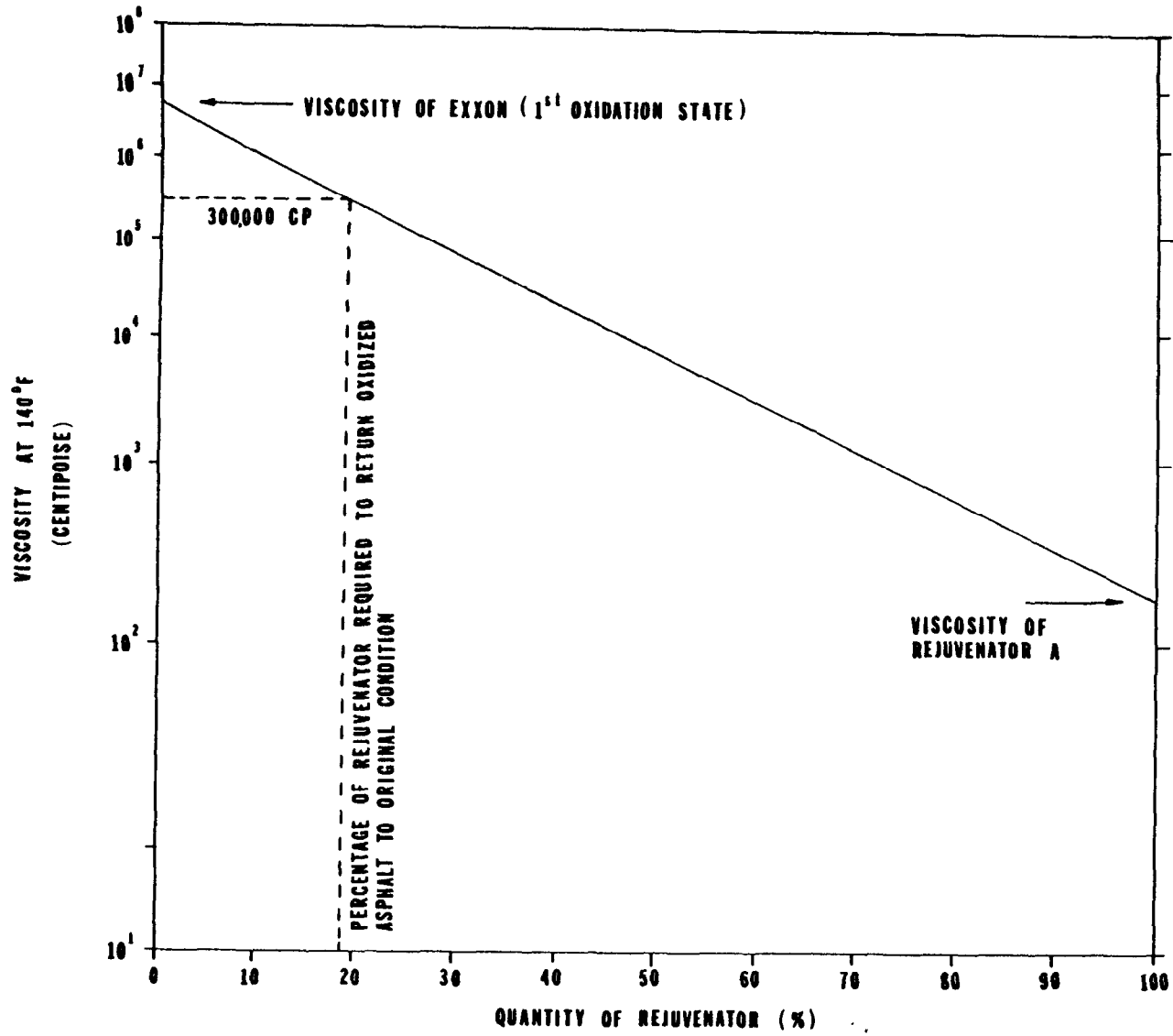
\*Mixes comprised of pre-blended oxidized asphalt and rejuvenator.

\*\*Mixes comprised of aggregate coated with oxidized asphalt to which rejuvenator was added.

APPENDIX B

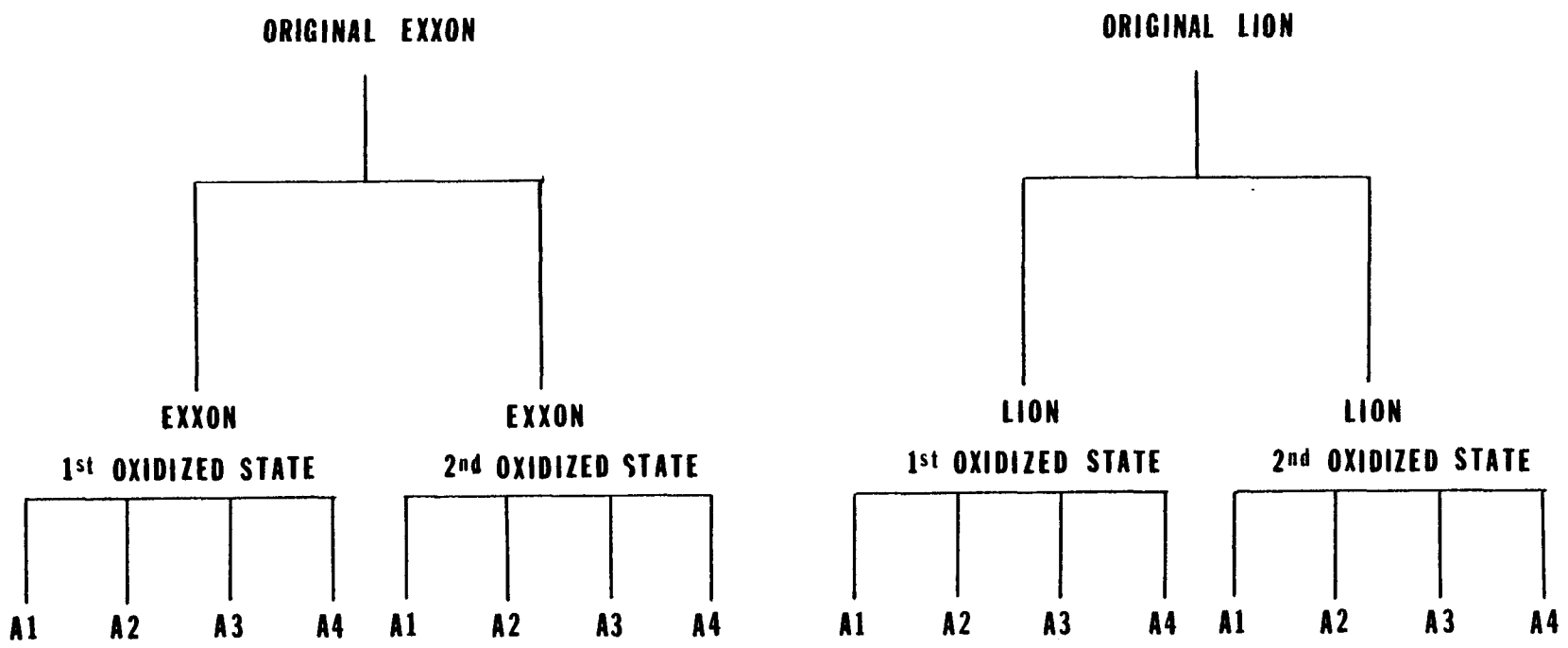
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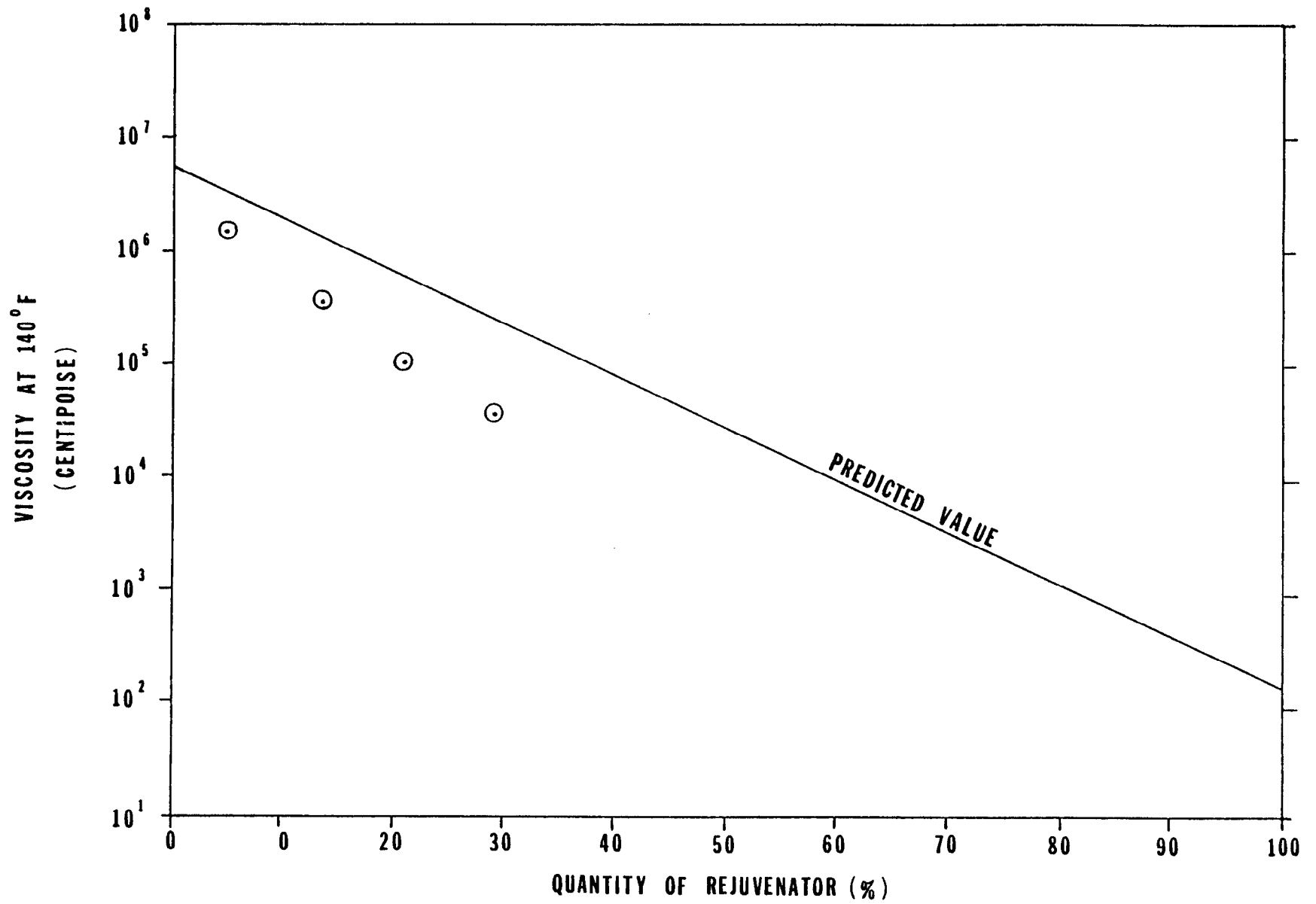
Prediction Equation Graphic  $\text{Log-Log } V = a + bp$

FIGURE 1



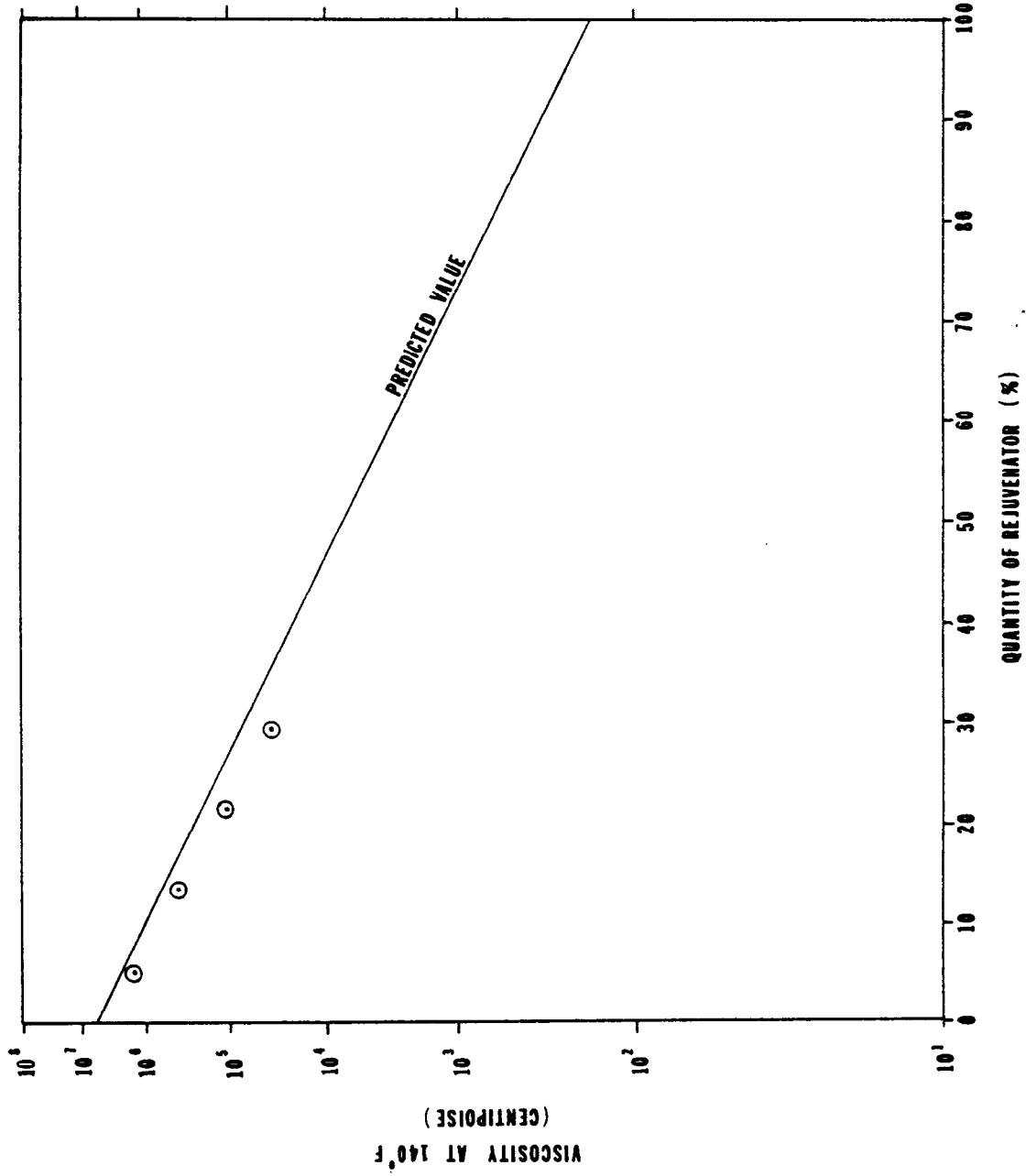
*Phase I Testing Matrix*

FIGURE 2



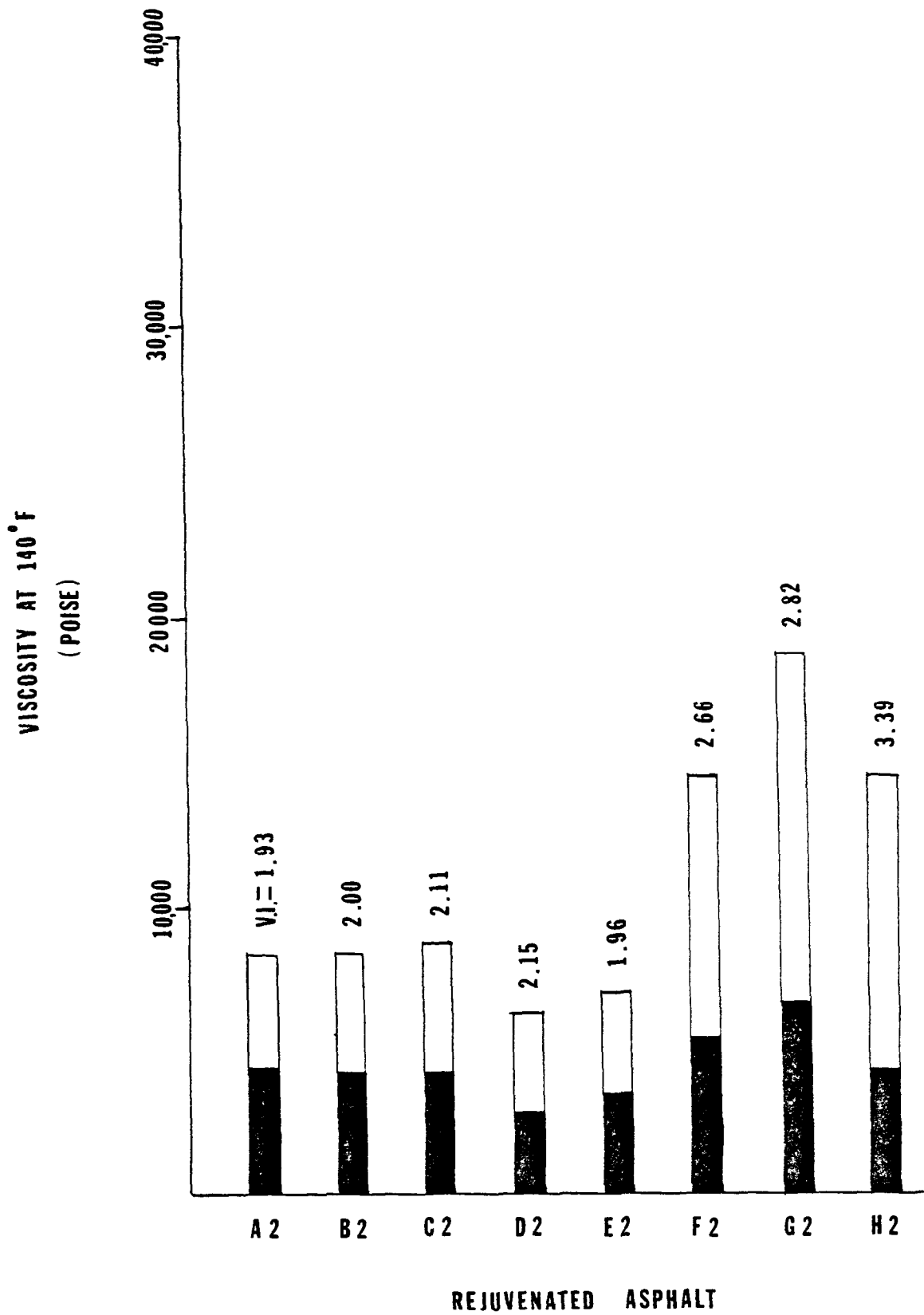
*Log V = a + bp Correlation Graphic*

FIGURE 3



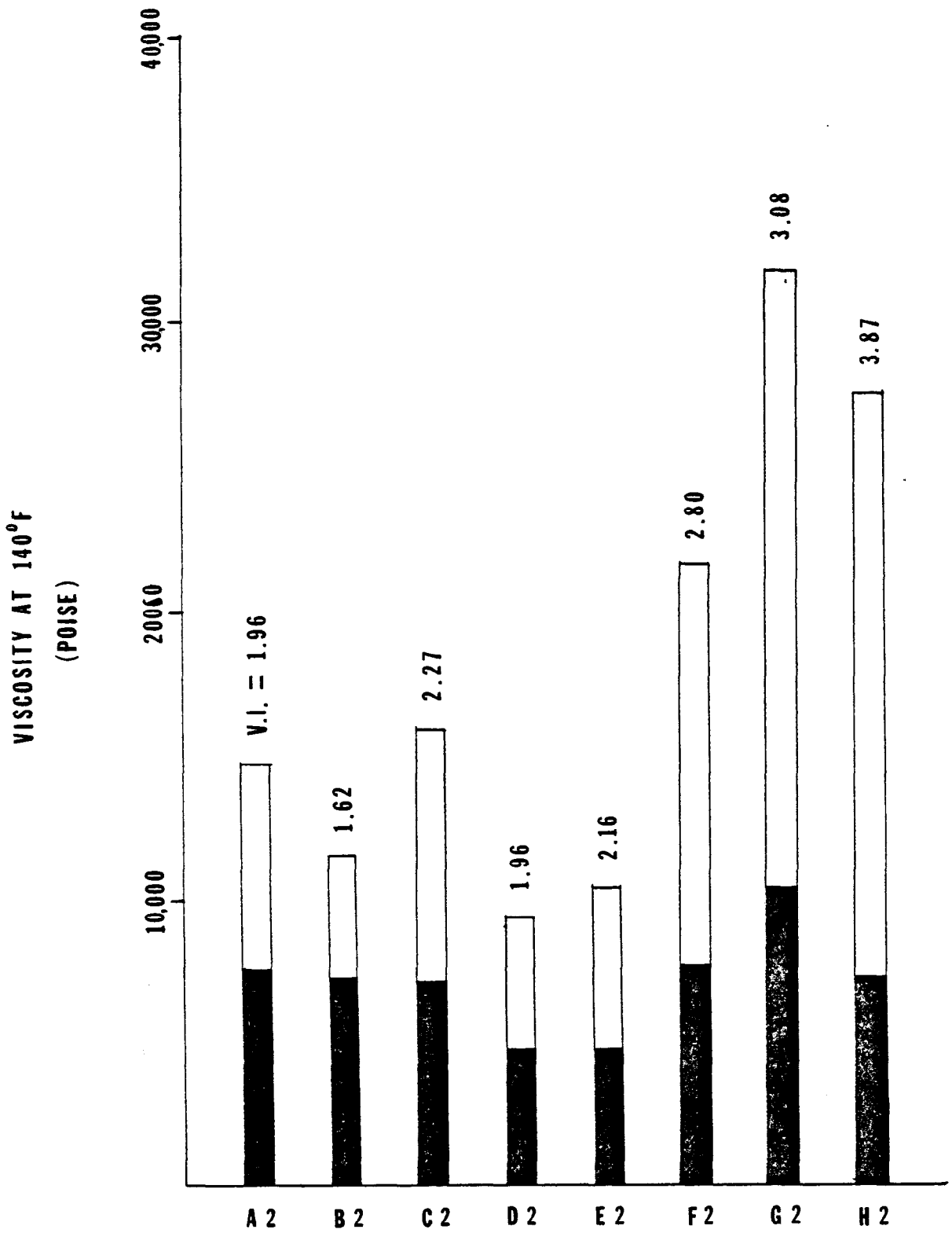
Log-Log  $V = a + bp$  Correlation Graphic

FIGURE 4



REJUVENATED ASPHALT  
 Viscosity Indices for Rejuvenated Exxon -  
 First Oxidation State

FIGURE 5

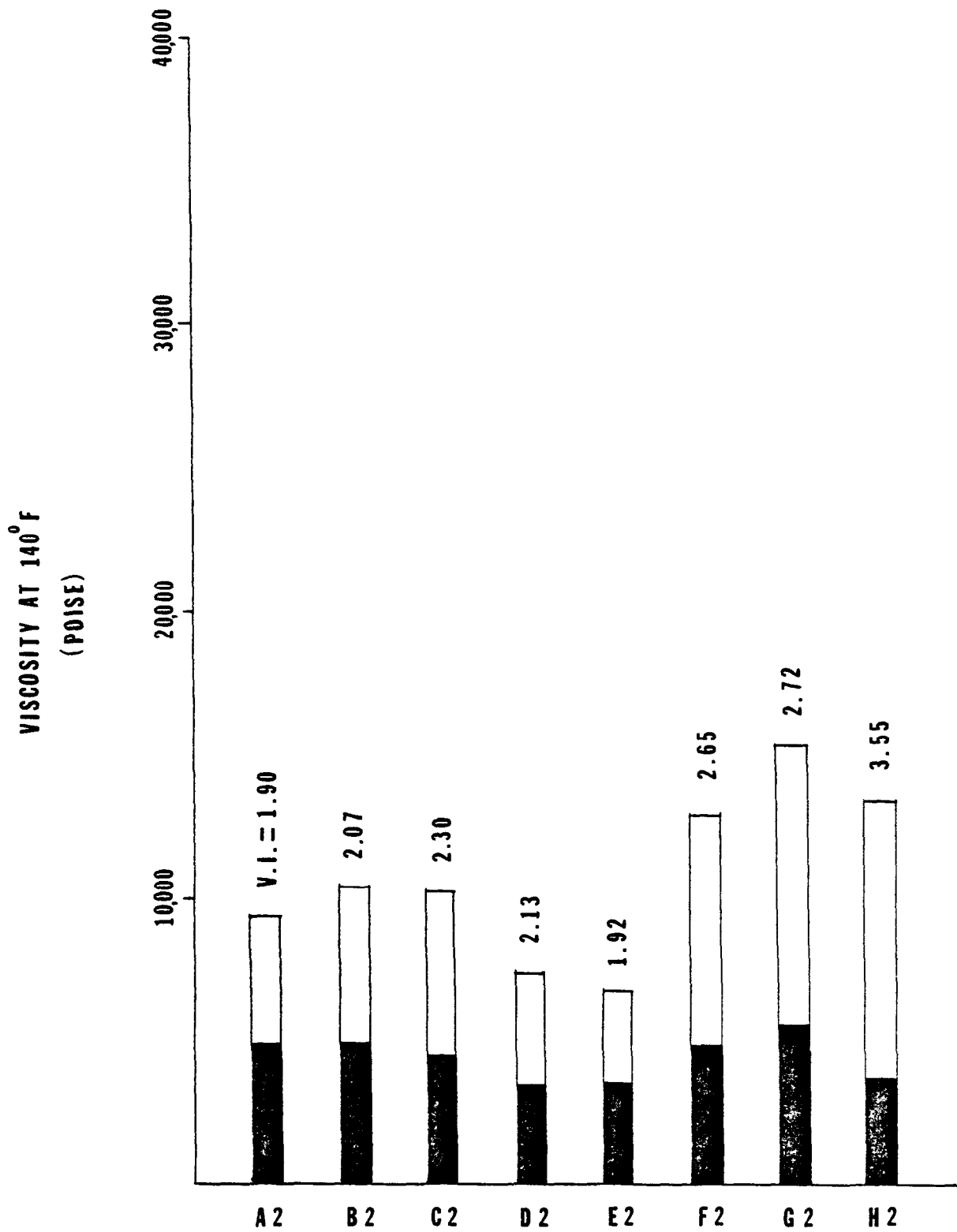


**REJUVENATED ASPHALT**

*Viscosity Indices for Rejuvenated Exxon -  
Second Oxidation State*

FIGURE 6

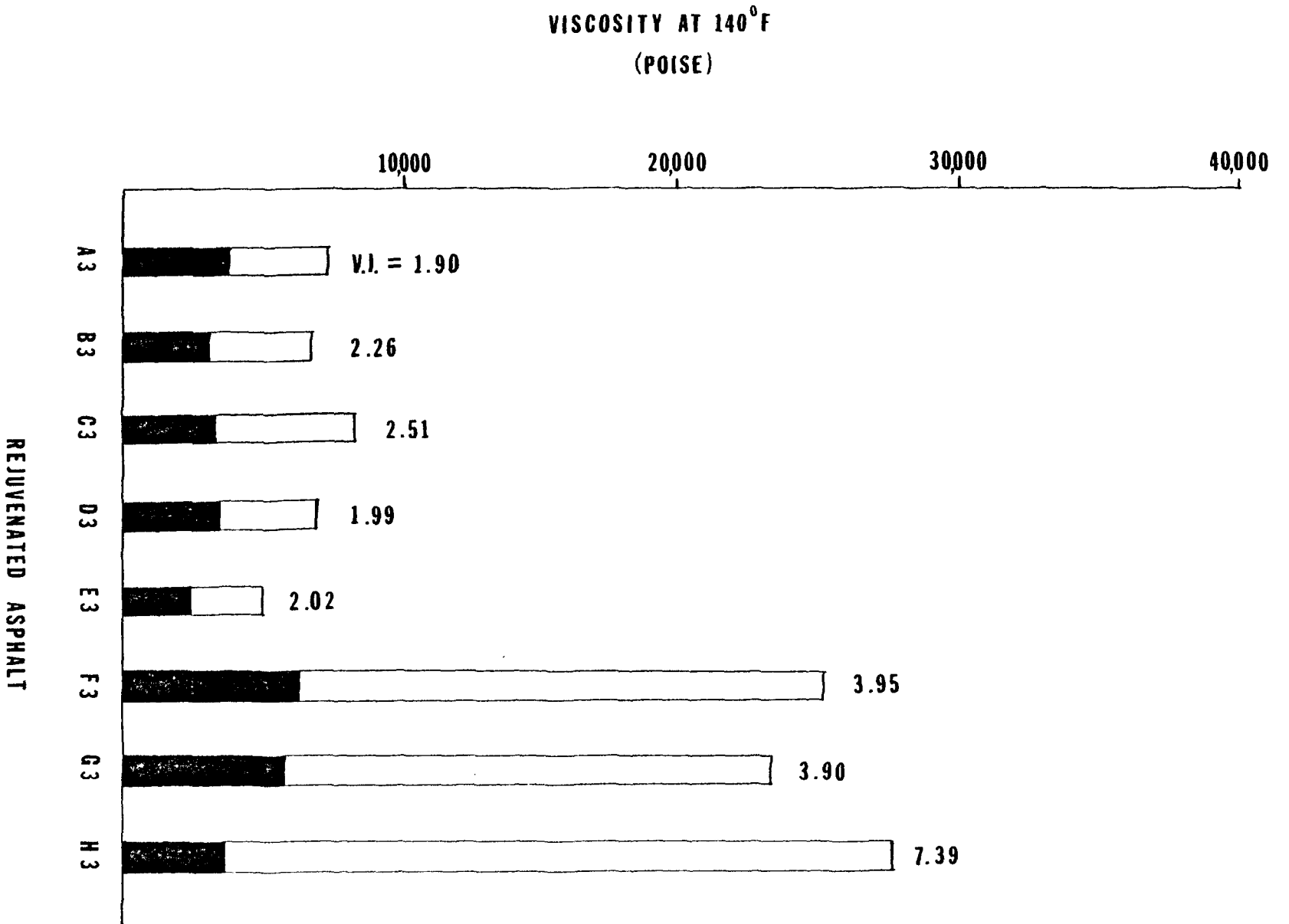




**REJUVENATED ASPHALT**

*Viscosity Indices for Rejuvenated Lion -  
First Oxidation State*

FIGURE 7



*Viscosity Indices for Rejuvenated Lion -  
Second Oxidation State*

FIGURE 8