



**ARIZONA DEPARTMENT OF TRANSPORTATION**

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# **CHEMICAL AND PHYSICAL PROPERTIES OF ASPHALT-RUBBER**

**Phase II - Product Specifications and Test Procedures**

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CHEMICAL AND PHYSICAL PROPERTIES  
OF ASPHALT - RUBBER MIXTURES - PHASE II  
PRODUCT SPECIFICATIONS AND TEST PROCEDURES

by

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## ABSTRACT

Asphalt-rubber was tested with conventional and non-conventional methods to begin development of testing methods for specification purposes and to find methods for studying the reaction between asphalt and reclaimed crumb rubber.

A single asphalt-rubber formulation consisting of 75 percent AR-1000 asphalt and 25 percent crumb rubber from automobile tire treads (sized from No. 16 to No. 25) was used. Independent variables were three levels of reaction temperature [350, 375, and 400 F (177, 191, and 204 C)], and three levels of reaction time (0.5, 1.0 and 2.0 hours).

Dependent variables included ring and ball softening point, absolute viscosity by capillary viscometer at 140 F (60 C), ductility and force-ductility at 39.2 F (4 C), elastic rebound, and low temperature viscosity with the Schweyer rheometer at 39.2 F (4 C).

Force-ductility was the only single test that detected the effect of reaction time and temperature. Variability of results was several times greater than is experienced for conventional asphalt cements and strongly suggests that conventional asphalt tests and criteria should not be used to specify this asphalt-rubber formulation.

Recommendations are made for new tests, modifications of conventional tests and improved laboratory mixing and formulation techniques. An additional experiment is discussed that will use improved techniques to evaluate properties of other formulations, compare properties of field and laboratory produced mixtures, and to compare certain mixture properties with performance of experimental field test sections.

1.0 PROJECT DESCRIPTION

1.1 This project had the following objectives:

- A. To evaluate the problems encountered when asphalt-rubber mixtures are tested with current asphalt test methods and to determine what changes in equipment and/or procedures would be necessary to test these mixtures with present apparatus.
- B. To develop new test methods to specifically measure the unique viscoelastic properties of asphalt-rubber mixtures, including the evaluation of new apparatus commercially available which show capabilities of testing asphalt-rubber mixtures.
- C. To develop asphalt-rubber performance specifications for highway applications using procedures developed in this study.
- D. To develop requirements as needed, to control those manufacturing, transportation and application procedures found to be critical to the final properties of the asphalt-rubber mixture.

1.2 Considerable success was realized for items A and B. Items C and D were not as successful but a good deal of useful information was generated. Each item will be considered individually and will be discussed in detail later in the report.

1.3 A series of laboratory experiments were conducted to find the effect of production variables on properties

of asphalt-rubber. Scope of the work limited the production variables to a single asphalt type, a single rubber type and gradation, three levels of reaction temperature, and three levels of reaction time.

1.3.1 Material properties measured as part of the study include:

- Ring and Ball Softening Point
- Absolute Viscosity
- Ductility
- Force-Ductility
- Elastic Rebound
- Rheologic properties (apparent viscosity and shear rate sensitivity) by the Schweyer Rheometer.

1.4 This study measured properties outlined above as part of a full factorial experiment with three levels of independent variables (time and temperature). The reason for using these independent variables was two-fold. First, of the four major production variables of asphalt type, rubber type, reaction temperature and reaction time; the latter two are considered to be the most probable to be subjected to uncontrolled variability due to job conditions. Secondly, the scope of the project was not large enough to consider the many possible combinations of asphalt and rubber types that are available. This was of special importance in view of the uncertainty of the validity of conventional

asphalt tests and procedures when used for asphalt-rubber mixtures. Furthermore, field experience has shown that both temperature and holding time affect the asphalt-rubber reactions and, subjectively at least, affect properties and performance of the material. These effects include breakdown of rubber with consequent reduction of viscosity and the addition of excessive oil fractions to the base asphalt. This results in reduced adhesion and durability. This study examined the sensitivity of several tests to detect changes in material properties resulting from variations in reaction time and temperature.

## 2.0 ASPHALT-RUBBER REACTIONS AND MECHANISMS

2.1 The reaction between asphalt and rubber has been studied and reported by Green and Tolonen (13). A generalization of the process, while far from being fully understood, is a combined chemical and physical reaction that produces a two phase composite material.

2.1.1 The original asphalt-rubber as developed by McDonald (5) used approximately 75 percent of relatively low viscosity (AR-1000) asphalt cement and 25 percent reclaimed rubber. Source of the rubber was reclaimed tread from conventional automobile tires that was pulverized and graded to a single size (No. 16 to No. 25 sieve). Rubber and asphalt are reacted in a single batch operation before incorporation into construction work. For this process, the finished material consists of readily visible rubber particles in a bituminous binder.

2.1.2 More recently, asphalt-rubber systems have been developed that use different types and sizes of rubber, different reclaiming methods that utilize the full tire carcass, different base bituminous materials, and certain additives. This study considered the system briefly described in the previous section. Properties of asphalt-rubber systems more recently developed are considered in another study that is presently being conducted by the Arizona Department of Transportation.

2.2 Physically, during the reaction, rubber particles in hot asphalt swell to two to three times their original volume (5). These swollen particles cause an enormous

increase in mass viscosity of the system as compared to conventional asphalt or to unswollen rubber and asphalt early in the reaction process.

2.3 While not fully proven, it is hypothesized that rubber and asphalt participate in an exchange of components (13). Automobile rubber contains vulcanized rubber and significant amounts of extender oils that can be characterized by the Rostler parameters (14). The quantity of these oils is, of course, dependent on the particular rubber formulation and the extent of weathering of the rubber before reclaiming or recycling. New, unused tires will contain more oil fractions than tires that have extended exposure to the service environment.

2.3.1 Asphalt also contains components with more or less the same Rostler characteristics as the extender oils of rubber. It is, therefore, hypothesized that under the influence of elevated temperature and/or time, that components can react or be exchanged from rubber to asphalt or vice versa. Laboratory studies have shown that an original chunky appearing asphalt-rubber with high mass viscosity will, with heat and time, degenerate to the point where rubber particles become "dissolved" and a more homogeneous material with a substantially reduced mass viscosity is produced.

This aspect has some connotations for field performance inasmuch as asphalts with certain component combinations could, perhaps, have some of the peptizing fractions unbalanced because of reacting with rubber components with consequent asphaltene precipitation

with reduced system durability. Needless to say, a good deal more work needs to be done on fundamental aspects of asphalt-rubber chemistry in order to produce a balanced composition with predictable performance.

- 2.4 Parameters (independent variables) for this study were selected to include levels that reflect service extremes of reaction temperature and holding times. Response tests were selected for the purposes of determining the effect of reaction time and temperature on physical properties as well as to examine test variability and sensitivity to these changes.

### 3.0 MATERIALS

#### 3.1 Rubber

3.1.1 Rubber utilized in this study was processed by Atlos Rubber Reclaiming, (Los Angeles, California). The material consisted of ground (crumb) rubber produced by mechanically grinding passenger car tread. No additional processing or depolymerization was used on the ground rubber product to alter material properties prior to mixing with asphalt. However, the introduction of no more than 4 percent calcium carbonate powder to the crumb rubber prior to packaging is permitted to prevent the rubber particles from sticking together during shipment. The Atlos designation for the crumb rubber product is TP044 which is a ground rubber produced as described above having a gradation conforming to the limits shown below:

| <u>Sieve</u> | <u>Percent Passing</u> |
|--------------|------------------------|
| No. 16       | 95-100                 |
| No. 25       | 0-10                   |

3.1.2 Texture and shape of the individual rubber particles appears smooth and cylindrical when viewed without magnification. However, with magnification, the notion of smooth cylindrical shapes vanishes. With magnification, an irregular surface texture appears with particles that are longer than they are wide. Processes used during reclaiming are responsible for texture. Magnification also reveals what appears to be particles of calcium carbonate used during packaging.

## 3.2 Asphalt

3.2.1 AR-1000 viscosity graded asphalt cement was used to produce the asphalt-rubber mixtures. Asphalt was supplied by Sahuaro Petroleum and Asphalt Company, Phoenix, Arizona.

3.2.2 Physical properties of asphalt were measured in accordance with the test procedures described by The Asphalt Institute in the Pacific Coast Division (PCD-7) Specifications. All tests in the characterization process were performed at the beginning of the study and also at the end. These results can be compared by referring to the tabulated results in Table 3-1. The characterization process was conducted twice to identify any asphalt property changes which may have occurred during storage.

## 3.3 Laboratory Preparation

3.3.1 Rubber was delivered to the ETL laboratory in six (6), fifty (50) pound paper bags. All the rubber was thoroughly mixed prior to storing in 30 gallon polyethylene drums. Additional mixing of the rubber immediately prior to making each asphalt-rubber batch was conducted to insure that a representative sample was obtained.

3.3.2 AR-1000 asphalt was pumped directly from the Sahuaro Asphalt storage tank into a 55-gallon drum, filling it to approximately 90 percent capacity. Retrieval of asphalt for testing was accomplished, without applying heat, by pumping compressed air into the drum forcing the AR-1000 out the opened drain spout.

Retrieval of the asphalt using compressed air required several hours due to the relative high viscosity of the AR-1000 at 77 F. Therefore, several six liter quantities were obtained in this way and frozen and stored at approximately 0 F (-18 C). The asphalt needed for each asphalt-rubber batch was then obtained by pulverizing frozen AR-1000 with a chisel in the six liter can and removing the necessary quantity of fractured material required for mixing with rubber.

Table 3-1

ASPHALT CHARACTERIZATION

| <u>Test</u>                    | <u>AR-1000</u> | <u>May, 1978</u> | <u>AR-1000</u> | <u>Aug., 1978</u> |
|--------------------------------|----------------|------------------|----------------|-------------------|
|                                | <u>AR-1000</u> | <u>RTFO 1000</u> | <u>AR-1000</u> | <u>RTFO 1000</u>  |
| Pen<br>(Std)                   | 134            | 86               | 138            | 94                |
| Abs. Vis., P<br>(140 F, 30 cm) | 613            | 1280             | 642            | 1166              |
| Kin. Vis., cst<br>(275 F)      | 159            | 230              | 155            | 215               |
| Duct., cm<br>(Std)             | 150+           | --               | 134+           | 134+              |
| Solub, %<br>(TCE)              | 99.6           | 99.8             | 99.7           | 99.2              |
| Softening<br>Pt., F            | 104            | 113              | --             | --                |
| Specific Gravity               | --             | 1.0155           | --             | --                |

#### 4.0 SPECIMEN PREPARATION

- 4.1 Specimen testing and preparation were based on complete randomization of all elements of the factorial including replications.
- 4.2 In order to facilitate testing, mixing, and fabrication, it was decided to mix batches of sufficient asphalt-rubber to provide material for an entire replication of all tests. After the mixture was prepared, it was frozen at 0 F (-18 C) and remained frozen until testing.
- 4.2.1 Statistical analysis of force-ductility, absolute viscosity, and ring and ball softening point showed no significant effects of freezing for periods of up to 14 days.
- 4.3 Mixing was accomplished in an open 3000 ml. stainless steel beaker with a three bladed propeller (3 inch, 45° pitch) turning at a constant speed of approximately 750 rpm. Mixer motor speed was controlled by a power-stat to maintain a constant voltage. Heat was applied by a Meeker burner and was controlled by a gas flow-meter. Temperature was monitored by an armored immersion thermometer. A view of the mixer is shown in Figure 1.
- 4.4 Mixing procedure was as follows:
- 4.4.1 Weigh 750 grams of asphalt into the beaker. Preweigh 250 grams of rubber.
- 4.4.2 Heat asphalt with thermometer placed  $\frac{1}{4}$  inch from bottom and  $\frac{1}{2}$  inch from side of beaker.

- 4.4.3 Manually agitate asphalt to prevent local overheating. Begin stirring with propeller as soon as possible. Maintain constant propeller speed by adjusting voltage to 115 volts with Powerstat.
- 4.4.4 When temperature is 25F below prescribed mixing temperature, lower gas flow and stabilize without overshoot. A maximum of five minutes is allowed to stabilize mixing temperature.
- 4.4.5 When temperature has stabilized, add rubber to hot asphalt. Rubber is at room temperature and is added within five seconds.
- 4.4.6 The mixture temperature will drop to approximately 50F (28.8C) below prescribed mixing temperature. This will occur in approximately five minutes. When temperature begins to rise, increase gas flow and propeller speed. In approximately 30 minutes, the temperature will stabilize at the prescribed mixing temperature. At this point, begin timing for prescribed holding time.
- 4.4.7 Begin manually scraping asphalt-rubber from sides of beaker, continue scraping for prescribed holding time.
- 4.4.8 At conclusion of holding period, remove burner and propeller and transfer mixture with heated spoon to five 8-ounce penetration tins.
- 4.4.9 Allow tins to cool for 60 minutes, mark with cell and replication designation and place in freezer at 0 F (-18 C).

## 5.0 EXPERIMENT DESIGN

5.1 The experiment was designed as a completely randomized full factorial with two replications. Analysis of data was by conventional two way analysis of variance and by the Newman-Keuls multiple range test when independent variables were found to be statistically significant (15).

5.2 Fixed factor model for analysis is as follows:

$$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon_{(ij)k}$$

Where:

$Y_{ijk}$  = Response of material to the  $i^{\text{th}}$  temperature and  $j^{\text{th}}$  holding time.

$\mu$  = Effect on response of the overall mean.

$T_i$  = Effect on response of the  $i^{\text{th}}$  temperature (fixed).

$H_j$  = Effect on response of the  $j^{\text{th}}$  holding time (fixed).

$(TH)_{ij}$  = Effect on response of the interaction of the  $i^{\text{th}}$  temperature and  $j^{\text{th}}$  holding time.

$\epsilon_{(ij)k}$  = Experimental error (random).

5.3 Levels of independent variables:

5.3.1 Temperature at three levels of 350, 375 and 400F (177, 191, and 204C).

5.3.2 Holding times at three levels of 0.5, 1.0 and 2.0 hours.

5.3.3 Factorial layout and cell designations are shown in Table 5-1.

Table 5-1  
FACTORIAL AND CELL DESIGNATIONS

|                   |            | TEMPERATURE ( $T_i$ ) |            |            |
|-------------------|------------|-----------------------|------------|------------|
|                   |            | i=1<br>350            | i=2<br>375 | i=3<br>400 |
| TIME<br>( $H_j$ ) | j=1<br>0.5 | 1,1                   | 2,1        | 3,1        |
|                   | j=2<br>1.0 | 1,2                   | 2,2        | 3,2        |
|                   | j=3<br>2.0 | 1,3                   | 2,3        | 3,3        |

5.4 Dependent (response) variables are briefly described in Section 1.3.1.

Additional information on the test and modifications to standard test methods is included in the specific section dealing with the particular property measured.

## 6.0 RING AND BALL SOFTENING POINT

- 6.1 Testing was performed in accordance with ASTM D36-76 (part 15).
- 6.2 Considerable difficulty was encountered in performing the test because of the nonhomogeneous nature of the asphalt rubber mixture and the relatively small size of the specimen used for the test. Testing of conventional asphalt cement results in the ball and asphalt sinking to the bottom plate. Asphalt rubber, on the other hand, forms a structural "basket" that usually breaks in the side allowing the ball to fall from the mixture. Figure 2 shows the completion of a typical test.
- 6.3 Preliminary testing showed that for cases where the ball did not fall through the side, softening points were essentially the same as for the more usual occurrence of a side break in the structural basket.
- 6.4 Data obtained for softening point testing are shown in Table A-1 (Appendix A).
- 6.4.1 Overall mean for all data is 158F (70C). Softening point for unaged asphalt is 104F (40C) and for the asphalt after aging in the rolling thin film circulating oven is 113F (45C).
- 6.4.2 ASTM D36 states that values obtained for each ball should not vary by more than 1C (1.8F). The precision statement requires replicate tests (average of left and right specimens constitutes a single test) should not vary by more than 1C (1.8F).

- 6.4.3 The top data matrix of Table A-1 (all data) shows that three runs (350F, 0.5 hr., replicate 2), (400F, 0.5 hr., replicate 1) and (400F, 2.0 hr., replicate 1) exceed the limitation of 1.8F between sides.
- 6.4.4 Table A-4 is data using the mean of left and right sides as a single test. All data exceed the precision requirement of ASTM D36.
- 6.4.5 In view of the relatively low variability "between sides" (or "within tests"), the cause for the large variability between tests can be attributed to material nonhomogeneity, sample size, sample preparation (ring filling), or some combination.
- 6.5 Analysis of Variance (ANOVA) for all data (right and left sides not averaged) is shown in Tables A-2 and A-3. Table A-3 shows that no factors of the model (temperature, holding time or interaction) are significant in affecting softening point of asphalt-rubber mixtures.
- 6.5.1 When the mean of left and right sides are used as a single test, the same conclusion of the significant factors is drawn. Analysis of these data are in Tables A-4 through A-6. Plots of softening point versus holding time for each reaction temperature are shown on Figure A-1. This plot shows means but not variability.
- 6.5.1.1 Some discussion regarding Table A-6 F values is warranted inasmuch as in all cases, calculated F values are less than unity. It is generally accepted that several circumstances can cause low calculated F values. Some possibilities are:

- A. Some uncontrolled factor may have affected the mean square error term.
- B. Proper experimental randomization may not have been employed.
- C. Insufficient number of measurements may not establish normality as was assumed for use of the model and calculations.
- D. The assumption of homogeneous variance has been violated.

6.5.1.2 In reviewing the above possibilities, the following may point to improved future testing and evaluations:

- A. Temperature and time factors were held constant (see Section 4, "Specimen Preparation"). It is quite possible that nonhomogeneity of material and relatively small specimen size (D36 rings) may be the uncontrolled factor responsible.
- B. Since the experiment was completely randomized, operator bias, learning curve effects, or equipment bias are probably not causes.
- C. Insufficient measurements may be a cause. If additional work is undertaken, increased replications, rejection of data that does not comply with D36 precision requirements, and modifying the apparatus may improve precision and reliability of the test.
- D. The assumption of homogeneous variance was not

violated in the analysis. Table A-5 shows results of the Foster and Burr q test for homogeneity of variance as described by Anderson and McLean (15). This check and appropriate transformation of data, if necessary, were standard analysis procedure for this study.

## 6.6 Conclusions

- 6.6.1 This experiment suggests that asphalt-rubber provides a substantially higher softening point material than does the conventional base asphalt. The mean value of softening point of 158F (70C) approaches maximum service temperatures for many Arizona pavements.
- 6.6.2 Variability of asphalt-rubber, as probably should be expected, is greater than conventional asphalt cement. If softening point is going to be used, additional work should be done to reduce or at least evaluate and set reasonable limits on variability. Steps that should be taken include increased replication testing, modification of ring and ball geometry to accommodate rubber particles, or the development of a flow index test procedure as suggested by L. C. Krchma (18).
- 6.6.3 The experiment did not detect any differences in softening point due to reaction temperature or reaction time. If the assumption (based on field experience) that time and temperature have an effect is considered, it can be concluded that the conventional test is not sufficiently sensitive or that softening point is, in fact, not affected.

6.6.3.1 Additional work and experimentation could resolve the question. However, in view of the usefulness of softening point for paving purposes, the expenditure probably cannot be justified.

7.0 ABSOLUTE VISCOSITY AT 140F (60C)

7.1 The standard method of ASTM D2171-66 (Part 15) was modified as outlined by Green and Tolonen (13). Modifications of the procedure include:

- Use of a large bore capillary viscometer (Asphalt Institute No. 400) to accommodate rubber particles.
- Reduction of applied vacuum from the standard 30 cm. Hg. to 10 cm. to produce acceptable flow times.

7.2 Use of reduced vacuum required a correction of tube constants that are provided by the manufacturer. The correction is based on the following:

$$K = \frac{M_H}{(H-h)}$$

Where:

K = Instrument constant in poises/sec. per cm. Hg.

$M_H$  = Multiplier constant for vacuum head of test (or calibration).

H = Applied vacuum head (cm. Hg.).

h = Average liquid head (cm. Hg.). Provided by manufacturer.

7.2.1 Viscosity is, of course, flow time in seconds, t, multiplied by the constant,  $M_H$ .

7.2.2 As an example for the tube no. 12-X522, (bulb B):

$$M_H = K (H-h) = 18.30 \text{ (Mfgs. data)}$$

$$H = 30 \text{ cm. Hg.}$$

$$h = 0.24 \text{ (Mfgs. data)}$$

$$K = \frac{M_H}{(H-h)} = \frac{18.30}{(30-0.24)} = 0.6149$$

For 10 cm. Hg., the multiplier constant,  $M_H$ , is:

$$M_{10} = K (H-h) = 0.6149 (10-0.24) = 6.00$$

7.2.3 A series of tests were undertaken using asphalt cement at the two levels of vacuum. Analysis showed no significant difference in viscosity due to the vacuum head, hence the concept of multiplier correction was considered valid and was used for viscosity experiments with asphalt-rubber.

7.3 Viscometer tubes were loaded by means of a syringe consisting of a glass tube with a wood plunger. The syringe tube outside diameter is such that it loosely fits inside the viscometer tube and the wood plunger, of course, snugly fits inside the syringe tube.

7.3.1 Procedure for filling the viscometer tube is as follows:

7.3.1.1 Asphalt-rubber is heated to 275F (135C) on a hotplate.

7.3.1.2 Viscometer, viscometer clamp stand, syringe tube, and plunger are preheated in an oven to 275F.

7.3.1.3 When the asphalt-rubber mixture reaches 275F, the syringe is loaded by suction from the wood plunger.

7.3.1.4 Transfer from the syringe to the viscometer is by the plunger forcing material from the syringe tube while the tube is being withdrawn from the viscometer. This operation takes place in the hood at room temperature.

7.3.1.5 Immediately after filling, the viscometer is placed in the 140F bath. After 60 minutes in the bath, temperature equilibrium is assumed and the test is conducted.

7.4 Data for viscosity tests are shown in Table B-1. Entries (poises) shown are averages of six timing marks for each viscometer section for each replication.

7.4.1 Overall mean for all data is 146,267 poises. Viscosity of unaged base asphalt is 613 poises and is 1280 poises for the asphalt after aging in the rolling thin film circulating oven.

7.4.2 ASTM D2171 does not specify a precision for values obtained between different sets of timing marks for a single tube but experience indicates that variability not exceeding 5 to 10 percent should be expected for conventional asphalt cement. The mean difference for this experiment is about 25 percent. This increased variability of asphalt-rubber is about the same as was observed in softening point testing.

7.4.2.1 D2171 repeatability precision statement requires that two values must agree to within 7 percent

of their mean. If data of Table B-1 is used to compute a test value based on the mean of the two readings from a single tube, none of the data will comply with the repeatability requirement for asphalt cements.

- 7.4.2.2 One source of this variability could be that a clean meniscus does not form at the surface as is the case for asphalt cement. This is shown on Figure 3. Timing between marks is difficult because the shape of the surface plane does not remain constant between marks. Another source of variability is, undoubtedly, related to material nonhomogeneity resulting from methods that were used to charge asphalt-rubber into the viscometer.
- 7.4.3 Logarithmic transformation of data was necessary to comply with the homogeneity of variance requirement for analysis of variance. Transformed data are shown in Table B-3. The analysis is shown in Table B-5. Analysis of variance shows holding time to be significant at the 0.05 but not at the 0.01 level. Temperature and interaction were not significant.
- 7.4.3.1 A Newman-Kuels test for multiple contrasts was conducted in an attempt to determine which reaction time(s) were responsible for an optimum (or pessimum) viscosity, but this analysis (Table B-7) shows no significant differences.
- 7.4.3.2 Figure B-1 is a plot of holding time versus transformed viscosity and Figure B-2 is a plot of mixing temperature versus transformed viscosity. Both plots are of mean cell values and do not, for

clarity, show variability. Both of these plots suggest that holding time may have an effect and that viscosity may maximize at about one hour.

7.4.4 Another analysis was made using transformed data where the replicate measurement is the mean viscosity based on the two timing marks of a single tube. These data are shown in Table B-8. Analysis of variance is shown in Table B-10. For this analysis no factor contributes significantly to a viscosity effect.

7.4.4.1 This result is probably a reflection of the variability of the test as well as some masking of information as a result of the logarithmic transformation and averaging process. This conclusion is additionally reinforced when it is remembered that the analysis of all data showed time significance at the 0.05 level but not at the 0.01.

## 7.5 Conclusions

7.5.1 Asphalt rubber exhibits viscosities at 140F (60C) approximately two orders of magnitude greater than the base asphalt used for the mixture.

7.5.2 Variability of asphalt rubber greatly exceeds that of conventional asphalt cement. Sources of variability are of concern because of the effect of mass viscosity on production and application. Viscosity affects mixing, pumping, nozzle size and application temperature. Theory and experience shows that viscosity is an indicator of the extent of reaction between asphalt and reclaimed rubber.

7.5.2.1 Sources of variability in viscosity measurement using capillary viscometers includes:

- Nonuniformity of material (asphalt and rubber separation in the tube) due to the method of loading or charging the viscometer.
- Uneven surface of the meniscus.
- Extremely long flow times between timing marks. Flow times in this study were on the order of 30 minutes whereas the standard method for asphalt cement (D2171) suggests times much shorter.
- Shear rates that are not constant along the length of the tube. See Section 7.6.

7.5.2.1.1 Laboratory experience obtained as part of this study indicates capillary viscometry with tube sizes used (The Asphalt Institute No. 400) are not adequate for asphalt-rubber from either a precision nor a time economy point of view. There are several alternative systems that should be considered for further study. These include:

- Vacuum capillary viscometers with bore sizes considerably larger than those used for this study. A No. 16 sieve size rubber particle will have a length of up to 3.5 mm. If the rule of bore diameter of 3 times the particle size to prevent interference is accepted, a minimum bore size of about 11 mm. (0.4 in.) will be necessary.

- Falling coaxial viscometers of the type described by Frobel, Jimenez, and Cluff (10).
- Rotating vane viscometers are a possibility. If properly calibrated, these devices could be incorporated into the laboratory mixer and could monitor the rate of reaction.

7.5.3 This experiment did not adequately detect a difference in viscosity of finished materials due to mixing temperature but did suggest that holding (reaction) time may have an effect. This experiment shows that viscosity, as measured in this part of the study, is more sensitive to the effect of holding time than is softening point, but is not more sensitive to the effect of mixing (reaction) temperature.

7.5.3.1 At this point of the study, the question of material changes due to reaction time and temperature are unresolved. Based on the variability of measured values and on field experience, a reasonable conclusion is that test sensitivity deserves additional study and that other properties should be considered.

## 7.6 Discussion

7.6.1 Since the test was intended to be run using as near "conventional" techniques as possible, corrections were not made to account for shear rates that were not  $0.05 \text{ sec.}^{-1}$ . This correction, or the lack of it, could, of course, affect results.

7.6.1.1 A study is presently underway, in conjunction with development of another mixing technique, that will

evaluate shear rate effects of asphalt-rubber in capillary viscometers. A separate report of these findings will be prepared and presented in the near future.

8.0 DUCTILITY AT 39.2F (4.0C)

8.1 Ductility values were determined according to ASTM D113-77 at a testing speed of 1 cm. per min.

8.2 Data obtained for ductility are shown in Table C-1.

8.2.1 Overall mean for all data is 17.0 cm. Ductility for the unaged asphalt used for the mixture was 100+cm. and for the asphalt after aging in the rolling thin film circulating oven, the measured value is 100+cm. It should be noted that when aged base asphalt cement is tested at 5 cm. per min. that ductility values drop significantly. Unaged asphalts retain the 100+cm. values. Beaker aged asphalts showed values from 2 cm. to 41 cm. and rolling thin film oven residues produce ductilities of about 6 cm.

8.2.2 Logarithmic transformation of data was necessary to comply with the homogeneity of variance requirement for analysis of variance. Transformed data are shown in Table C-3. The analysis is shown in Table C-5.

8.2.2.1 For the case of all data, mixing temperature is highly significant; holding time and the interaction are not significant.

8.2.2.2 Newman-Keuls test for multiple contrasts were run on the mean of all data with mixing temperature as the single factor. This analysis (Table C-8) shows ductility for a mixing temperature of 400F is significantly greater than 350F but not greater than 375F and ductility for a mixing temperature of 375F is significantly greater than 350F. Stated

another way, ductility of materials mixed at 350F are lower than those mixed at 375 or 400F and there is no significant difference between 375 and 400F. This effect is shown graphically on Figures C-1 and C-2 where transformed ductility is plotted versus temperature and holding times.

8.2.3 Another analysis was made using the mean of three ductility measurements as a single test or replicate. These data are shown in Table C-9.

8.2.3.1 Analysis of these data is shown in Table C-11. Results are essentially the same as were found when all data were used in the analysis, with some information being masked by the averaging process. For this case, temperature is significant at the 0.05 level but not at the 0.01.

### 8.3 Effect of Temperature on Ductility

8.3.1 A report by Jimenez (19) shows that asphalt-rubber ductility is relatively unaffected by temperatures in the range of 77-33F (25-0.6C) as compared to the well known pronounced effect of temperature on the ductility of straight asphalt cement. Jimenez shows ductility of straight asphalt cement (tested at 5 cm. per min.) at 55F (13C) to be 150+cm. and a value of 0 cm. at 33F (0.6C) whereas asphalt-rubber exhibited a constant ductility value of approximately 24 cm. for the temperature of 77, 55 and 35F (25, 13 and 0.6C).

8.3.2 To further consider this property, a small experiment was designed to produce asphalt-rubber using the same mixing times and temperatures as the previous experiment and to test for ductility at 1 cm. per min. at 77F

(25C). The data are shown in Table D-1.

8.3.2.1 Overall mean for all tests was 16 cm. (Overall mean for 39.2F was 17 cm.)

8.3.2.2 Since the experiment was not replicated, two-way analysis of variance could not be used to analyze main effects and interactions. A one-way analysis was made using each combination of time and temperature as a single treatment. This analysis is shown in Table D-2. Analysis of the data shows there is no significant difference due to treatment, hence 77F ductilities are statistically the same regardless of reaction temperature and time combinations.

8.3.2.3 An analysis was made to compare ductility at 77F with ductility at 39.2F. Data from Tables C-1 and D-1 were compared with a t-test of means.

|           | <u>39.2F</u> | <u>77F</u> |
|-----------|--------------|------------|
| $\Sigma$  | 916.0        | 380.5      |
| $\bar{x}$ | 16.96        | 15.85      |
| s         | 2.25         | 2.87       |
| n         | 54           | 24         |

$$F = \frac{8.24}{5.06} = 1.63 \quad (F_{0.05, 53, 23} = 1.88)$$

$$\therefore \sigma_{39.2}^2 = \sigma_{77}^2$$

$$S^2 = \frac{[(54-1)(2.25)^2 + (24-1)(2.87)^2]}{54 + 24 - 2} = 6.02$$

$$s_{\bar{x}_1 - \bar{x}_2}^2 = \frac{(6.02)}{54} + \frac{(6.02)}{24} = 0.36$$

$$t = \frac{16.96 - 15.85}{0.602} = 1.84$$

$$t_{0.975, 76} = 1.99$$

Since the calculated t value of 1.84 is less than the critical value, it is concluded that there is no significant difference between ductilities at 77 and 39.2F.

#### 8.4 Conclusions

- 8.4.1 Ductility of asphalt-rubber at 39.2F (4C) at test rates of 1 cm. per minute is considerably less than aged or unaged asphalt (AR-1000) that was used to prepare the asphalt-rubber mixture.
- 8.4.2 Ductility of asphalt-rubber at 39.2F (4C) at test rates of 1 cm. per minute is considerably more than the ductility of thin film circulating oven aged base asphalt if the base asphalt is tested at 5 cm. per minute.
- 8.4.3 The ductility test is sensitive to the effects of mixing temperature but not sensitive to the effect of reaction (holding) time nor to the interaction between holding time and temperature.
- 8.4.4 Ductility of asphalt-rubber is essentially constant for the temperature range of 77-39.2F (25-4C).

## 9.0 FORCE-DUCTILITY

9.1 This test is a modification of the standard ASTM D113 inasmuch as a force ring is added to the briquet clip in somewhat the same fashion as described by Anderson and Wiley (16). Reason for using this device was to increase the amount of data generated during ductility testing. Strength as well as a measure of energy (by area under the force-elongations curve) to deform or fail the specimen are easily obtained. Furthermore, it has been suggested that, by modifying the cross-section and longitudinal geometry of the specimen to provide a constant cross-section, the method can be used to evaluate creep compliance of asphalt-rubber.

9.1.1 For this study, deformation of a proving ring was measured with a LVDT. LVDT movement was recorded on a timed strip chart. Figures 4 and 5 show the force-ductility set up used for this study.

9.2 Maximum Ductility Load at 39.2F (4C), 1cm. per minute.

9.2.1 Data for the experiment are shown in Table E-1. Forces shown are in Newtons (1 lbf = 4.448222N).

9.2.1.1 Data were transformed by  $x' = \sqrt{B} - \sqrt{B-x}$  to provide homogeneity of variance required for analysis of variance. Analysis is shown in Table E-4.

9.2.1.2 Analysis of these data show that holding time is significant at the 0.05 level but not at the 0.01 level. The interaction term is highly significant which can also be seen by the intersecting or crossing curves of Figure E-1 and E-2.

9.2.2 Since data from other tests is not available, variability comparisons cannot be made.

### 9.2.3 Conclusions

9.2.3.1 Of all tests and experiments described thus far maximum ductility load (and, as will be shown later, force-ductility) are the only methods that are sensitive enough to detect the interaction between reaction time and reaction temperature. This may be important in the production of asphalt-rubber if it can be shown that lower temperature combined with longer holding times will produce the same material as the reverse combination of higher temperature and shorter holding time.

### 9.3 Force-Ductility Energy

9.3.1 Energy is calculated as the area under the curve of elongation versus force expressed in Newton-Metres (N-M). Data for the experiment is shown in Table F-1.

9.3.1.1 Analysis of these data show (as did maximum ductility load) that reaction time and the interaction between reaction time and temperature to be highly significant.

### 9.3.2 Conclusions

9.3.2.1 Force-ductility energy is probably more sensitive to the effect of holding time than is the maximum ductility load because energy is significant at the 0.01 level whereas maximum load is not. The test also suggests that one hour may be the

optimum mixing time, at least 375 and 400F (191 and 204C) as can be seen on Figures F-1 and F-2.

- 9.3.2.2 Based on experience gained during this study, the force-ductility test appears to show promise as an analytical tool and perhaps as a specification test if a correlation can be generated between force-ductility parameters and asphalt-rubber field performance. Furthermore, energy calculations based on area under the load-deformation curve can be related to toughness in much the same manner used for the analysis of metals. This will provide a means of comparing different asphalt-rubber formulations, particularly if performance can be correlated with this property.

## 10.0 Schweyer Rheometer

10.1 The recently available Schweyer rheometer is described as a constant stress rheometer (17) that produces a rheogram of apparent viscosity ( $\eta_a$ ) versus shear rate ( $\dot{\gamma}$ ). A schematic of the device is shown on Figure 10-1 and overviews are shown on Figures 6, 7 and 8.

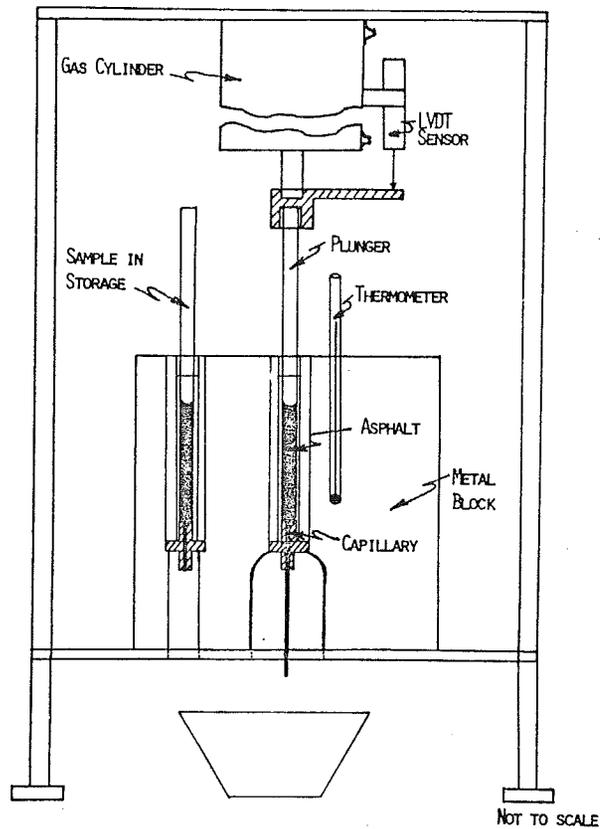


Figure 10-1

Schweyer Rheometer Schematic

10.2 Principal of operation is that a sample of material is forced through a precision capillary by means of a

constant load. Load is applied to the specimen by means of a plunger and downward movement of the plunger is monitored by a LVDT.

- 10.2.1 A timed strip chart is produced that records plunger movement as a function of time. The corrected plunger movement is equated to specimen flow through the capillary tube. Figure 10-2 shows a typical output curve.
- 10.2.2 Movement of the plunger begins as nonlinear with time until equilibrium (constant) flow is established when the curve becomes linear. Velocity measurements are made on the linear portion of the curve.
- 10.2.3 Calculations are straightforward and simple.
  - 10.2.3.1 Pressure on the loading ram is read directly from a gage on front of the instrument. Pressure is supplied by pressurized gaseous nitrogen and can be controlled to suit test conditions and material characteristics.
  - 10.2.3.2 Gage pressure (P) is multiplied by a machine constant (M) to calculate force applied to the specimen.
  - 10.2.3.3 Force is then multiplied by a shear stress tube constant ( $K_{\tau}$ ) to calculate shear stress ( $\tau$ ).
  - 10.2.3.4 Hence:  $\tau = (P \times M) (K_{\tau})$
  - 10.2.3.5 Velocity (V) is read from the linear portion of the recorded curve of movement versus time.

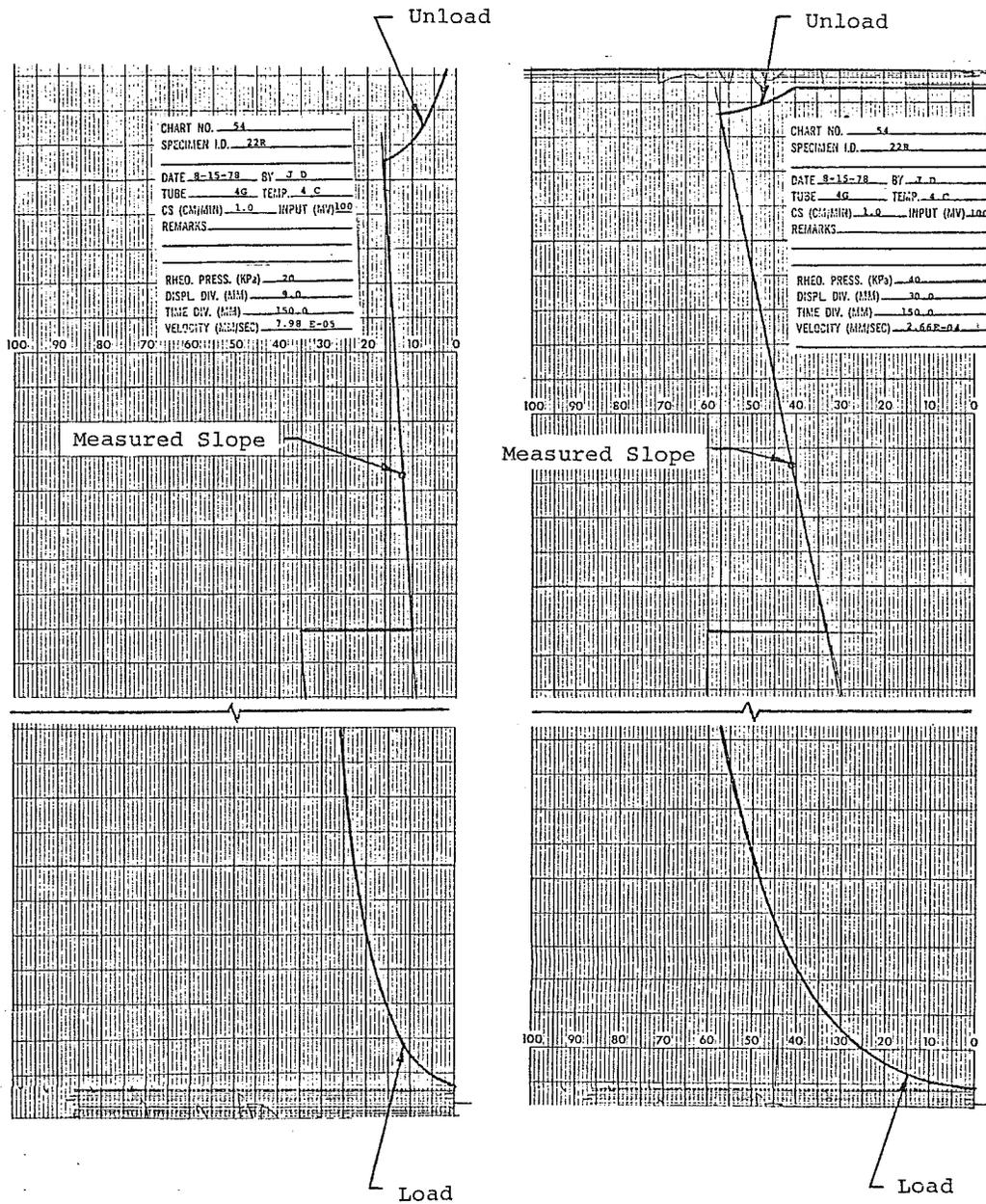


Figure 10-2  
Rheometer Traces

10.2.3.6 Shear rate ( $\dot{\gamma}$ ) is calculated by multiplying velocity by a shear rate tube constant ( $K_T$ ).

10.2.3.7 Hence:  $\dot{\gamma} = VK_{\dot{\gamma}}$

10.2.3.8 Apparent viscosity ( $\eta_a$ ) is given as:

$$\eta_a = \frac{\text{shear stress}}{\text{shear rate}} = \frac{\tau}{\dot{\gamma}}$$

and is expressed in Pascal-seconds (Pa.s)

10.2.3.9 After the test load is removed and the specimen returns to rest equilibrium, a different load is applied and a new apparent viscosity is calculated for the new shear stress and shear rate.

10.2.3.10 Several runs are made and a rheogram is developed that is a plot of log of apparent viscosity ( $\log \eta_a$ ) versus log of shear rate ( $\log \dot{\gamma}$ ) where each point on the curve represents one run. These points form a straight line amenable to linear regression analysis and, theoretically, allow calculation of apparent viscosity for any shear rate. In this study, viscosities are reported for shear rates of 0.05 and 1.0  $\text{sec.}^{-1}$ .

10.2.3.11 A typical rheogram and data used to generate it are shown on Figure 10-3.

10.3 Straight lines on a log scale are presented by the power law equation:

$$y = a x^b$$

or for the rheogram:

SCHWEYER RHEOMETER

IDENTIFICATION 22R

CHART NO. 54 TEMP. 4C TUBE 4G

|                                  |          |          |          |          |          |
|----------------------------------|----------|----------|----------|----------|----------|
| (L), LVDT Chart Dsp., mm         | 4.2      | 9.0      | 13.4     | 17.0     | 30.0     |
| (T), Time Chart Dsp., mm         | 150.0    | 150.0    | 150.0    | 150.0    | 150.0    |
| (CS), Chart Speed, cm/min        | 1        | 1        | 1        | 1        | 1        |
| (P), Pressure, KPa               | 15       | 20       | 25       | 30       | 40       |
| $\tau$ Pa                        | 1.1E04   | 1.4E04   | 1.8E04   | 2.2E04   | 2.9E04   |
| $\dot{\gamma}$ mm/sec            | 3.72E-05 | 7.98E-05 | 1.19E-04 | 1.51E-04 | 2.66E-04 |
| $\dot{\gamma}$ sec <sup>-1</sup> | 3.07E-05 | 6.58E-05 | 9.80E-05 | 1.24E-04 | 2.19E-04 |
| $\eta_a$ Pa-sec                  | 3.5E08   | 2.2E08   | 1.8E08   | 1.8E08   | 1.3E08   |

Regression ( $\eta_a = a\dot{\gamma}^b$ ):  $a = 1.961E06$   $b = -4.956E-01$   $R^2 = 0.982$   
 Rheologic Constants :  $\eta_{a,0.5} = 8.9E06$   $\eta_{1.0} = 2.0E06$   $c = 5.1E-01$

Plotting Points:

|                |         |         |         |
|----------------|---------|---------|---------|
| $\dot{\gamma}$ | 1.0E-05 | 1.0E-04 | 1.0E-03 |
| $\eta_a$       | 5.9E08  | 1.9E08  | 6.1E07  |

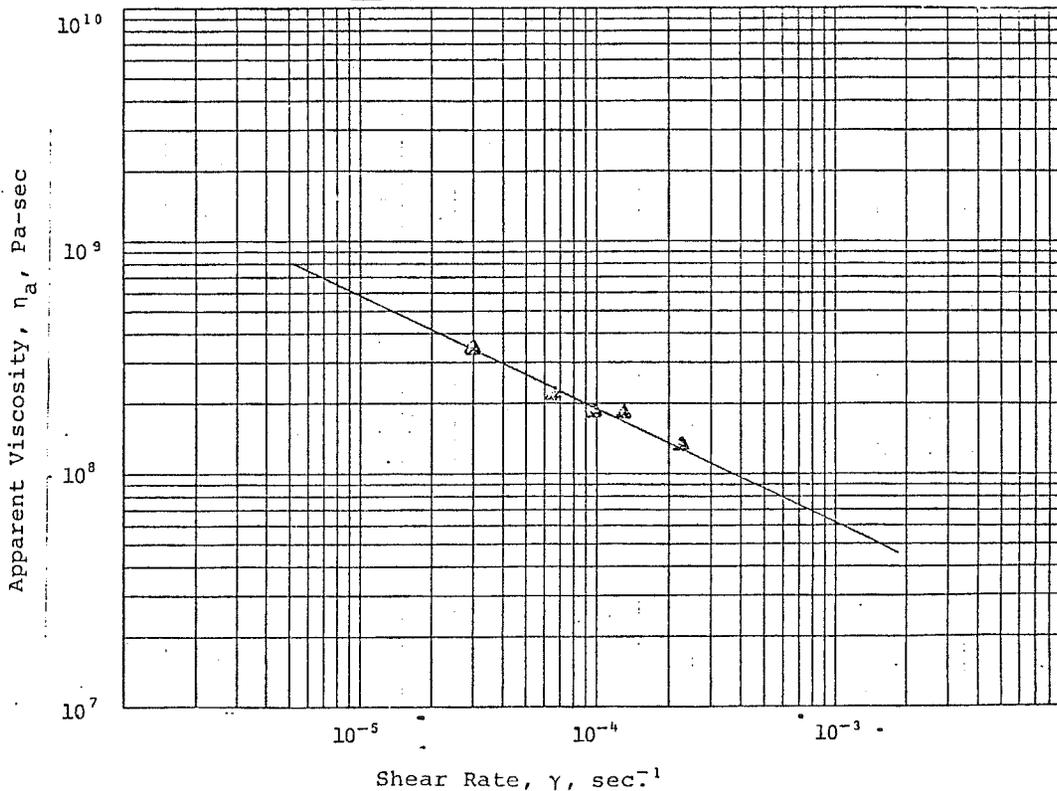


Figure 10-3  
Rheogram

$$\eta_a = a(\dot{\gamma})^b$$

where b is the slope of the straight line of the log plot.

- 10.3.1 Materials with horizontal rheogram plots ( $b = 0$ ) are Newtonian fluids (20) and hence, are not shear susceptible. That is, apparent viscosity is constant over a range of shear rates.
- 10.3.2 Materials with slopes down to the right ( $b < 0$ ) are termed pseudoplastic. These materials are shear susceptible inasmuch as viscosity decreases when shear rate increases. Paint that becomes "thinner" during stirring is an example.
- 10.3.3 Materials with slopes up to the right ( $b > 0$ ) are termed dilatant. Shear susceptibility of these materials is exhibited by increased viscosity with increased shear rate. Certain flocculating agents produce dilatant fluids. This property may be of interest in the asphalt-rubber field since observations have been reported that performance in runway touch-down areas (high shear) is better than in areas where traffic is relatively slow moving. If the observations are valid, this performance is the opposite of what would be expected from pseudoplastic, and probably, Newtonian materials.
- 10.4 Shear susceptibility, then, is a function of the slope of the rheogram. Newtonian materials have zero slope, pseudoplastic materials have negative slopes, and dilatant materials have positive slopes. The rheogram is described by a characteristic apparent viscosity at

a given shear rate (say  $0.05 \text{ sec}^{-1}$ ) and the power law parameters  $a$  and  $b$  (slope).

10.4.1 Shear susceptibility in this study is reported by the parameter  $c$  as described by Schweyer (17). The shear susceptibility index is defined as:

$$c = 1 + \text{slope} = 1 + b$$

10.4.1.1 Newtonian materials ( $b = 0$ ) have a shear susceptibility index of 1.

10.4.1.2 Pseudoplastic materials ( $b < 0$ ) have shear susceptibility indices of less than 1.

10.4.1.3 Dilatant materials have shear susceptibility indices greater than 1.

10.5 Specimen tubes used for the constant stress rheometer (Figure 8) consist of a specimen or flow tube of 9.47 mm. (approximately  $3/8$  in.) diameter. The capillary is threaded onto the end of the flow tube and is much smaller in diameter (down to less than 1 mm.) than the flow tube. Selection of capillary size is based on relative viscosity of the material under test conditions. Thinner or low viscosity materials would, of course, use smaller capillaries.

10.5.1 The pressurized ram or plunger forces the large plug of material down the flow tube (50 mm.) and through the capillary. The capillary controls flow rate and hence shear rate and shear stress. There is some question as to size effects on the capillary on measured viscosity. It should be expected that, for

a given material under constant environmental conditions, different tubes would give the same viscosity if capillary size effects were not present. In the case of asphalt rubber, the question of particle interference arises inasmuch as rubber particles could clog a capillary opening smaller than the rubber particle. If interference exists, measurements would be confounded since, instead of representing asphalt rubber mass viscosity, elasticity of rubber and/or flow of asphalt around rubber particles could be the response.

10.6 To investigate the size effect and to aid in selecting a tube size for the study, a small experiment was conducted on asphalt cement. The data are shown in Table G-1. Rheograms of the two asphalts (stock and aged) for three test temperatures are shown in Figures G-1 through G-9. These figures show the strong possibility of an effect due to capillary diameter on both apparent viscosity and slope of the rheogram (which indirectly indicates shear susceptibility). Discussions with Dr. Scheweyer indicate that this is not necessarily unexpected and that sensitivity to capillary size may, indeed, be characteristic of a given material.

10.6.1 It is possible to use a single capillary for a given combination of material and temperature but not necessarily for all materials nor for a given material at all temperatures. This is because ram movements are excessively fast when relatively low viscosity materials are tested in large bore capillaries and conversely, ram pressures are not great enough to force high viscosity materials through small capillaries in reasonable amounts of time.

10.6.2 A series of tests were run on asphalt-rubber using the E, F, and G tubes. Capillary diameter of these tubes are 3.186, 4.650, and 9.700 mm. respectively. The same parameters of mixing temperature and holding time that were used in previous experiments were used for this factorial. In addition, three tube sizes were included. Data generated include a parameter of the rheogram, shear susceptibility index (c),  $R^2$  (correlation coefficient of data), apparent viscosity at a shear rate of  $0.05 \text{ sec.}^{-1}$  ( $\eta_{0.05}$ ). Data of the experiment are shown in appendix G.

10.6.2.1 The fixed factor model used for this analysis is:

$$Y_{ijkl} = \mu + T_i + C_j + H_k + (TC)_{ik} + (CH)_{ik} + (TCH)_{ijk} + E_{(ijk)l}$$

where:

$Y_{ijkl}$  = response measured (a, c,  $R^2$ ,  $N_a$ , etc.)

$\mu$  = Effect of the overall mean.

$T_i$  = Effect of  $i^{\text{th}}$  mixing temperature.

$C_j$  = Effect of  $j^{\text{th}}$  capillary size.

$H_j$  = Effect of  $n^{\text{th}}$  reaction time.

$(TC)_{ij}$  = Interaction of temperature by capillary.

$(TH)_{ik}$  = Interaction of temperature by time.

$(CH)_{ik}$  = Interaction of capillary by time.

$(TCH)_{ijk}$  = Three way main effect interaction of temperature, capillary and reaction time.

$E_{(ijk)l}$  = Experimental error.

10.6.2.2 Analysis was made to determine the effect of capillary size, mixing temperature, reaction time, and

all interactions. For all cases, capillary size is highly significant at the 0.01 level. Each parameter will be discussed below.

10.6.3 The a parameter of a linear system in cartesian coordinates represents the intercept of the curve with the y-axis when x equals 0. For the case of logarithmic or "power functions" as is found for rheograms ( $\eta_a = a \dot{\gamma}^b$ ), the a parameter serves as a shift factor for linear functions on a log log plot. The b parameter is, of course, the slope of the functions on a log log plot.

10.6.3.1 Analysis of variance, see appendix G, shows the following effects on the a parameter.

- A. Capillary diameter (tube) is significant at the 0.01 level. The level of significance (or effect) of capillary size is greater than that of mixing temperature or reaction time.
- B. Mixing temperature is significant at the 0.05 but not at the 0.01 level.
- C. Reaction time is significant at the 0.05 level but not at the 0.01 level.
- D. The only interaction term that is significant is the three way interaction of the main effects (temperature by capillary by holding time). This term is significant at the 0.01 level and should be expected in view of the strong effect of capillary size and the significance of time and temperature.

10.6.4 Shear susceptibility index (c) is defined as:

$$c = (1 + \text{slope}) = 1 + b$$

analysis of the effect of experimental factors on b were not made because the same factors that affect the slope (b) will be reflected in the shear susceptibility index (c).

10.6.4.1 Analysis of variance, see appendix G-21, shows that the only factor that affects the shear susceptibility index is capillary size. No other main effects (temperature and time) have a significant effect. The three way interaction is highly significant, probably due to the strong influence of capillary size.

10.6.5 Correlation coefficient ( $R^2$ ) is defined as:

$$R^2 = \frac{\text{Sum of Squares Due to Regression}}{\text{Sum of Squares About the Mean}}$$

This value is a measure of the "goodness to fit" of the data to the calculated regression model (a and b parameters). It can be interpreted as a measure of the total variability that is explained by the model. Values close to unity represent a good fit and values approaching zero indicate a poor fit and hence, probably the selection of an incorrect model to represent the data.

10.6.5.1 Analysis of variance, see appendix G-21, shows tube size to be highly significant.

10.6.6 Viscosity at 0.05 reciprocal seconds ( $\eta_{0.05}$ ).

10.6.6.1 Values for this property are obtained from the rheogram by plotting calculated values of apparent

viscosity ( $\eta_a$ ) versus shear ( $\dot{\gamma}$ ) on a logarithmic scale. Since the shear rate of exactly 0.05 is rarely obtained during the test, a best fit straight line is drawn through the data and the viscosity value is read from the curve or extrapolated for a particular shear rate.

10.6.6.2 For this study, a least square regression (power curve fit) was used to plot the curve and obtain the viscosity for a given shear rate. It was this regression that generated a, b, and  $R^2$  that were analyzed earlier.

10.6.6.3 In order to satisfy homogeneity of variance requirements, the data was transformed as follows:

$$N_{0.05}^1 = \log_{10} (N_{0.05} + 1.00 \text{ E} + 07)$$

Where:

$N_{0.05}^1$  = transformed viscosity.

$N_{0.05}$  = measured viscosity.

E+07 = scientific notation for powers of 10.

10.6.6.4 Analysis of variance, see appendix G, of transformed data shows capillary size to be highly significant and that holding time is significant at the 0.05 level but not at the 0.01 level.

10.6.6.5 Data (see appendix G) show a range of values from 0.3134 E+07 (tube E, 375F, 0.5 hr.) to 94.05 E+07 (tube F, 375F, 9.5 hr.) Pascal-Seconds (Pa·S). Since 1 Pa·S is 10 Poises, the range is 3.134 E+07

to 9.405 E+09 Poises. It should be noted that, at 32.9F, viscosity values are approaching the maximum (or asymptotic) values of  $10^9$  to  $10^{10}$  Poises that are sometimes used to define the change of state of fluids to solids.

10.6.6.6 Viscosity measurements on the base asphalt used for the asphalt-rubber after the asphalt was heated to 375F for 1 hour show ranges of values from 5.0 E+07 to 95 E+07 Pa·S. (All measurements and data are not included in this report due to space limitations, but are available upon request.)

11.0 Elastic Rebound

11.1 Asphalt rubber mixtures were prepared at Engineers Testing Laboratories and sent to Mr. R. L. Dunning of Petroleum Sciences, Spokane, Washington for testing. Testing was performed at 77F (25C) by the method described by Green and Tolonen (13). The experiment was not replicated, hence there is a single data point for each temperature - holding time combination. Rebound data (in percent) are shown in Table 11-1.

Table 11-1  
Elastic Rebound

|      |   | Temperature |      |      |
|------|---|-------------|------|------|
|      |   | 350         | 375  | 400  |
| Time | ½ | 78.2        | 74.0 | 76.0 |
|      | 1 | 81.6        | 81.8 | 95.6 |
|      | 2 | 81.4        | 90.2 | 82.0 |

11.2 Since the data was not replicated, two way analysis of variance was not performed.

11.2.1 Grouping the data by temperatures and performing one way analysis of variance showed no significant effect due to treatment (temperature).

11.2.2 Grouping the data by holding time and performing one way analysis of variance showed no significant effect due to treatment (holding time).

11.2.3 Elastic rebounds ranged from a high of 95.6 to a low of 74.0 percent. Mean for the 9 data points is 82.3 percent and standard deviation is 6.77 percent.

## 12.0 Conclusions

12.1 Conclusions are, of course, based on results of tests and experiments conducted during this study. Experiments were designed to be completely randomized fixed model factorials with two replications. Independent variables were three levels of mixing temperature (350, 375, and 400F(176.7, 190.6, and 204.4C), and three levels of reaction time (0.5, 1.0, and 2.0 hours).

12.1.1 Tests performed as part of this study can be classified as either:

- Modifications of standard or conventional asphalt tests.
- Non-standard tests.

12.1.2 Standard (modified) or conventional tests include:

- Ring and Ball Softening Point
- Absolute Viscosity with a Capillary Viscometer.
- Ductility at Low Temperatures.

12.1.3 Non-Standard tests include:

- Force-Ductility
- Apparent Viscosity by Schweyer Rheometer
- Elastic Rebound by Sliding Plate Microviscometer.

12.2 A tabulation of results of testing and analysis of variance is shown in Table 12-1.

12.3 In general, conventional tests, with precision constraints that are applied to asphalt testing, are not applicable for testing asphalt-rubber without modification of both the test method and the precision statement.

Table 12-1  
Summary of Results

| Measurement               | Analysis of Variance Significance |      |             |
|---------------------------|-----------------------------------|------|-------------|
|                           | Temp                              | Time | Interaction |
| Softening Point           |                                   |      |             |
| All Data                  | No                                | No   | No          |
| Mean of Runs              | No                                | No   | No          |
| Absolute Viscosity (140F) |                                   |      |             |
| All Data                  | No                                | Yes  | No          |
| Mean of Runs              | No                                | No   | No          |
| Ductility at 39.2F        |                                   |      |             |
| All Data                  | Yes                               | No   | No          |
| Mean of Runs              | Yes                               | No   | No          |
| Max. Ductility Load       |                                   |      |             |
| All Data                  | No                                | Yes  | Yes         |
| Force - Ductility         |                                   |      |             |
| All Data                  | No                                | Yes  | Yes         |
| Schweyer Rheometer        |                                   |      |             |
| a                         | Yes                               | Yes  | No          |
| c                         | No                                | No   | No          |
| R <sup>2</sup>            | No                                | No   | No          |
| $\eta_{0.05}$             | No                                | Yes  | No          |
| Elastic Rebound           | No                                | No   | -           |

- 12.4 There appears to be a measurable effect of mixing temperature and reaction time on properties measured by the Force - Ductility test.
- 12.5 Force - Ductility (utilizing elongation at failure and either maximum load at failure or area under the load elongation curve) is the most sensitive to detection of effects of time and temperature of all tests conducted during this study.
- 12.6 The Schwyer Rheometer is effective and efficient for determination of viscosity at low temperatures. There is, as was expected, an effect due to capillary size (tube effect) used for the test. Furthermore, there is a strong possibility that measurements obtained with the device can be used to calculate properties such as stiffness and relaxation time.
- 12.7 Laboratory mixing procedures may not adequately simulate field techniques and hence, laboratory prepared materials may not be the same as those manufactured in the field. This aspect will be discussed further in the following section.
- 12.8 Without correlation of field performance to specific properties measured in the laboratory, materials specifications should be based on a combination of manufacturer's recommendations, experience with producing and placing the material, and strict control of material components and quantities used for production. This is not to say that laboratory testing is not effective. Tests considered during this study show promise, with additional development, of providing both quality control and behavior information.

## 13.0 Discussion and Recommendations

### 13.1 Test Methods and Variability

13.1.1 As was briefly discussed previously, sources of variability of tests can most probably be attributed to interference or sidewall effects caused by rubber particles in the asphalt rubber mixture. These effects are observed in all the tests of this study with the possible exception of ductility. Ring and ball softening point and absolute viscosity with capillary viscometers are especially affected.

13.1.1.1 Two recommendations are offered for softening point determinations if softening point is, indeed, a property that should be measured. The first is to develop a larger ring and ball that would have the same relative geometry and volume - geometry ratios, that are used for the standard ASTM D36 test. Correlations would, of course, be necessary. The second, is to further develop the flow index concept of Krchma (18) that has good possibilities of characterizing asphalt rubber flow or consistency properties. This technique promises relatively rapid and economical measurements.

13.1.1.2 With regards to capillary viscometry, there appears to be two areas that can be considered. These are increased bore size to the number 800 tube and revised loading techniques and shear rate corrections. Additional reduction in vacuum may be necessary to produce acceptable flow times and shear rates and to prevent vacuum separation of asphalt from rubber particles. A modest experiment could be conducted

to compare viscosity and variability of the larger bore tube and the tube - vacuum combination that was used for this study. Loading techniques that have been considered (but not used in this study in order to correlate data obtained in this study with other viscosity measurements) include vibration of the tube during loading and packing with high pressure syringes.

- 13.1.1.3 There is no question that viscosity is an important property from both production and application as well as from research points of view. Viscosity is affected by asphalt properties, rubber type and particle size, proportions of asphalt and rubber components and the extent of physico-chemical reaction between rubber and asphalt. In addition, sprayability and consequent job quality and aggregate adhesion are severely affected by viscosity. Hence, it is strongly recommended that additional work be conducted to develop reliable and sensitive methods of measuring and evaluating mass viscosity of asphalt-rubber systems at elevated temperatures [in the region of field applications such as 350-400 F (177-204 C)]. Measuring systems and techniques should be developed that have both laboratory and field capability or that can be reliably correlated. The principle feature that has to be considered is to provide for specimen size adequate to accommodate rubber particles without particle to particle interference, and without sidewall or end effects. It would be worthwhile to consider both coaxial cylinder [as described by Jiminez (19)] and vane or rotational viscometers. The possible use of the laboratory mixer as a viscometer will be discussed below. It

would seem advisable to devote a separate study to consider several possibilities of viscosity measurement of asphalt-rubber at elevated temperatures to establish a standard method for specification, production, and application quality control purposes.

13.1.1.4 Flow characteristics at lower temperatures appear to be measurable with the Schweyer Rheometer, although some modifications of the systems should be considered. The first suggested modification is to increase the frame stiffness of the instrument. At low temperatures, relatively large loads are necessary to force the specimen through the capillary. Extension of the frame during this loading is not constant and hence a simple linear correction cannot be applied. Stiffening of the frame would reduce variability of output and is a straightforward operation. A second suggested modification of capillary sizes available is intended to reduce the effect of particle interference on measurements.

13.1.1.4.1 Asphalt-rubber mixtures contain agglomerations of several particles held together with thin asphalt films. These clusters in turn, can produce interference similar to that of simple large particles. Measurements made with different capillary sizes showed, as should be expected, an effect due to capillary size. It should also be noted that asphalt (without rubber) showed the same size effect and the question is open as to the cause of the phenomena. Without additional study, it is difficult to estimate the tube size that

would be required to eliminate, or at least to stabilize, the effect of particle interference. The largest tube size available for the instrument used for this study is 9.47 mm. (approximately 3/8 inch). This capillary size does not provide for the effect of clusters or agglomerations of particles. It is recommended that larger size tubes be developed, along with appropriate constants, that can be accommodated by the existing instrument. The chamber of the instrument can accept tubes with outside diameters of approximately 3/4 inch. It may also be possible to machine the chamber to accept even larger tubes and to provide sleeves to hold the original tubes.

13.1.1.5 Force-ductility appears to show good possibilities for measurement of low temperature properties of asphalt-rubber and should receive additional development. Force-ductility was the only simple test used during this study that was sensitive to both mixing temperature and holding (or reaction) time. In addition, the test provides data and information on tensile strength and can, as will be subsequently discussed, provide creep compliance or stiffness modulus information compatible with theoretical elastic or viscoelastic models.

13.1.1.5.1 Recommendations for modification of the test method and apparatus include changes of load read-out and specimen geometry. Load cells with improved precision over the force ring used for this study are recommended along with compatible recording equipment. These load cells have been installed as part of another project. Standard ductility specimens,

as were used in this study, have a constantly decreasing cross section down to a minimum area of 1 cm.<sup>2</sup> Preliminary tests show the feasibility of modifying specimen geometry to provide a constant cross section of 1 cm.<sup>2</sup> for a length of approximately 6 cm.

- 13.1.1.5.2 The test envisioned is neither constant stress nor constant strain that would be ideal but requires expensive equipment, preferably electrohydraulic closed loop devices with process controllers. The test would be a constant deformation rate as provided by the ductility drive mechanism. Load versus time is provided by the load cell and elongation versus time is provided by recording head movement and the increase of gage length of the specimen. Strain can be calculated on the basis of engineering strain ( $\epsilon'$ ) or true strain ( $\epsilon$ ) for large deformations. These strains are calculated as follows:

$$\epsilon' = \frac{\Delta L}{L}$$

$$\epsilon = \ln (1 + \epsilon')$$

where  $\Delta L$  = change of length

L = original gage length

- 13.1.5.3 Stress calculations can be based on original cross sectional area or on reduced areas measured manually by micrometer. A plot of strain versus time and stress versus time can be used to generate the logarithmic function curve of compliance or stiffness versus time. Stiffness at short times (traffic loads) and at long times (thermal effects) would not be measured directly as part of the test but

could be extrapolated, with sufficient precision and accuracy, for initial use in theoretical examinations of field behavior of asphalt rubber membranes and interlayers.

## 13.2 Asphalt - Rubber Reactions

13.2.1 Green and Tolonen (13) have discussed physico - chemical aspects of the asphalt rubber reaction in some detail. Discussions presented here are background for recommendations for testing and development programs and to report observations made which the scanning electron microscope (SEM).

13.2.2 It is generally agreed that rubber particles of the type used for this study swell to two to three times their original volume when fully reacted in hot asphalt. It is further postulated that swelling is the result of rubber particles imbibing or reacting with certain components of the base asphalt. Heat is necessary to liquify asphalt and contributes to the rate of reaction. Laboratory testing has been conducted that shows practically complete breakdown (dissolving) of rubber particles if the asphalt rubber mixture is subjected to sufficient reaction time and temperature. Rubber particles heated in air do not show a tendency to swell or melt at times of up to one hour at 400F (204C), hence the conclusion of a physio-chemical reaction between rubber and asphalt appears to be justified.

13.2.3 It has also been pointed out that there are different types of rubber (synthetic types and natural) used for different tires (truck tires versus conventional automo-

bile tires and radial tires versus bias tires). In fact, the rubber types may be different for tread rubber than for sidewall material (21).

- 13.2.4 Tire rubber compounding involves the addition of extender oils to impart certain properties and characteristics to rubber to better function for an intended purpose. In addition, rubber compounding often uses reclaimed tire rubber that has been treated with petroleum products (chemically unsaturated resin oil obtained in the refining of gasoline (22)). These extender oils and reclaiming agents can be characterized by the same techniques used to determine the fractional composition of asphalt. In fact, some components of asphalt maltenes are very similar to extender oils and reclaiming agents when analyzed by the acid precipitation method described by Rostler (14).
- 13.2.5 It follows, then, if asphalt and rubber react chemically, that chemical composition or proportions of components of either asphalt or rubber can affect properties and probably performance of asphalt-rubber mixtures. This study showed that mixing temperature and reaction time have measurable effects on certain properties of these mixtures. Only minimal control can be exercised over reclaimed tires (sorting by type, separation of treads and sidewalls, selection of grinding method, and particle size). It is generally accepted that asphalts from the same specification grade can vary chemically due to different crude sources and with different refining methods.
- 13.2.6 It is recommended that studies be initiated to consider the effects of asphalt and rubber properties on

characteristics of asphalt-rubber mixtures. Information on these procedures and on mixtures using a single asphalt-rubber combination generated from this study can serve as datum to develop subsequent studies. Presently, research is being conducted that will consider several chemical and physical aspects of asphalt-rubber mixtures. This study should be complemented by others that vary certain components of asphalt (such as the Rostler parameters) and use rubber of known composition. These rubbers can be aged materials prepared from stocks of known composition or perhaps special lots of reclaimed tires can be processed through a conventional reclaiming plant for this study.

### 13.3 Test Specimen Preparation

13.3.1 As was discussed previously, laboratory specimens for this study were prepared by heating asphalt in an open beaker, introducing rubber and then mixing with a high speed propeller. This technique was established by Jimenez (19) in a previous study and, for consistency, used in this project.

13.3.2 Measurements made after subjecting the asphalt (without rubber) to the same mixing showed that about the same amount of hardening takes place as occurs in the rolling thin film circulating oven exposure. Laboratory mixing obviously removes some of the more volatile components of the asphalt. One aspect of this loss is that these fractions are not available to react with the rubber.

13.3.3 Field preparation of the mixture, on the other hand, is accomplished in a closed tank that is nearly full and hence, in an atmosphere that has a greatly re-

duced oxygen content compared to laboratory preparation. Furthermore, the closed field system probably has much less loss of volatiles than does the laboratory method.

13.3.4 This brings up the question of similarity of properties of laboratory versus field mixed materials. Field mixed materials were not feasible for use in this study because of the obvious logistics difficulties as well as the lack of control of mixing temperature and holding times for field mixtures. The companion study that is presently underway is considering two sub studies to provide insight into the question.

- Field mixed materials were sampled and will be subjected to the same tests that were performed during this study.
- A mixing device has been developed by Arizona Department of Transportation that shows promise of retaining volatile components of asphalt during the mixing operation.

13.3.5 An asphalt-rubber field experimental project is being conducted by Arizona Department of Transportation (Buckeye - Liberty Project S-371-924). This project includes 9 test sections on a two-lane, heavily trafficked highway. This project, along with considering several structural applications, had included several combinations of asphalt and rubber types along with the combinations of material used for this project.

- 13.3.5.1 All field produced asphalt rubbers were sampled and frozen for future testing as part of an ongoing study.
- 13.3.5.2 All tests made during this study will be conducted on the field prepared materials and comparisons will be made between properties and variability of field and laboratory mixed materials. It is conceded that some confounding may occur because neither the asphalt nor the rubber used for the field study was from the same lot as was used for the laboratory study. If necessary, however, laboratory mixtures can be prepared from component materials (asphalt, rubber, and additives) samples obtained from the field project.
- 13.3.6 The ongoing project will use a laboratory mixing device recently developed by the Arizona Department of Transportation referred to as the "Arizona Torque Fork", (ATF). This device has several features that show promise for both research purposes and for routine laboratory preparations of asphalt rubber mixtures.
- 13.3.6.1 First, the device uses a controlled electrical heating system that is considerably safer than the open flame that was employed as part of this study. Elimination of the open flame is of importance if high volatility additives such as kerosene and certain extender oils are to be used in laboratory preparations.
- 13.3.6.2 The mixing chamber of the device is covered (but not sealed) and should significantly reduce the

loss of volatile asphalt components during long term mixing.

13.3.6.3 One of the most important features of the mixer is the ability to simultaneously record temperature, viscosity, and mixing time. Time is recorded on the strip chart and temperature is sensed by a resistance thermometer and recorded on the same strip chart. Torque of the mixer motor is indirectly measured by current flow required to maintain constant speed and is also recorded on the same strip chart. Viscosity can be determined from torque by calibration with standard materials of known viscosity.

13.3.6.4 Instantaneous measurement of viscosity during mixing provides several research opportunities. First, the effect of heat and time on base asphalt can be determined much more conveniently with this device than with conventional capillary viscometers. Secondly, if viscosity is an indicator of the extent of reaction between particular asphalt and rubber combinations, the device can easily monitor the rate and extent of these reactions for a large number of material combinations.

13.3.7 Two experiments utilizing the ATF are recommended:

- A comparison of field mixed asphalt rubber properties with those prepared in the laboratory. This will be an effort to standardize laboratory preparation methods and techniques.

- A study of the rate of reaction of various combinations of asphalt type, rubber type, and additives.

13.3.7.1 The first study would be a combined field and laboratory effort. Typical field projects using two commercial asphalt-rubber producers would be selected for study. Base materials (asphalt, rubber, and additives) would be extensively sampled for later laboratory study. Field production would be monitored (by field viscosity) throughout the production and application of the material. Laboratory mixtures would be prepared with the open beaker and ATF and be constantly monitored for viscosity as a function of mixing time and temperature. If correlations exist between field and laboratory viscosities, standard laboratory preparation procedures could be developed. If desirable, force-ductility properties could be used as a property of the prepared mixture for comparison of field and laboratory prepared mixtures. Incidentally, test variability (repeatability) information would also be generated during the program.

13.3.7.2 The second study would be a laboratory effort designed to measure the interaction of asphalt type, rubber type, and additives on viscosity and force-ductility parameters. Asphalts would be characterized by the usual specification properties and by the Rostler parameters. Rubber would be characterized by size, origin (natural or devulcanized content), and processing method. The experiment would monitor viscosity during mixing with the ATF for each combination of asphalt and rubber and

would measure force ductility parameters of the final product.

#### 13.4 Correlations of Field Performance and Material Properties to Establish Specification Criteria and Limits.

13.4.1 In order to develop specifications two areas must be considered:

- Material components, properties, and characteristics necessary for a specific function or application.
- Production and application procedures that provide the desired quality of material to a particular service environment.

These areas must be considered for either a prescription or performance specification and must be supported with standardized sampling and test methods.

Specifications for conventional asphalt materials and applications have developed with years of scientific and applied study and experience. Observations relating field performance to materials properties and application methods has resulted in the development of specifications that use a combination of several properties that are used to evaluate the material and hence, to predict performance.

13.4.2 Asphalt-rubber, on the other hand, is a new material that has some characteristics in common with conventional materials but is also quite different in many respects. Furthermore, the material is being used

for some structural purposes that are in themselves, a departure from conventional paving techniques (for example, stress absorbing membrane interlayer (SAMI) and membranes for soil encapsulation). Field experience has shown that the material is capable of extraordinary performance and has also shown that, in a few isolated instances, that substandard performance and failure can occur. Without doubt, a complete specification could be developed with time, that would be adequate for most construction purposes. It seems reasonable, however, to recommend a systematic experimental program that can more efficiently and effectively develop a specification system than would a conventional trial-and-error approach.

13.4.3 This study has shown that conventional asphalt testing and technology will require modification, but can probably be used for asphalt-rubber. Additional field-laboratory studies, in somewhat the same format as the Buckeye-Liberty Project, should be designed to further consider both material components and the effect of production techniques on performance. Components and techniques could be selected in such a manner that accelerated results would be available in a reasonable time frame.

13.5 Scanning Electron Microscope (SEM) Study of Asphalt-Rubber.

13.5.1 A limited study of asphalt-rubber and of the rubber was conducted to provide some insight on the nature of the material. A secondary objective was to consider the possibility of reacted particles of rubber connecting

or being welded together to form a continuous network of rubber particles.

- 13.5.2 Review of unreacted rubber particles showed a gradation of sizes and shapes. Some particles are elongated, some are cubical and all have irregular fractured faces. Examination of the asphalt rubber suggests a system of rubber aggregate particles with an asphalt binder.
- 13.5.3 An asphalt rubber mixture was extracted with cold (room temperature) trichloroethylene with repeated washings until the solvent was clear and light straw colored. Subjective estimates were that 95 percent or more of the binder was removed but that either some binder remained because rubber particles were knitted together to form a coherent rubber structure. Viewing of SEM photomicrographs of this material shows some weldment that is probably the asphalt binder and not rubber to rubber adhesion.
- 13.5.4 Additional cold washing with trichloroethylene were repeated until individual particles rather than the coherent net were produced. Viewing of photomicrographs of these particles showed no evidence of rubber to rubber weldment. Since the possibility exists that the solvent may have destroyed rubber to rubber weldment, particles of rubber were soaked in the solvent for 24 hours at room temperature, dried and examined with the SEM. Large magnifications were used to examine fractures within particles and contact surfaces between particles. It was reasoned that rounding or other evidence of chemical action of the solvent would be apparent, at least subjectively. All

edges were sharp with no apparent rounding due to rubber solution by the solvent.

13.5.5 Based on these limited observations, there is no conclusive evidence that rubber to rubber weldment occurs to produce a continuous rubber network.

13.5.6 This study, while probably not definitive for establishing the presence or absence of a connected rubber network, did show the value of scanning electron microscopy as a tool in asphalt rubber research and evaluation. It is strongly recommended that the method be employed for all future work for both analysis and documentation of materials used.

#### 14.0 Acknowledgements

- 14.1 Engineers Testing Laboratories, Inc. wishes to express their appreciation to the Research Division of Arizona Department of Transportation and to the U.S. Department of Transportation, Federal Highway Administration for funding and administration of the study. They wish to also express their appreciation for direction and helpful input from the Project Research Advisory Panel.

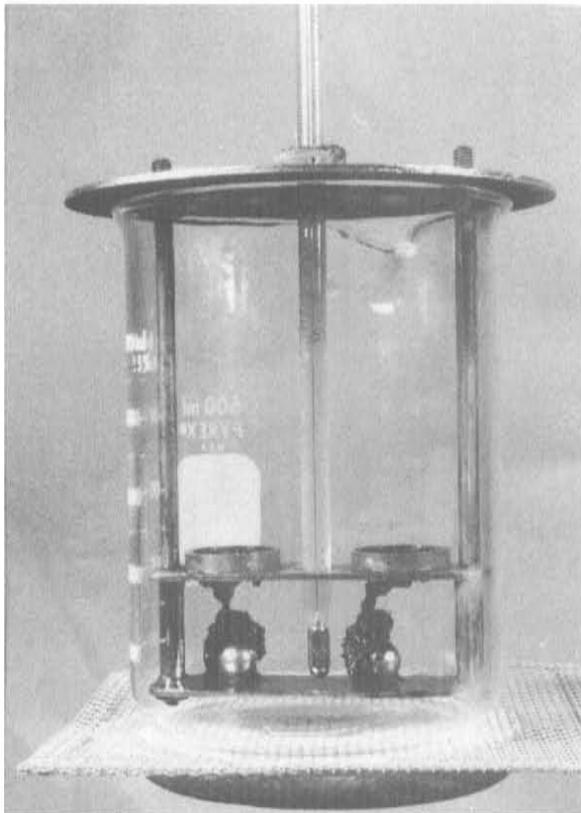
## REFERENCES

1. Rostler, F.S., "An Annotated Bibliography on the Use of Rubber in Asphalt Pavements", Federal Highway Administration Report Number FHWA-RD-72-1, United States Department of Transportation, Washington, D.C., May, 1971.
2. Wood, L. E. and Pavlovich, R.D., "The Use of Replacement Materials in Asphaltic Concrete", Proceedings, Twenty-Second Annual Arizona Conference on Roads and Streets, University of Arizona, Tucson, Arizona, April, 1973.
3. Stephens, J. E. and Mokrzewski, S. A., "The Effect of Reclaimed Rubber on Bituminous Paving Mixtures", School of Engineering Report CE 74-75, The University of Connecticut, Storrs, Connecticut, March, 1974.
4. Galloway, B. M. and La Grove, B. D., "Use of Rubber Aggregate in a Strain Relieving Interlayer for Arresting Reflection Cracks in Pavements", a paper presented at the International Symposium of the Use of Rubber in Asphalt Pavements, Salt Lake City, Utah, May, 1971.
5. McDonald, C. H., "Bituminous Paving as Related to Large Commercial Airports in the Urban Environment", 50th Annual Meeting of the Highway Research Board, Washington, D.C., January, 1971.
6. Lansdon, H. G., "Construction Techniques of Placement of Asphalt-Rubber Membranes", The University of New Mexico Paving Conference, 1976.

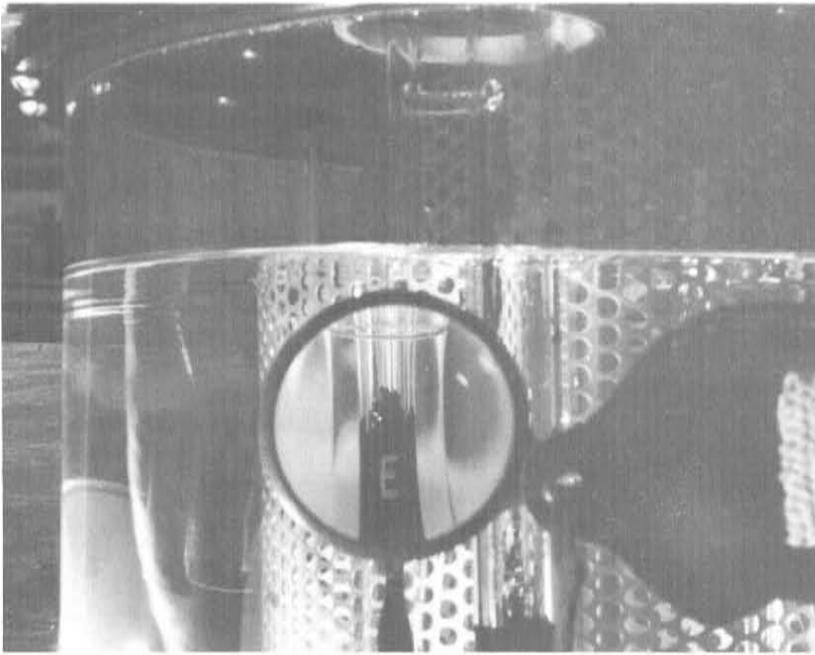
7. Oliver, J. W. H., "A Critical Review of the Use of Rubbers and Polymers in Bitumen Bound Paving Materials", Project Number 1037, Use of Rubbers and Polymers in Bituminous Services, Australian Road Research Board, 1977.
8. Morris, G. R. and McDonald, C.H., "Asphalt-Rubber Stress Absorbing Membranes - Field Performance and State-of-the Art", Arizona Department of Transportation, Research Report, 1975.
9. Pavlovich, R. D. and Morris, G. R. "Utilization of Recycled Tire Rubber as an additive to Asphalt Cement"; The International Conference on the Use of By-Products and Waste in Civil Engineering, Paris, France, November, 1978.
10. Frobel, R. K., Jiminez, R. A., and Cluff, C. B., "Laboratory and Field Development of Asphalt-Rubber for Use as a Waterproof Membrane", Arizona Transportation and Traffic Institute, University of Arizona, Tucson, Arizona 1977.
11. "Scrap Tires Can Yield Marketable Products", Solide Wates-II, Articles from Volumes 4 - 7 of Environmental Science and Technology collected by Stanton S. Miller, American Chemical Society, Washington, D.C., 1973.
12. Landsdon, H. G., "The Blending of Granulated Rubber and Asphalt for Use as a Crack Sealant", reprint available from Sahuaro Petroleum and Asphalt Company, 731 N. 19th Avenue, Phoenix, Arizona 85005.
13. Green, E. L. and Tolonen, W. J., "The Chemical and Physical Properties of Asphalt-Rubber Mixtures", Arizona Department of Transportation, 1977.

14. Rostler, F. S., "Fractional Composition: Analytical and Functional Significance", from Bituminous Materials: Asphalts, Tars and Pitches, Volume II, Asphalts, edited by A. J. Hoiberg, Interscience, 1965.
15. Anderson, V. L. and McLean, R.A., "Design of Experiments, a Realistic Approach", Marcel Decker, Inc., 1974.
16. Anderson, D. I. and Wiley, M. L., "Force Ductility, an Asphalt Performance Indicator", Volume 45, Proc. AAPT, 1976.
17. Schweyer, H. E. and Burns, A. M., Proc. AAPT, 1978, in manuscript.
18. Krchma, L. C. Proc. AAPT, Vol. 16, 1967.
19. Jimenez, R. A., "Testing Methods for Asphalt-Rubber," Arizona Department of Transportation Report: ADOT-RS-15 (152), January, 1978.
20. Van Wazer, J. R., Lyons, J. W., Kim, K. Y., and Colwell, R. E., "Viscosity and Flow Measurement - a Laboratory Handbook of Rheology", Interscience, 1963.
21. "The Vanderbilt Rubber Handbook", edited by R. O. Babbit, The Vanderbilt Company, Inc., 30 Winfield Street, Norwalk, CT, 06855, 1978.
22. Smith, F. G., "Reclaimed Rubber," Vanderbilt Handbook, op. cit.
23. Schweyer, H. E., Smith, L. L., and Fish, G. W., "A Constant Stress Rheometer for Asphalt Cements," Proc. AAPT, Vol. 45, 1976.

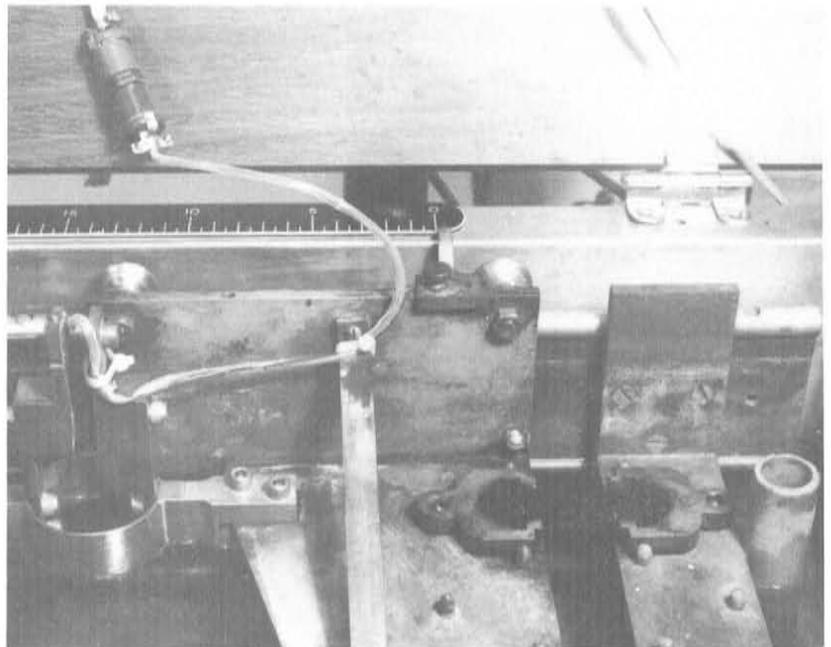
**Figure 1**  
**Mixing System**



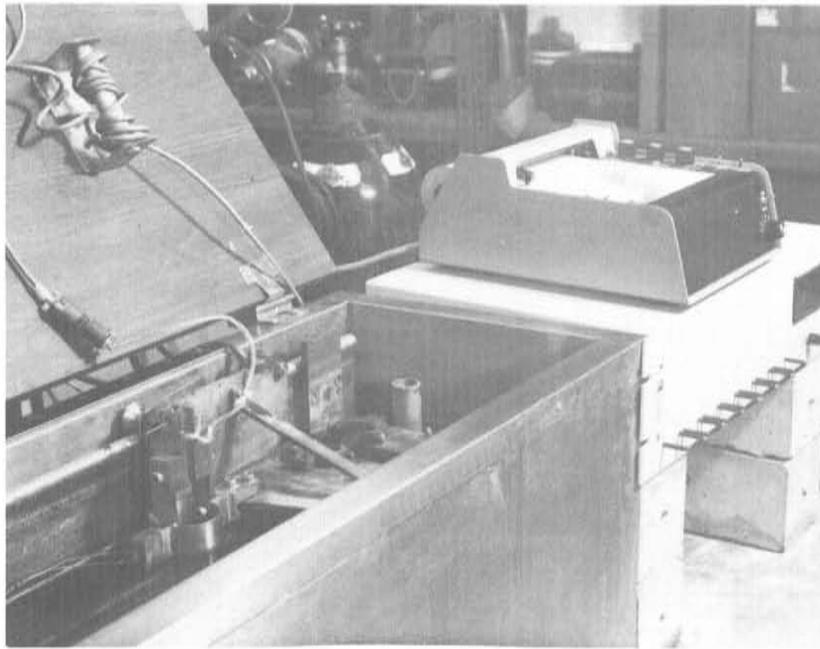
**Figure 2**  
**Ring and Ball Softening Point**



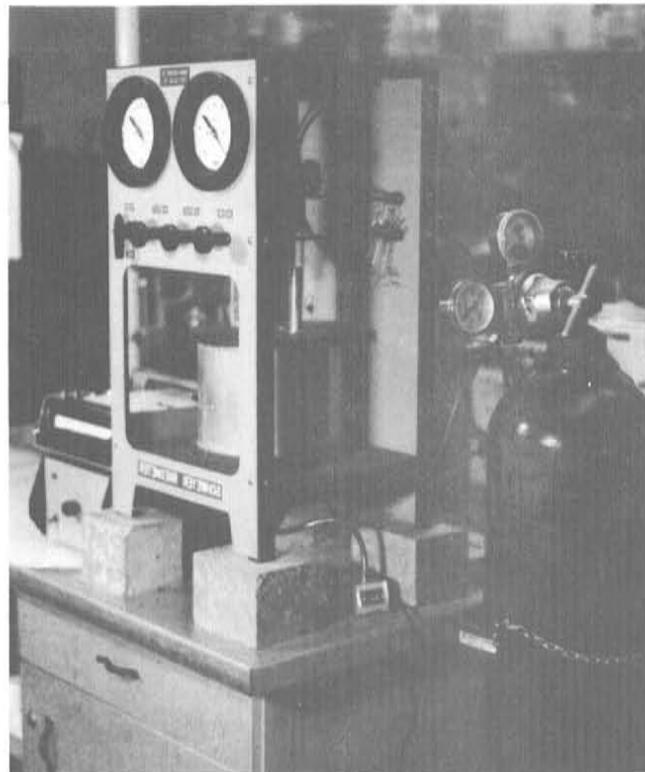
**Figure 3**  
**Absolute Viscosity**



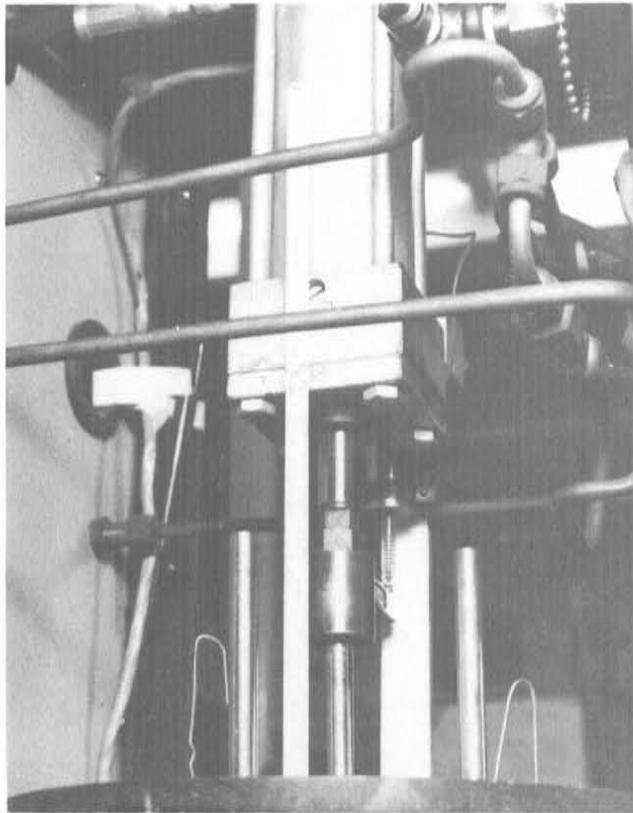
**Figure 4**  
**Force Ring and Specimen**



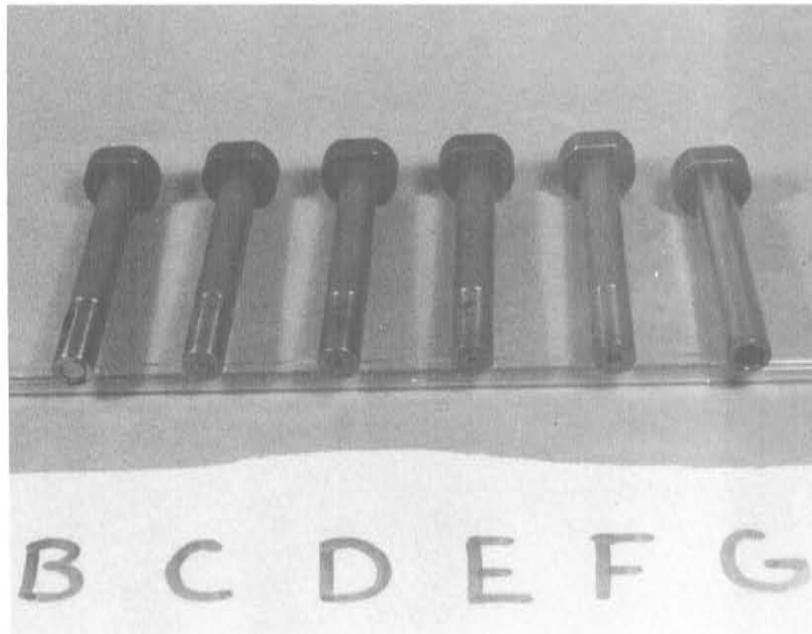
**Figure 5**  
**Force-Ductility Set-up**



**Figure 6**  
**Schweyer Rheometer**  
75



**Figure 7**  
**Rheometer Load Piston**



**Figure 8**  
**Rheometer Tubes**  
76

Table A-1  
SOFTENING POINT  
All Data

DATA MATRIX

| TIME   | TEMPERATURE |       |      |       |     |       |
|--------|-------------|-------|------|-------|-----|-------|
|        | 350         |       | 375  |       | 400 |       |
| 0.5    | 161         | 162.0 | 162  | 161.5 | 158 | 156.5 |
|        | 163         | 2.0   | 161  | 1.0   | 155 | 3.0   |
|        | 148         | 149.4 | 155  | 154.5 | 160 | 160.0 |
| 1.0    | 151         | 3.0   | 154  | 1.0   | 160 | 0     |
|        | 155         | 155.5 | 160  | 159.5 | 162 | 161.0 |
|        | 156         | 1.0   | 159  | 1.0   | 160 | 2.0   |
| 2.0    | 159         | 158.5 | 163  | 163.5 | 164 | 165.0 |
|        | 158         | 1.0   | 164  | 1.0   | 166 | 2.0   |
|        | 152         | 153.0 | 162  | 162.5 | 161 | 162.5 |
| Totals | 154         | 2.0   | 163  | 1.0   | 164 | 3.0   |
|        | 159         | 160.0 | 156  | 155.5 | 146 | 145.5 |
|        | 161         | 2.0   | 155  | 1.0   | 145 | 1.0   |
|        |             | 1877  | 1914 | 1901  |     |       |

Left Ball 162  
Right Ball 163  
Mean 162.0  
Range 2.0

Range: 145.5 - 165.0

CELL MEANS AND RANGE  
(Based on Replicate Means)

| TIME | TEMPERATURE |        |        |        |        |        |
|------|-------------|--------|--------|--------|--------|--------|
|      | 350         |        | 375    |        | 400    |        |
| 0.5  | 155.75      | 158.00 | 158.00 | 158.25 | 158.25 | 158.25 |
|      | 12.5        | 7.0    | 7.0    | 3.5    | 3.5    | 3.5    |
| 1.0  | 157.00      | 161.50 | 161.50 | 163.00 | 163.00 | 163.00 |
|      | 3.0         | 4.0    | 4.0    | 4.0    | 4.0    | 4.0    |
| 2.0  | 156.50      | 159.00 | 159.00 | 154.00 | 154.00 | 154.00 |
|      | 7.0         | 7.0    | 7.0    | 17.0   | 17.0   | 17.0   |

Mean 155.75  
Range 12.5

TABLE A-2  
ANOVA  
SOFTENING POINT  
All Data

FOSTER & BURR CI:  
Matrix  $IX_i$  5692  
Matrix  $IX_i^2$  900.892  
 $v$  3.00  
 $q$  0.269  
 $q_{0.99}$  0.287  
 $q_{0.999}$  0.363

$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon_{(ij)k}$

TOTALS

Temperature ( $T_i$ )

|           |   |      |                |           |   |      |
|-----------|---|------|----------------|-----------|---|------|
| $T_{1..}$ | = | 1877 | Time ( $H_j$ ) | $T_{.1.}$ | = | 1888 |
| $T_{2..}$ | = | 1914 |                | $T_{.2.}$ | = | 1926 |
| $T_{3..}$ | = | 1901 |                | $T_{.3.}$ | = | 1878 |
|           |   | 5692 |                |           |   | 5692 |

Temperature x Time ( $TH$ )<sub>ij</sub>

|           |   |     |
|-----------|---|-----|
| $T_{11.}$ | = | 623 |
| $T_{12.}$ | = | 628 |
| $T_{13.}$ | = | 626 |

|           |   |     |
|-----------|---|-----|
| $T_{21.}$ | = | 632 |
| $T_{22.}$ | = | 646 |
| $T_{23.}$ | = | 636 |

|           |   |     |
|-----------|---|-----|
| $T_{31.}$ | = | 633 |
| $T_{32.}$ | = | 652 |
| $T_{33.}$ | = | 616 |

$T_{...}$  = 5692  
 $TEY_{ijk}$  =

TABLE A-3  
ANOVA  
SOFTENING POINT  
All Data

(0)  $CT = (T \dots)^2 \div n = (5692)^2 \div (36) = \underline{8.99968444E + 05}$

(1)  $SS (Temp.) = \frac{1}{(12)} [(1877)^2 + \dots + (1901)^2] - CT = \underline{58.72}$

(2)  $SS (Time) = \frac{1}{(12)} [(1888)^2 + \dots + (1878)^2] - CT = \underline{106.89}$

(3)  $SS (H \times T) = \frac{1}{(4)} [(623)^2 + \dots + (616)^2] - CT - SS (T) - SS (H)$   
 $= \underline{84.44}$

(4)  $SS (Total) = \sum \sum x_{ijk}^2 - CT = \underline{923.56}$

(5)  $SS (Error) = SS (Total) - SS (T) - SS (H) - SS (T \times H)$   
 $= (4) - (1) - (2) - (3) = \underline{673.50}$

TABLE A-4

SOFTENING POINT  
Mean of Runs

DATA MATRIX

| TIME   | TEMPERATURE |       |       | Totals |
|--------|-------------|-------|-------|--------|
|        | 350         | 375   | 400   |        |
| 0.5    | 162.0       | 161.5 | 156.5 | 944.0  |
| 1      | 149.5       | 154.5 | 160.0 |        |
| 2      | 155.5       | 159.5 | 161.0 |        |
|        | 158.5       | 163.5 | 165.0 | 963.0  |
|        | 153.0       | 162.5 | 162.5 | 939.0  |
|        | 160.0       | 155.5 | 145.5 | 961.0  |
| Totals | 938.5       | 957.0 | 950.5 | 2846.0 |

| ANALYSIS OF VARIANCE |    |        |       |      |      |      |
|----------------------|----|--------|-------|------|------|------|
| Source               | df | SS     | MS    | F    | F.05 | F.01 |
| T <sub>i</sub>       | 2  | 58.72  | 29.36 | 1.18 | 3.35 |      |
| H <sub>j</sub>       | 2  | 106.89 | 53.44 | 2.14 | 3.35 |      |
| (TH) <sub>ij</sub>   | 4  | 84.44  | 21.11 | 0.85 | 2.73 |      |
| Error                | 27 | 673.50 | 24.94 |      |      |      |
| Total                | 35 | 923.56 |       |      |      |      |

TABLE A-5  
ANOVA  
SOFTENING POINT  
Mean of Runs

FOSTER & BURR q:

Matrix  $Ex_i$  2846.0  
Matrix  $Ex_i^2$  450,432.0

$v$  1.00  
 $q$  0.278  $q_{0.99}$  0.576  $q_{0.111}$  0.750

$y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon_{(ij)k}$

TOTALS

Temperature ( $T_i$ )

$T_{1..}$  = 938.5  
 $T_{2..}$  = 957.0  
 $T_{3..}$  = 950.5  
 $T_{...}$  = 2846.0

Time ( $H_j$ )

$T_{.1.}$  = 944.0  
 $T_{.2.}$  = 963.0  
 $T_{.3.}$  = 939.0  
 $T_{...}$  = 2846.0

Temperature x Time ( $TH$ )  $_{ij}$

$T_{11.}$  = 311.5  
 $T_{12.}$  = 314.0  
 $T_{13.}$  = 313.0  
 $T_{21.}$  = 316.0  
 $T_{22.}$  = 323.0  
 $T_{23.}$  = 318.0  
 $T_{31.}$  = 316.5  
 $T_{32.}$  = 326.0  
 $T_{33.}$  = 308.0  
 $T_{...}$  = 2846.0

$T_{...}$  = 2846.0  
 $THY_{ijk}$  = \_\_\_\_\_

TABLE A-6  
ANOVA  
SOFTENING POINT  
Mean of Runs

(0)  $CT = (T_{...})^2 + n = (2846)^2 + (18) = 4,499,842,222E + 05$   
(1)  $SS (Temp.) = \frac{1}{(6)} [(938.5)^2 + \dots + (950.5)^2] - CT = 29.36$   
(2)  $SS (Time) = \frac{1}{(6)} [(944.0)^2 + \dots + (939.0)^2] - CT = 53.44$   
(3)  $SS (H \times T) = \frac{1}{(2)} [(311.5)^2 + \dots + (308.0)^2] - CT - SS (T) - SS (H)$

= 42.22

(4)  $SS (Total) = THY_{ijk}^2 - CT = 447.78$

(5)  $SS (Error) = SS (Total) - SS (T) - SS (H) - SS (T \times H)$

=  $(4) - (1) - (2) - (3) = 322.75$

| ANALYSIS OF VARIANCE |    |        |       |      |      |      |
|----------------------|----|--------|-------|------|------|------|
| Source               | df | SS     | MS    | F    | F.05 | F.01 |
| (1) $T_i$            | 2  | 29.36  | 14.68 | 0.41 | 4.26 |      |
| (2) $H_j$            | 2  | 53.44  | 26.72 | 0.75 | 4.26 |      |
| (3) $(TH)_{ij}$      | 4  | 42.22  | 10.56 | 0.29 | 3.63 |      |
| (5) Error            | 9  | 322.75 | 35.86 |      |      |      |
| (4) Total            | 17 | 447.78 |       |      |      |      |

SOFTENING POINT  
All Data  
Cell Means

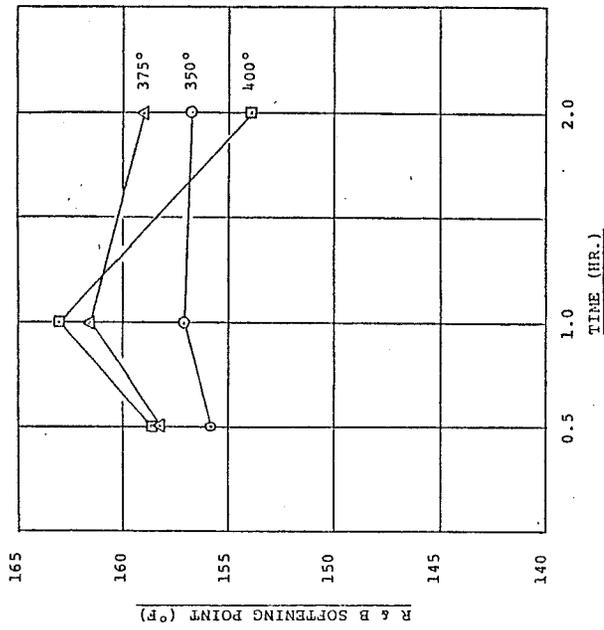


FIGURE A-1

R & B SOFT. VS TIME

SOFTENING POINT  
All Data  
Cell Means

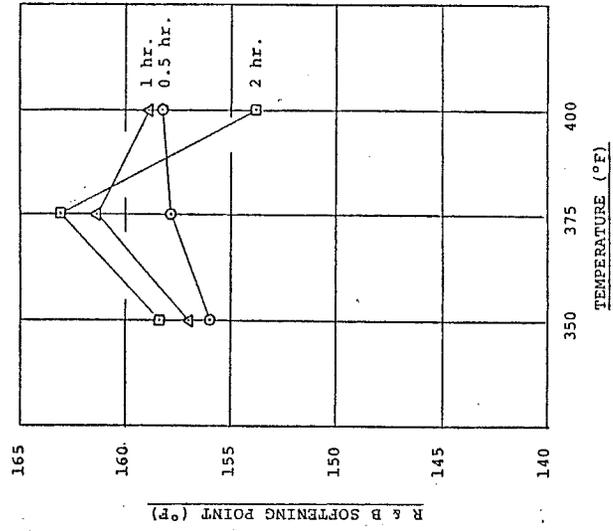


FIGURE A-2

R & B SOFT. VS TEMP.

TABLE B-2  
ANOVA  
ABSOLUTE VISCOSITY (140F)  
All Data

FOSTER & BURR q:

|                          |                   |   |       |       |   |       |       |
|--------------------------|-------------------|---|-------|-------|---|-------|-------|
| Matrix Lx <sub>1</sub>   | 5.265597000E + 06 | q | 0.999 | 0.287 | q | 0.999 | 0.363 |
| Matrix Lx <sub>1,2</sub> | 1.177345470E + 12 | v | 3.00  |       |   |       |       |
|                          |                   | q | 0.483 |       |   |       |       |

$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon(ijk)$

TOTALS

Temperature (T<sub>i</sub>)

Time (H<sub>j</sub>)

|                  |   |  |
|------------------|---|--|
| T <sub>1..</sub> | = |  |
| T <sub>2..</sub> | = |  |
| T <sub>3..</sub> | = |  |

Temperature x Time (TH)<sub>ij</sub>

|                  |   |  |
|------------------|---|--|
| T <sub>11.</sub> | = |  |
| T <sub>12.</sub> | = |  |
| T <sub>13.</sub> | = |  |

|                  |   |  |
|------------------|---|--|
| T <sub>21.</sub> | = |  |
| T <sub>22.</sub> | = |  |
| T <sub>23.</sub> | = |  |

|                  |   |  |
|------------------|---|--|
| T <sub>31.</sub> | = |  |
| T <sub>32.</sub> | = |  |
| T <sub>33.</sub> | = |  |

T...

TTY<sub>ijk</sub>

TABLE B-1  
ABSOLUTE VISCOSITY (140F)  
All Data

DATA MATRIX

| TIME   | TEMPERATURE                              |   |  | Totals    |
|--------|--|---|--|-----------|
|        | 350                                      | 375   | 400  |           |
| 0.5    | 91,257<br>80,402<br>45,934<br>27,577     | 138,699<br>115,924<br>93,152<br>54,305<br>36,755<br>36,922    | 118,100<br>89,400<br>60,700<br>192,062<br>153,996<br>115,930 | 1,055,036 |
| 1.0    | 67,955<br>39,609<br>119,580<br>70,469    | 80,275<br>53,782<br>103,441<br>210,284<br>95,025              | 321,130<br>315,980<br>202,898<br>175,697<br>148,495          | 1,875,258 |
| 2.0    | 133,217<br>112,890<br>228,928<br>169,667 | 228,988<br>123,054<br>229,612<br>127,319<br>199,298<br>87,894 | 477,016<br>459,450<br>441,883<br>44,438<br>107,607<br>53,451 | 2,335,303 |
| Totals | 1,187,485                                | 1,586,029   | 2,492,083  | 5,265,597 |

|       |        |        |      |
|-------|--------|--------|------|
| Upper | 91,257 | 85,829 | Mean |
| Lower | 80,402 |        |      |

Range: 477,016 - 27,577

CELL MEANS

| TIME | TEMPERATURE |         |         |
|------|-------------|---------|---------|
|      | 350         | 375     | 400     |
| 0.5  | 61,292      | 80,768  | 121,698 |
| 1.0  | 74,403      | 147,286 | 247,126 |
| 2.0  | 161,176     | 168,453 | 254,197 |

TABLE B-4  
ANOVA  
ABSOLUTE VISCOSITY (140F)  
All Data (Log<sub>10</sub> Transform)

FOSTER & BURR  $\sigma$ :

Matrix  $Lx_1$  = 182.196

Matrix  $Lx_1^2$  = 925.410

$v$  = 3.0

$q$  = 0.277

$q_{0.99}$  = 0.287

$q_{0.999}$  = 0.363

$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon_{(ij)k}$

TOTALS

Temperature ( $T_i$ )

|           |   |         |
|-----------|---|---------|
| $T_{1..}$ | = | 59.092  |
| $T_{2..}$ | = | 60.716  |
| $T_{3..}$ | = | 62.388  |
| $T_{...}$ | = | 182.196 |

Time ( $H_j$ )

|           |   |         |
|-----------|---|---------|
| $T_{.1.}$ | = | 58.513  |
| $T_{.2.}$ | = | 61.375  |
| $T_{.3.}$ | = | 62.238  |
| $T_{...}$ | = | 182.196 |

Temperature x Time (TH)  $_{ij}$

|           |   |        |
|-----------|---|--------|
| $T_{11.}$ | = | 18.968 |
| $T_{12.}$ | = | 19.356 |
| $T_{13.}$ | = | 20.768 |

|           |   |        |
|-----------|---|--------|
| $T_{21.}$ | = | 19.413 |
| $T_{22.}$ | = | 20.533 |
| $T_{23.}$ | = | 20.770 |

|           |   |         |
|-----------|---|---------|
| $T_{31.}$ | = | 20.202  |
| $T_{32.}$ | = | 21.486  |
| $T_{33.}$ | = | 20.700  |
| $T_{...}$ | = | 182.196 |

$T_{...}$  = 182.196

$TEY_{ijk}$  =

TABLE B-3  
LOG<sub>10</sub> ABSOLUTE VISCOSITY (140F)  
All Data

TRANSFORMED DATA MATRIX (LOG<sub>10</sub>)

| TIME   | TEMPERATURE |        |        | Totals  |
|--------|-------------|--------|--------|---------|
|        | 350         | 375    | 400    |         |
| 0.5    | 4.960       | 5.142  | 5.072  | 58.583  |
|        | 4.905       | 4.969  | 4.783  |         |
|        | 4.662       | 4.735  | 5.283  |         |
| 1.0    | 4.441       | 4.567  | 5.064  | 61.375  |
|        | 4.832       | 4.905  | 5.507  |         |
|        | 4.598       | 5.015  | 5.500  |         |
| 2.0    | 5.078       | 5.323  | 5.307  | 62.238  |
|        | 4.848       | 5.290  | 5.172  |         |
|        | 5.125       | 5.360  | 5.679  |         |
| Totals | 59.092      | 60.716 | 62.388 | 182.196 |

CELL MEANS

| TIME   | TEMPERATURE |       |       | Totals |
|--------|-------------|-------|-------|--------|
|        | 350         | 375   | 400   |        |
| 0.5    | 4.742       | 4.853 | 5.051 | 4.882  |
| 1.0    | 4.839       | 5.133 | 5.372 | 5.116  |
| 2.0    | 5.192       | 5.193 | 5.175 | 5.187  |
| Totals | 4.924       | 5.060 | 5.199 | 5.061  |

TABLE B-5  
ANOVA  
LOG<sub>10</sub> ABSOLUTE VISCOSITY  
All Data

(0)  $CT = (T \dots)^2 + n = (182.196)^2 + (36) = 922.094$

(1)  $SS (Temp.) = \frac{1}{(12)} [(59.092)^2 + \dots + (62.388)^2] - CT = 0.453$

(2)  $SS (Time) = \frac{1}{(12)} [(58.583)^2 + \dots + (62.238)^2] - CT = 0.608$

(3)  $SS (H \times T) = \frac{1}{(4)} [(18.968)^2 + \dots + (20.700)^2] - CT - SS (T) - SS (H)$

$= \frac{0.313}{}$

(4)  $SS (Total) = \sum \sum x_{ijk}^2 - CT = 3.316$

(5)  $SS (Error) = SS (Total) - SS (T) - SS (H) - SS (T \times H)$

$= (4) - (1) - (2) - (3) = 1.942$

| ANALYSIS OF VARIANCE   |    |       |       |       |      |      |
|------------------------|----|-------|-------|-------|------|------|
| Source                 | df | SS    | MS    | F     | F.05 | F.01 |
| (1) T <sub>i</sub>     | 2  | 0.453 | 0.226 | 3.146 | 3.35 | 5.40 |
| (2) H <sub>j</sub>     | 2  | 0.608 | 0.304 | 4.228 | 3.35 | 5.49 |
| (3) (TH) <sub>ij</sub> | 4  | 0.313 | 0.078 | 1.086 | 2.73 | 4.11 |
| (5) Error              | 27 | 1.942 | 0.072 |       |      |      |
| (4) Total              | 35 | 3.316 |       |       |      |      |

TABLE B-6  
LOG<sub>10</sub> ABSOLUTE VISCOSITY  
All Data  
Newman-Keuls

Cell Means: Table B-3  
Ms (error): Table B-5

$s_{\bar{y}_{ij}} = \sqrt{\frac{MS (error)}{n}} = \sqrt{\frac{0.072}{4}} = 0.1342$

$R_k = q_{\alpha, s} (k, df) s_{\bar{y}_{ij}}$ ;  $df = 27$  [from Ms (error)]

| $\bar{y}_k$ | (k, df) | $\frac{R_k}{s_{\bar{y}_{ij}}}$ | (Appendix 6, Ref. (13)) |
|-------------|---------|--------------------------------|-------------------------|
| 9           | 4, 764  | 0.639                          |                         |
| 8           | 4, 643  | 0.623                          |                         |
| 7           | 4, 503  | 0.604                          |                         |
| 6           | 4, 338  | 0.582                          |                         |
| 5           | 4, 134  | 0.555                          |                         |
| 4           | 3, 873  | 0.520                          |                         |
| 3           | 3, 509  | 0.471                          |                         |
| 2           | 2, 904  | 0.390                          |                         |

TABLE B-7  
 LOG<sub>10</sub> ABSOLUTE VISCOSITY  
 All Data

Newman-Keuls, Continued

| RANK   | 1     | 2     | 3     | 4     | 5     | 6     | 7     | 8     | 9     |
|--------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| (i, j) | 3,2   | 2,3   | 1,3   | 3,3   | 2,2   | 3,1   | 2,1   | 1,2   | 1,1   |
| Mean   | 5.372 | 5.193 | 5.192 | 5.175 | 5.133 | 5.051 | 4.853 | 4.839 | 4.742 |

| RANK | K    | K-1   | K-2   | K-3   | K-4   | K-5   | K-6   | K-7   | K-8 |
|------|------|-------|-------|-------|-------|-------|-------|-------|-----|
| 1    | 0.64 | 0.630 | 0.533 | 0.321 | 0.239 | 0.197 | 0.180 | 0.179 | 0   |
| 2    | 0.62 | 0.451 | 0.354 | 0.340 | 0.142 | 0.060 | 0.018 | 0     |     |
| 3    | 0.60 | 0.450 | 0.353 | 0.339 | 0.141 | 0.059 | 0.017 | 0     |     |
| 4    | 0.58 | 0.433 | 0.336 | 0.322 | 0.124 | 0.042 | 0     |       |     |
| 5    | 0.56 | 0.391 | 0.294 | 0.280 | 0.092 | 0     |       |       |     |
| 6    | 0.52 | 0.309 | 0.212 | 0.198 | 0     |       |       |       |     |
| 7    | 0.47 | 0.111 | 0.014 | 0     |       |       |       |       |     |
| 8    | 0.39 | 0.097 | 0     |       |       |       |       |       |     |

No Significant Differences

LOG<sub>10</sub> ABSOLUTE VISCOSITY  
 All Data, Cell Means

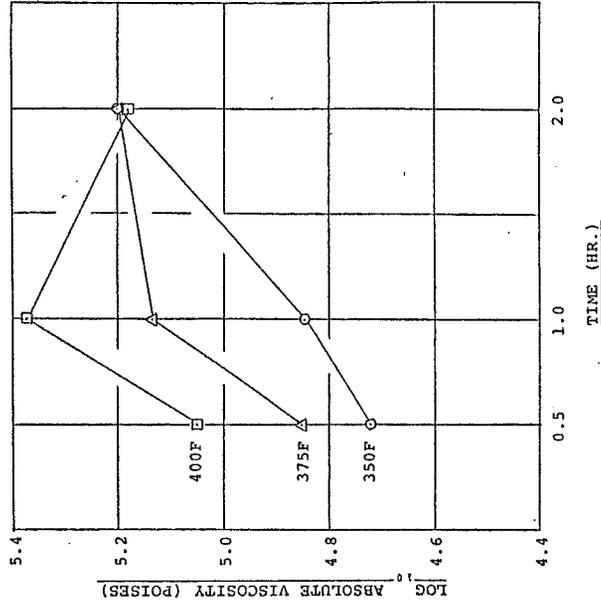


FIGURE B-1  
 LOG<sub>10</sub> ABSOLUTE VISCOSITY  
 VS  
 TIME

LOG<sub>10</sub> ABSOLUTE VISCOSITY  
All Data, Cell Means

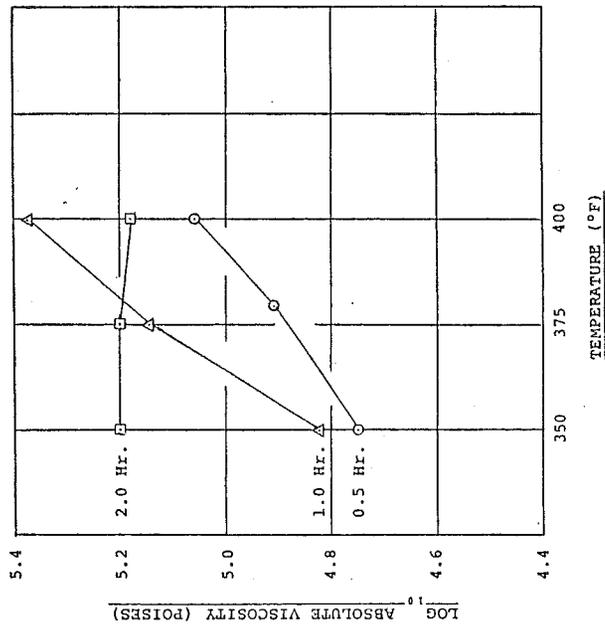


FIGURE B-2  
LOG<sub>10</sub> ABSOLUTE VISCOSITY  
vs  
TEMPERATURE

TABLE B-8  
LOG<sub>10</sub> ABSOLUTE VISCOSITY  
Mean of Runs

DATA MATRIX: (Entries calculated from raw data matrix, Table B-1)

| TIME   | TEMPERATURE |        |        | Totals |
|--------|-------------|--------|--------|--------|
|        | 350         | 375    | 400    |        |
| 0.5    | 4.934       | 5.064  | 4.951  | 29.361 |
| 1.0    | 4.731       | 4.963  | 5.503  | 30.727 |
| 2.0    | 5.090       | 5.360  | 5.662  | 31.134 |
| Totals | 29.598      | 30.385 | 31.239 | 91.222 |

CELL MEANS

| TIME | TEMPERATURE |       |       |
|------|-------------|-------|-------|
|      | 350         | 375   | 400   |
| 0.5  | 4.750       | 4.862 | 5.070 |
| 1.0  | 4.855       | 5.135 | 5.374 |
| 2.0  | 5.195       | 5.196 | 5.176 |

TABLE B-9  
ANOVA  
LOG<sub>10</sub> ABSOLUTE VISCOSITY  
Mean of Runs

FOSTER & BURR q:

Matrix Ex<sub>1</sub> = 91.222

Matrix Ex<sub>2</sub> = 463.816

v = 1.0

q = 0.332

q<sub>0.999</sub> = 0.576

q<sub>0.999</sub> = 0.750

$$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon_{(ijk)}$$

TOTALS

Temperature (T<sub>i</sub>)

Time (H<sub>j</sub>)

T<sub>1..</sub> = 29.598

T<sub>2..</sub> = 30.385

T<sub>3..</sub> = 31.239

T<sub>..1</sub> = 29.361

T<sub>..2</sub> = 30.727

T<sub>..3</sub> = 31.134

T<sub>..</sub> = 91.222

Temperature x Time (TH)<sub>ij</sub>

T<sub>11.</sub> = 9.499

T<sub>12.</sub> = 9.709

T<sub>13.</sub> = 10.390

T<sub>21.</sub> = 9.723

T<sub>22.</sub> = 10.270

T<sub>23.</sub> = 10.392

T<sub>31.</sub> = 10.139

T<sub>32.</sub> = 10.748

T<sub>33.</sub> = 10.352

T<sub>..</sub> = 91.222

T.... = 91.222

TEY<sub>ijk</sub> =

TABLE B-10  
ANOVA  
LOG<sub>10</sub> ABSOLUTE VISCOSITY  
Mean of Runs

- (0) CT = (T....)<sup>2</sup> ÷ n = (91.222)<sup>2</sup> ÷ ( 18 ) = 462.303
- (1) SS (Temp.) =  $\frac{1}{(6)} [(29.598)^2 + \dots + (31.239)^2] - CT = 0.245$
- (2) SS (Time) =  $\frac{1}{(6)} [(29.361)^2 + \dots + (31.134)^2] - CT = 0.308$
- (3) SS (H x T) =  $\frac{1}{(2)} [(9.499)^2 + \dots + (10.352)^2] - CT - SS (T) - SS (H)$   
= 0.132
- (4) SS (Total) =  $\sum \sum x_{ijk}^2 - CT = 1.533$
- (5) SS (Error) = SS (Total) - SS (T) - SS (H) - SS (T x H)  
= (4) - (1) - (2) - (3) = 0.849

| ANALYSIS OF VARIANCE |    |       |       |       |      |      |
|----------------------|----|-------|-------|-------|------|------|
| Source               | df | SS    | MS    | F     | F.05 | F.01 |
| T <sub>i</sub>       | 2  | 0.245 | 0.122 | 1.297 | 4.36 |      |
| H <sub>j</sub>       | 2  | 0.308 | 0.154 | 1.631 | 4.26 |      |
| (TH) <sub>ij</sub>   | 4  | 0.132 | 0.033 | 0.34  | 3.63 |      |
| Error                | 9  | 0.849 | 0.094 |       |      |      |
| Total                | 17 | 1.533 |       |       |      |      |

TABLE B-11  
 LOG<sub>10</sub> ABSOLUTE VISCOSITY  
 Mean of Runs  
 Newman-Keuls

Cell Means: Table B-5; MS(error): Table B-10

$$S_{ij} = \sqrt{0.094/2} = 0.2168$$

| $\bar{y}_k$ | (K, 9) | $R_k$ | $\bar{y}_k$ | (K, 9) | $R_k$ |
|-------------|--------|-------|-------------|--------|-------|
| 9           | 5.595  | 1.21  | 5           | 4.756  | 1.03  |
| 8           | 5.432  | 1.18  | 4           | 4.415  | 0.96  |
| 7           | 5.244  | 1.14  | 3           | 3.949  | 0.86  |
| 6           | 5.024  | 1.09  | 2           | 3.199  | 0.69  |

| RANK   | 1     | 2     | 3     | 4     | 5     | 6     | 7     | 8     | 9     |
|--------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| (i, j) | 3,2   | 2,3   | 1,3   | 3,3   | 2,2   | 3,1   | 2,1   | 1,2   | 1,1   |
| Mean   | 5.372 | 5.196 | 5.195 | 5.176 | 5.135 | 5.070 | 4.862 | 4.855 | 4.750 |

| RANK | LEAST RANGE     | K     |       |       |       |       |       |       |       | K-8 |
|------|-----------------|-------|-------|-------|-------|-------|-------|-------|-------|-----|
|      |                 | K-1   | K-2   | K-3   | K-4   | K-5   | K-6   | K-7   |       |     |
| 1    | 1 <sup>21</sup> | 0.624 | 0.519 | 0.512 | 0.304 | 0.239 | 0.198 | 0.179 | 0.178 | 0   |
| 2    | 1 <sup>18</sup> | 0.446 | 0.341 | 0.334 | 0.126 | 0.061 | 0.020 | 0.001 | 0     |     |
| 3    | 1 <sup>14</sup> | 0.445 | 0.340 | 0.333 | 0.125 | 0.060 | 0.019 | 0     |       |     |
| 4    | 1 <sup>09</sup> | 0.426 | 0.321 | 0.314 | 0.106 | 0.041 | 0     |       |       |     |
| 5    | 1 <sup>03</sup> | 0.385 | 0.280 | 0.273 | 0.065 | 0     |       |       |       |     |
| 6    | 0 <sup>96</sup> | 0.320 | 0.215 | 0.208 | 0     |       |       |       |       |     |
| 7    | 0 <sup>86</sup> | 0.112 | 0.007 | 0     |       |       |       |       |       |     |
| 8    | 0 <sup>59</sup> | 0.105 | 0     |       |       |       |       |       |       |     |

No Significant Differences

TABLE C-1  
DUCTILITY @ 39.3F (Cm.)  
All Data

DATA MATRIX: (\*Missing Data by ave.)

| TIME   | TEMPERATURE                                   |  |  | Totals |
|--------|---|--|--|--------|
|        | 350   | 375  | 400  |        |
| 0.5    | 13.5<br>13.5<br>14.0<br>12.0<br>16.0<br>14.0  | 17.0<br>16.5<br>16.5<br>19.5<br>17.0<br>18.0 | 18.0<br>19.5<br>16.0<br>20.0<br>18.5<br>19.5 | 299.0  |
| 1.0    | 16.5<br>17.0<br>15.0<br>14.5<br>16.5<br>*15.5 | 17.0<br>18.0<br>15.5<br>17.5<br>16.5<br>18.0 | 14.5<br>15.5<br>18.0<br>17.0<br>17.0<br>18.5 | 298.0  |
| 2.0    | 14.5<br>16.5<br>16.0<br>14.0<br>16.0          | 17.0<br>18.0<br>15.5<br>20.5<br>20.0<br>19.0 | 18.5<br>15.0<br>16.0<br>22.0<br>24.0<br>20.0 | 319.0  |
| Totals | 271.5   | 317.0  | 327.5  | 916.0  |

CELL MEANS

| TIME   | TEMPERATURE |       |       | Totals |
|--------|-------------|-------|-------|--------|
|        | 350         | 375   | 400   |        |
| 0.5    | 13.83       | 17.42 | 18.58 | 16.61  |
| 1.0    | 15.83       | 17.08 | 16.75 | 16.55  |
| 2.0    | 15.58       | 18.33 | 19.25 | 17.72  |
| Totals | 15.08       | 17.61 | 18.19 | 16.96  |

TABLE C-2  
ANOVA  
DUCTILITY @ 39.2F (Cm.)  
All Data

FOSTER & BURR q:

Matrix  $Lx_i$  \_\_\_\_\_ 216.0 \_\_\_\_\_

Matrix  $Ex_i^2$  \_\_\_\_\_ 15,806.00 \_\_\_\_\_

$v$  \_\_\_\_\_ 5.0 \_\_\_\_\_

$q$  \_\_\_\_\_ 0.256 \_\_\_\_\_  $q_{0.99}$  \_\_\_\_\_ 0.217 \_\_\_\_\_  $q_{0.999}$  \_\_\_\_\_ 0.261 \_\_\_\_\_

$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon(ijk)$

TOTALS

Temperature ( $T_i$ )

|           |   |       |
|-----------|---|-------|
| $T_{1..}$ | = | _____ |
| $T_{2..}$ | = | _____ |
| $T_{3..}$ | = | _____ |

Temperature x Time (TH)  $_{ij}$

|           |   |       |
|-----------|---|-------|
| $T_{11.}$ | = | _____ |
| $T_{12.}$ | = | _____ |
| $T_{13.}$ | = | _____ |
| $T_{21.}$ | = | _____ |
| $T_{22.}$ | = | _____ |
| $T_{23.}$ | = | _____ |
| $T_{31.}$ | = | _____ |
| $T_{32.}$ | = | _____ |
| $T_{33.}$ | = | _____ |

T...

$\sum \sum Y_{ijk}$

TABLE C-3  
LOG<sub>10</sub> DUCTILITY @ 39.2F (Cm.)

All Data

DATA MATRIX:

|        | TEMPERATURE |         |         | Totals  |
|--------|-------------|---------|---------|---------|
|        | 350         | 375     | 400     |         |
| 0.5    | 1.1303      | 1.2304  | 1.2553  | 21.8848 |
|        | 1.1303      | 1.2175  | 1.2900  |         |
|        | 1.1461      | 1.2175  | 1.2041  |         |
| 1.0    | 1.0792      | 1.2500  | 1.3010  | 21.9200 |
|        | 1.2041      | 1.2304  | 1.2672  |         |
|        | 1.1461      | 1.2553  | 1.2900  |         |
| 2.0    | 1.2175      | 1.2304  | 1.1614  | 22.3893 |
|        | 1.2304      | 1.2553  | 1.1903  |         |
|        | 1.1761      | 1.1903  | 1.2553  |         |
| 2.0    | 1.1614      | 1.2430  | 1.2304  | 66.1941 |
|        | 1.2175      | 1.2175  | 1.2304  |         |
|        | 1.1903      | 1.2553  | 1.2672  |         |
| 2.0    | 1.1614      | 1.2304  | 1.2672  | 66.1941 |
|        | 1.2175      | 1.2553  | 1.1761  |         |
|        | 1.2175      | 1.1903  | 1.2041  |         |
| 2.0    | 1.2041      | 1.3118  | 1.3424  | 22.3893 |
|        | 1.1461      | 1.3010  | 1.3802  |         |
|        | 1.2041      | 1.2788  | 1.3010  |         |
| Totals | 21.1800     | 22.4005 | 22.6136 | 66.1941 |

CELL MEANS

|        | TEMPERATURE |        |        | Totals |
|--------|-------------|--------|--------|--------|
|        | 350         | 375    | 400    |        |
| 0.5    | 1.1394      | 1.2402 | 1.2679 | 1.2158 |
|        | 1.1989      | 1.2320 | 1.2225 |        |
| 1.0    | 1.1918      | 1.2613 | 1.2785 | 1.2178 |
|        | 1.1767      | 1.2445 | 1.2563 |        |
| Totals | 1.1767      | 1.2445 | 1.2563 | 1.2258 |

Table C-4  
ANOVA  
LOG<sub>10</sub> DUCTILITY @ 39.2F (Cm.)  
All Data

FOSTER & BURR  $\sigma_i$

Matrix  $\Sigma x_i$  = 66.1941

Matrix  $\Sigma x_i^2$  = 81.3146

$v$  = 5.0

$q$  = 0.207

$q_{0.999}$  = 0.217

$q_{0.999}$  = 0.261

$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon_{(ijk)}$

TOTALS

Temperature ( $T_i$ )

$T_{1..} = 21.1800$

$T_{2..} = 22.4005$

$T_{3..} = 22.6136$

$T_{...} = 66.1941$

Time ( $H_j$ )

$T_{.1.} = 21.8848$

$T_{.2.} = 21.9200$

$T_{.3.} = 22.3893$

$T_{...} = 66.1941$

Temperature x Time (TH)  $ij$

$T_{11.} = 6.8361$

$T_{12.} = 7.1932$

$T_{13.} = 7.1507$

$T_{21.} = 7.4411$

$T_{22.} = 7.3918$

$T_{23.} = 7.5676$

$T_{31.} = 7.6076$

$T_{32.} = 7.3350$

$T_{33.} = 7.6710$

$T_{...} = 66.1941$

$\Sigma Y_{ijk} =$

TABLE C-5  
ANOVA  
LOG<sub>10</sub> DUCTILITY @ 39.2F (Cm.)  
All Data

(0)  $CT = (T \dots)^2 \div n = (66.1941)^2 \div (54) = 8.114183102E + 01$

(1)  $SS (Temp.) = \frac{1}{(18)} [(21.1800)^2 + \dots + (22.6136)^2] - CT = 0.0665$

(2)  $SS (Time) = \frac{1}{(18)} [(21.8848)^2 + \dots + (22.3893)^2] - CT = 0.0088$

(3)  $SS (H \times T) = \frac{1}{(6)} [(6.8361)^2 + \dots + (7.6710)^2] - CT - SS (T) - SS (H)$   
 $= 0.0172$

(4)  $SS (Total) = \sum \sum x_{ijk}^2 - CT = 0.1728$

(5)  $SS (Error) = SS (Total) - SS (T) - SS (H) - SS (T \times H)$   
 $= (4) - (1) - (2) - (3) = 0.0802$

TABLE C-6  
LOG<sub>10</sub> DUCTILITY @ 39.2F (Cm.)  
All Data  
Newman-Keuls

Cell Means: Table C-3  
Ms (Error): Table C-5

$$S^2_{yij} = \frac{Ms(Error)}{6} = \frac{0.018}{6} = 0.003$$

| $\bar{Y}_k$ | $\frac{R_k}{k}$ | $\frac{R_k}{k}$ |
|-------------|-----------------|-----------------|
| 9           | 4.614           | 0.252           |
| 8           | 4.501           | 0.246           |
| 7           | 4.370           | 0.239           |
| 6           | 4.215           | 0.231           |
| 5           | 4.024           | 0.220           |
| 4           | 3.778           | 0.207           |
| 3           | 3.431           | 0.188           |
| 2           | 2.851           | 0.156           |

(Approx. 6, A & McL)  
df = 45

| ANALYSIS OF VARIANCE   |    |        |        |      |      |      |
|------------------------|----|--------|--------|------|------|------|
| Source                 | df | SS     | MS     | F    | F.05 | F.01 |
| (1) T <sub>i</sub>     | 2  | 0.0665 | 0.0332 | 18.6 | 3.21 | 5.13 |
| (2) H <sub>j</sub>     | 2  | 0.0088 | 0.0044 | 2.47 | 3.21 | 5.13 |
| (3) (TH) <sub>ij</sub> | 4  | 0.0172 | 0.0043 | 2.42 | 2.59 | 3.79 |
| (5) Error              | 45 | 0.0802 | 0.0018 |      |      |      |
| (4) Total              | 53 |        |        |      |      |      |

TABLE C-7  
LOG<sub>10</sub> DUCTILITY @ 39.2 (1 cm.)

All Data  
Newman-Keuls, Continued

| RANK   | 1      | 2      | 3      | 4      | 5      | 6      | 7      | 8      | 9      |
|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| (i, j) | 3,3    | 3,1    | 2,3    | 2,1    | 2,2    | 3,2    | 1,2    | 1,3    | 1,1    |
| Mean   | 1.2785 | 1.2679 | 1.2613 | 1.2402 | 1.2320 | 1.2225 | 1.1989 | 1.1918 | 1.1394 |

| RANK | LSR  | k     |       |       |       |       |       |       |       |   |
|------|------|-------|-------|-------|-------|-------|-------|-------|-------|---|
|      |      | k-1   | k-2   | k-3   | k-4   | k-5   | k-6   | k-7   | k-8   |   |
| 1    | 0.25 | 0.139 | 0.087 | 0.080 | 0.056 | 0.047 | 0.038 | 0.017 | 0.011 | 0 |
| 2    | 0.25 | 0.129 | 0.076 | 0.069 | 0.045 | 0.036 | 0.028 | 0.007 | 0     |   |
| 3    | 0.24 | 0.122 | 0.070 | 0.062 | 0.039 | 0.029 | 0.021 | 0     |       |   |
| 4    | 0.23 | 0.101 | 0.048 | 0.041 | 0.018 | 0.008 | 0     |       |       |   |
| 5    | 0.22 | 0.093 | 0.040 | 0.033 | 0.010 | 0     |       |       |       |   |
| 6    | 0.21 | 0.083 | 0.031 | 0.024 | 0     |       |       |       |       |   |
| 7    | 0.19 | 0.060 | 0.007 | 0     |       |       |       |       |       |   |
| 8    | 0.16 | 0.052 | 0     |       |       |       |       |       |       |   |

No Significant Differences

TABLE C-8  
LOG<sub>10</sub> DUCTILITY @ 39.2 (1 cm.)  
TEMPERATURE  
NEWMAN - KEULS

One-Way Anova of Temp. (q = 0.396 < q crit.)

| ANALYSIS OF VARIANCE |    |        |        |       |      |      |
|----------------------|----|--------|--------|-------|------|------|
| Source               | df | SS     | MS     | F     | F.01 |      |
| Treat.               | 2  | 0.0665 | 0.0332 | 16.09 | 3.19 | 5.07 |
| Error                | 51 | 0.1053 | 0.0021 |       |      |      |
| Total                | 53 | 0.1718 |        |       |      |      |

$$S\bar{Y} = \sqrt{\frac{0.396}{18}} = 0.0108$$

| RANK | 1      | 2      | 3      |
|------|--------|--------|--------|
| (i)  | 3      | 2      | 1      |
| Mean | 1.2563 | 1.2445 | 1.1767 |

| RANK | *LSR   |        |
|------|--------|--------|
|      | k      | k-1    |
| 1    | 0.0369 | 0.0796 |
| 2    | 0.0307 | 0.0678 |

\*Least Significant Range

D<sub>400</sub> greater than D<sub>350</sub>

D<sub>375</sub> greater than D<sub>350</sub>

LOG<sub>10</sub> DUCTILITY @ 39.3F (Cm.)  
All Data, Cell Means

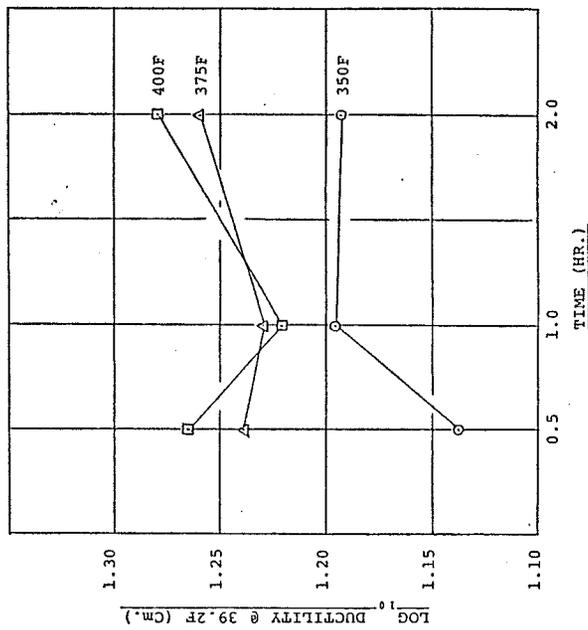


FIGURE C-1  
LOG<sub>10</sub> DUCTILITY @ 39.2F  
VS  
TIME

LOG<sub>10</sub> DUCTILITY @ 39.2F (Cm.)  
All Data, Cell Means

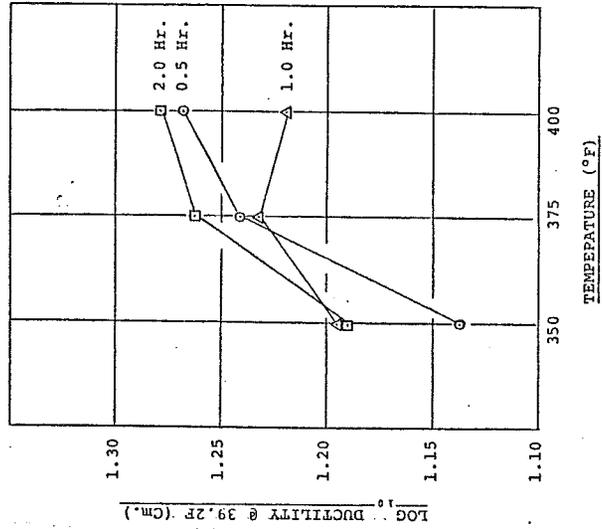


FIGURE C-2  
LOG<sub>10</sub> DUCTILITY @ 39.2F  
VS  
TEMPERATURE

TABLE C-9  
 DUCTILITY @ 39.2F (Cm.)  
 Means of Runs

DATA MATRIX: (Each entry is mean of three specimens.)

| TIME   | TEMPERATURE |        |        | Totals |
|--------|-------------|--------|--------|--------|
|        | 350         | 375    | 400    |        |
| 0.5    | 13.67       | 16.67  | 17.83  | 99.67  |
| 1.0    | 14.00       | 18.17  | 19.33  | 99.33  |
| 1.0    | 16.17       | 16.83  | 16.00  | 99.33  |
| 1.0    | 15.50       | 17.33  | 17.50  | 99.33  |
| 2.0    | 15.83       | 16.83  | 16.50  | 106.32 |
| 2.0    | 15.33       | 19.83  | 22.00  | 106.32 |
| Totals | 90.50       | 105.66 | 109.16 | 305.32 |

CELL MEANS

| TIME | TEMPERATURE |       |       |
|------|-------------|-------|-------|
|      | 350         | 375   | 400   |
| 0.5  | 13.84       | 17.42 | 18.58 |
| 1.0  | 15.84       | 17.08 | 16.75 |
| 2.0  | 15.58       | 18.33 | 19.25 |

Table C-10  
 ANOVA  
 DUCTILITY @ 39.2F (Cm.)  
 Means of Runs

FOSTER & BURR  $q_1$

|                    |                   |
|--------------------|-------------------|
| Matrix $\{x_i\}$   | 305.32            |
| Matrix $\{x_i\}^2$ | 5248.337          |
| $v$                | 1.0               |
| $q$                | 0.457             |
|                    | $q_{0.99}$ 0.576  |
|                    | $q_{0.999}$ 0.750 |

$$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon(ijk)$$

TOTALS

Temperature ( $T_i$ )

|           |          |
|-----------|----------|
| $T_{1..}$ | = 90.50  |
| $T_{2..}$ | = 105.66 |
| $T_{3..}$ | = 109.16 |
|           | = 305.32 |

Time ( $H_j$ )

|           |          |
|-----------|----------|
| $T_{.1.}$ | = 99.67  |
| $T_{.2.}$ | = 99.33  |
| $T_{.3.}$ | = 106.32 |
|           | = 305.32 |

Temperature x Time ( $TH$ )<sub>ij</sub>

|           |          |
|-----------|----------|
| $T_{11.}$ | = 27.67  |
| $T_{12.}$ | = 31.67  |
| $T_{13.}$ | = 31.16  |
| $T_{21.}$ | = 34.84  |
| $T_{22.}$ | = 34.16  |
| $T_{23.}$ | = 36.66  |
| $T_{31.}$ | = 37.16  |
| $T_{32.}$ | = 33.50  |
| $T_{33.}$ | = 38.50  |
|           | = 305.32 |

|                  |          |
|------------------|----------|
| $T_{...}$        | = 305.32 |
| $\Sigma Y_{ijk}$ | =        |

TABLE C-11  
ANOVA  
DUCTILITY @ 39.2F  
Mean of Runs

(0)  $CT = (T...)^2 \div n = (305.32)^2 \div (18) = 5.178905689E + 03$

(1)  $SS (Temp.) = \frac{1}{(6)} [(90.50)^2 + \dots + (109.16)^2] - CT = 32.79$

(2)  $SS (Time) = \frac{1}{(6)} [(99.67)^2 + \dots + (106.32)^2] - CT = 5.18$

(3)  $SS (T \times H) = \frac{1}{(2)} [(27.67)^2 + \dots + (38.50)^2] - CT - SS (T) - SS (H)$   
 $= 7.93$

(4)  $SS (Total) = \sum \sum x_{ijk}^2 - CT = 69.43$

(5)  $SS (Error) = SS (Total) - SS (T) - SS (H) - SS (T \times H)$   
 $= (4) - (1) - (2) - (3) = 23.53$

| ANALYSIS OF VARIANCE |    |       |       |      |      |      |
|----------------------|----|-------|-------|------|------|------|
| Source               | df | SS    | MS    | F    | F.05 | F.01 |
| (1) T                | 2  | 32.79 | 16.40 | 6.27 | 4.26 | 8.02 |
| (2) H                | 2  | 5.18  | 2.59  | 0.99 | 4.26 | 8.02 |
| (3) (TH) ij          | 4  | 7.93  | 1.98  | 0.76 | 3.63 | 6.42 |
| (5) Error            | 9  | 23.53 | 2.61  |      |      |      |
| (4) Total            | 17 | 69.43 |       |      |      |      |

TABLE C-12  
DUCTILITY @ 39.2F (Cm.)  
Means of Runs  
Newman-Keuls

Cell Means: Table C-9; Ms(Error): Table C-11

$S_{Yij} = \sqrt{2.61/2} = 1.14$

| $\bar{Y}$ | (k, df) | $\frac{R_k - \bar{Y}}{S_{Yij}}$ | $\bar{Y}$ | (k, df) | $\frac{R_k - \bar{Y}}{S_{Yij}}$ |
|-----------|---------|---------------------------------|-----------|---------|---------------------------------|
| 9         | 5, 995  | 6.39                            | 5         | 4, 756  | 5.43                            |
| 8         | 5, 432  | 6.21                            | 4         | 4, 415  | 5.04                            |
| 7         | 5, 244  | 5.99                            | 3         | 3, 949  | 4.51                            |
| 6         | 5, 024  | 5.74                            | 2         | 3, 199  | 3.65                            |

| RANK   | 1     | 2     | 3     | 4     | 5     | 6     | 7     | 8     | 9     |
|--------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| (i, j) | 3, 3  | 3, 1  | 2, 3  | 2, 1  | 2, 2  | 3, 2  | 1, 2  | 1, 3  | 1, 1  |
| Mean   | 19.25 | 18.58 | 18.33 | 17.42 | 17.08 | 16.75 | 15.84 | 15.58 | 13.84 |

| RANK | MSR  |      |      |      |      |      |      |      |      |
|------|------|------|------|------|------|------|------|------|------|
|      | k    | k-1  | k-2  | k-3  | k-4  | k-5  | k-6  | k-7  | k-8  |
| 1    | 6.39 | 5.41 | 3.67 | 3.41 | 2.50 | 2.17 | 1.83 | 0.92 | 0.67 |
| 2    | 6.21 | 4.74 | 3.00 | 2.74 | 1.83 | 1.50 | 1.16 | 0.25 | 0    |
| 3    | 5.99 | 4.49 | 2.75 | 2.49 | 1.58 | 1.25 | 0.91 | 0    |      |
| 4    | 5.74 | 3.58 | 1.84 | 1.58 | 0.67 | 0.34 | 0    |      |      |
| 5    | 5.43 | 3.24 | 1.50 | 1.24 | 0.33 | 0    |      |      |      |
| 6    | 5.04 | 2.91 | 1.17 | 0.91 | 0    |      |      |      |      |
| 7    | 4.51 | 2.00 | 0.26 | 0    |      |      |      |      |      |
| 8    | 3.65 | 1.74 | 0    |      |      |      |      |      |      |

TABLE D-2  
DUCTILITY AT 77F

| Source | df | ANALYSIS OF VARIANCE |       |      |      |
|--------|----|----------------------|-------|------|------|
|        |    | SS                   | MS    | F    | F.01 |
| Treat. | 8  | 105.82               | 13.23 | 2.36 | 2.64 |
| Error  | 15 | 83.92                | 5.59  |      | 4.00 |
| Total  | 23 | 189.74               |       |      |      |

TABLE D-1  
DUCTILITY AT 77F

Data Matrix

| Time | Temperature |      |      |  |
|------|-------------|------|------|--|
|      | 350         | 375  | 400  |  |
| 0.5  | 8.5         | 15   | 17   |  |
|      | 10          | 13   | 18.5 |  |
|      | 18          | 13   |      |  |
| 1.0  | 14          | 18   | 16   |  |
|      | 15          | 14   | 21   |  |
|      | 15.5        | 16   |      |  |
| 2.0  | 15          | 16.5 | 18.5 |  |
|      | 17          | 17   | 20   |  |
|      | 18          | 16   |      |  |

Cell Means

| Time | Temperature |      |      |
|------|-------------|------|------|
|      | 350         | 375  | 400  |
| 0.5  | 12.2        | 13.7 | 17.8 |
| 1.0  | 14.8        | 16.0 | 18.5 |
| 2.0  | 16.7        | 16.5 | 19.2 |

TABLE B-1  
MAX. DUCTILITY LOAD, 39.2F, (N)  
All Data

|        | TEMPERATURE  |  |  | Totals  |
|--------|--|--|--|---------|
|        | 350  | 375  | 400  |         |
| 0.5    | 53.44<br>52.95<br>51.49<br>34.90<br>28.56<br>26.61 | 47.10<br>46.12<br>48.56<br>40.76<br>43.19<br>39.78 | 42.71<br>48.56<br>44.17<br>46.12<br>46.12<br>39.78 | 780.92  |
| 1.0    | 38.32<br>43.19<br>42.71<br>51.00<br>51.00<br>38.85 | 55.39<br>56.36<br>51.00<br>59.29<br>53.59<br>62.21 | 55.27<br>54.41<br>51.00<br>48.05<br>46.12<br>49.05 | 907.61  |
| 2.0    | 51.00<br>46.61<br>59.29<br>53.92<br>51.00<br>52.95 | 53.44<br>40.76<br>51.47<br>30.03<br>32.95<br>28.56 | 56.36<br>48.07<br>53.44<br>21.26<br>23.69<br>21.74 | 776.54  |
| Totals | 827.72   | 840.36   | 796.92   | 2465.07 |

DATA MATRIX

ANOVA  
MAX. DUCTILITY LOAD 39.2F, (N)  
All Data

FOSTER & BURR  $\sigma$ :

Matrix  $\Sigma x_i$  2477.22

Matrix  $\Sigma x_i^2$  118,821.694

$v$  5.0

$q$  0.280

$q_{0.99}$  0.217

$q_{0.999}$  0.261

$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon_{(ij)k}$

TOTALS

Temperature ( $T_i$ )

Time ( $H_j$ )

$T_{1..}$  = \_\_\_\_\_  
 $T_{2..}$  = \_\_\_\_\_  
 $T_{3..}$  = \_\_\_\_\_

$T_{.1.}$  = \_\_\_\_\_  
 $T_{.2.}$  = \_\_\_\_\_  
 $T_{.3.}$  = \_\_\_\_\_

Temperature x Time ( $TH$ )<sub>ij</sub>

$T_{11.}$  = \_\_\_\_\_  
 $T_{12.}$  = \_\_\_\_\_  
 $T_{13.}$  = \_\_\_\_\_

$T_{21.}$  = \_\_\_\_\_  
 $T_{22.}$  = \_\_\_\_\_  
 $T_{23.}$  = \_\_\_\_\_

$T_{31.}$  = \_\_\_\_\_  
 $T_{32.}$  = \_\_\_\_\_  
 $T_{33.}$  = \_\_\_\_\_

$T_{...}$  = \_\_\_\_\_  
 $\Sigma Y_{ijk}$  = \_\_\_\_\_

CELL MEANS

|     | TEMPERATURE |       |       |
|-----|-------------|-------|-------|
|     | 350         | 375   | 400   |
| 0.5 | 41.33       | 44.25 | 44.58 |
| 1.0 | 42.51       | 56.27 | 50.82 |
| 2.0 | 52.46       | 39.54 | 37.43 |

TABLE E-2  
 MAX. DUCTILITY LOAD, 39.2F (N)  
 $\sqrt{B} - \sqrt{B-x}$  TRANSFORM  
 All Data

FOSTER & BURR q:  
 Matrix  $\Sigma x_i$  216.773  
 Matrix  $\Sigma x_i^2$  954.471485  
 v 5.0  
 q 0.206  
 q<sub>0.99</sub> 0.217  
 q<sub>0.999</sub> 0.261

$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon(ijk)$

TOTALS

Temperature (T<sub>i</sub>)

|                  |   |         |
|------------------|---|---------|
| T <sub>1..</sub> | = | 72.227  |
| T <sub>2..</sub> | = | 75.840  |
| T <sub>3..</sub> | = | 68.706  |
| T <sub>...</sub> | = | 216.773 |

Time (H<sub>j</sub>)

|                   |   |         |
|-------------------|---|---------|
| T <sub>.1.</sub>  | = | 55.260  |
| T <sub>.2.</sub>  | = | 83.678  |
| T <sub>.3.</sub>  | = | 67.835  |
| T <sub>....</sub> | = | 216.773 |

Temperature x Time (TH)<sub>ij</sub>

|                  |   |         |
|------------------|---|---------|
| T <sub>11.</sub> | = | 21.051  |
| T <sub>12.</sub> | = | 22.129  |
| T <sub>13.</sub> | = | 29.047  |
| T <sub>21.</sub> | = | 22.002  |
| T <sub>22.</sub> | = | 34.267  |
| T <sub>23.</sub> | = | 19.571  |
| T <sub>31.</sub> | = | 22.207  |
| T <sub>32.</sub> | = | 27.282  |
| T <sub>33.</sub> | = | 19.217  |
| T <sub>...</sub> | = | 216.773 |

$\Sigma Y_{ijk} = 216.773$

TABLE E-3  
 ANOVA (MAXIMUM DUCTILITY LOAD)  
 $\sqrt{B} - \sqrt{B-x}$  All Data

FOSTER & BURR q:  
 Matrix  $\Sigma x_i$  216.773  
 Matrix  $\Sigma x_i^2$  954.471485  
 v 5.0  
 q 0.206  
 q<sub>0.99</sub> 0.217  
 q<sub>0.999</sub> 0.261

DATA MATRIX

B = 62.21 = Max. Value  
 x = Measured Value

|        | TEMPERATURE  |  |  | Totals  |
|--------|--|--|--|---------|
|        | 350  | 375  | 400  |         |
| 0.5    | 4.926<br>4.844<br>4.613<br>2.661<br>2.086<br>1.921 | 4.000<br>3.876<br>4.193<br>3.256<br>3.526<br>3.151 | 3.471<br>4.193<br>3.640<br>3.876<br>3.876<br>3.151 | 65.260  |
| 1.0    | 3.000<br>3.526<br>3.471<br>4.539<br>4.539<br>3.054 | 5.276<br>5.469<br>4.539<br>6.179<br>4.817<br>7.887 | 5.253<br>5.094<br>4.539<br>4.280<br>3.876<br>4.260 | 83.678  |
| 2.0    | 4.539<br>6.179<br>5.008<br>4.539<br>4.844          | 4.926<br>3.256<br>4.610<br>2.215<br>2.478<br>2.086 | 5.469<br>4.127<br>4.926<br>1.488<br>1.681<br>1.526 | 67.835  |
| Totals | 72.227   | 75.840   | 68.706   | 216.773 |

CELL MEANS

| TIME | TEMPERATURE |       |       |
|------|-------------|-------|-------|
|      | 350         | 375   | 400   |
| 0.5  | 3.509       | 3.667 | 3.701 |
| 1.0  | 3.688       | 5.711 | 4.547 |
| 2.0  | 4.841       | 3.262 | 3.203 |

TABLE E-4  
ANOVA (MAXIMUM DUCTILITY LOAD)

$\sqrt{B} - \sqrt{B-x}$  All Data

$$(0) CT = (T \dots)^2 \div n = (216.773)^2 \div (54) = 8.701950654E+02$$

$$(1) SS (Temp.) = \frac{1}{(18)} [(72.227)^2 + \dots + (68.706)^2] - CT = 1.414$$

$$(2) SS (Time) = \frac{1}{(18)} [(65.260)^2 + \dots + (67.835)^2] - CT = 11.053$$

$$(3) SS (H \times T) = \frac{1}{(6)} [(21.051)^2 + \dots + (19.217)^2] - CT - SS (T) - SS (H)$$

$$= 21.448$$

$$(4) SS (Total) = \sum \sum x_{ijk}^2 - CT = 84.276$$

$$(5) SS (Error) = SS (Total) - SS (T) - SS (H) - SS (T \times H)$$

$$= (4) - (1) - (2) - (3) = 50.362$$

| ANALYSIS OF VARIANCE |    |        |       |      |      |
|----------------------|----|--------|-------|------|------|
| Source               | df | SS     | MS    | F    | F.05 |
| (1) $T_i$            | 2  | 1.414  | 0.707 | 0.63 | 3.21 |
| (2) $H_j$            | 2  | 11.053 | 5.526 | 4.94 | 3.21 |
| (3) $(TH)_{ij}$      | 4  | 21.448 | 5.362 | 4.79 | 2.59 |
| (5) Error            | 45 | 50.362 | 1.119 |      |      |
| (4) Total            | 84 | 84.276 |       |      |      |

MAX. DUCTILITY LOAD 39.2F (N)  
 $\sqrt{B} - \sqrt{B-x}$  Transform (All Data)  
B = 62.21 = Max., All Data; x = Max. Load

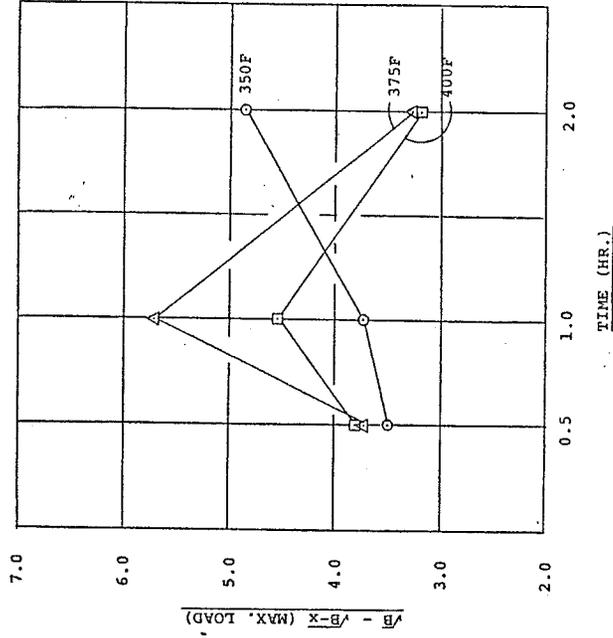


FIGURE E-1  
MAX. DUCTILITY LOAD, 39.2F  
 $\sqrt{B} - \sqrt{B-x}$  TRANSFORMATION  
VS TIME

MAX. DUCTILITY LOAD, 39.2F (N)  
 $\sqrt{B} - \sqrt{B-X}$  Transform (All Data)  
 B = 62.21 = Max., All Data; x = Max. Load

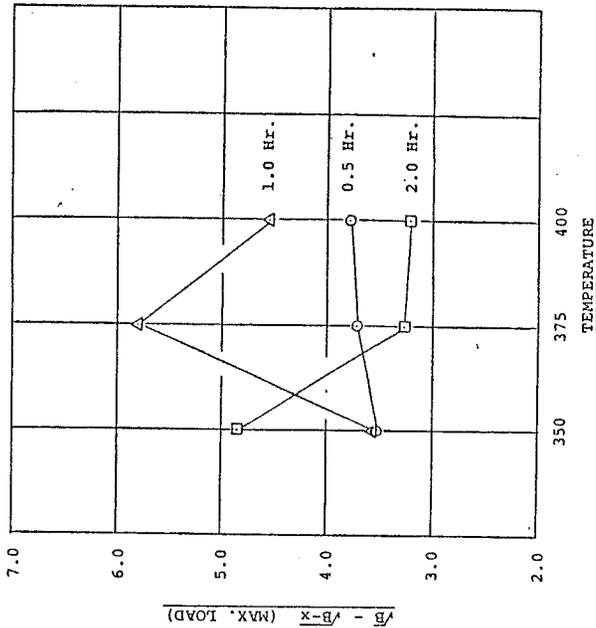


FIGURE E-2  
 MAX. DUCTILITY LOAD, 39.2F  
 $\sqrt{B} - \sqrt{B-X}$  Transformation  
 VS TIME

TABLE E-5  
 MAX. DUCTILITY LOAD, 39.2F (N)  
 $\sqrt{B} - \sqrt{B-X}$  Transform (All Data)  
 Newman-Keuls  
 Cell Means, E-1; Ms(Error), E-4  
 $S_{Yij} = \sqrt{1.119/6} = 0.4319$

| $\bar{Y}$ | (k, df) | $\frac{R_k}{k}$ | $\bar{Y}$ | (k, df) | $\frac{R_k}{k}$ |
|-----------|---------|-----------------|-----------|---------|-----------------|
| 9         | 5-595   | 2.416           | 5         | 4-756   | 2.054           |
| 8         | 5-432   | 2.346           | 4         | 4-415   | 1.907           |
| 7         | 5-244   | 2.265           | 3         | 3-949   | 1.705           |
| 6         | 5-024   | 2.170           | 2         | 3-199   | 1.382           |

| RANK (i,j) | 1     | 2     | 3     | 4     | 5     | 6     | 7     | 8     | 9     |
|------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| Mean       | 5.711 | 4.841 | 4.547 | 3.701 | 3.688 | 3.667 | 3.509 | 3.262 | 3.203 |

| RANK | 1    | 2     | 3     | 4     | 5     | 6     | 7     | 8     | 9     |
|------|------|-------|-------|-------|-------|-------|-------|-------|-------|
| Mean | 2.42 | 2.508 | 2.449 | 2.202 | 2.044 | 2.023 | 2.010 | 1.164 | 0.870 |
| Mean | 2.35 | 1.638 | 1.579 | 1.332 | 1.174 | 1.153 | 1.140 | 0.294 | 0     |
| Mean | 2.26 | 1.344 | 1.285 | 1.038 | 0.880 | 0.859 | 1.846 | 0     |       |
| Mean | 2.17 | 0.498 | 0.439 | 0.192 | 0.034 | 0.013 | 0     |       |       |
| Mean | 2.05 | 0.485 | 0.426 | 0.179 | 0.021 | 0     |       |       |       |
| Mean | 1.91 | 0.464 | 0.405 | 0.158 | 0     |       |       |       |       |
| Mean | 1.70 | 0.306 | 0.247 | 0     |       |       |       |       |       |
| Mean | 1.39 | 0.059 | 0     |       |       |       |       |       |       |

Cell 2,2 Greater Than All Others.

TABLE F-2  
ANOVA  
FORCE - DUCTILITY ENERGY (N-M)  
All Data

FOSTER & BURR q:

Matrix  $\Sigma x_i$  302.86  
Matrix  $\Sigma x_i^2$  1773.512400

v 5.0

q 0.174

q<sub>0.99</sub> 0.217

q<sub>0.999</sub> 0.261

TABLE F-1  
FORCE - DUCTILITY ENERGY (N-M)  
All Data

DATA MATRIX: (\*Mean, missing data)

| TIME   | TEMPERATURE                                   |  |  | Totals |
|--------|---|--|--|--------|
|        | 350   | 375  | 400  |        |
| 0.5    | 5.34<br>5.46<br>5.61<br>3.49<br>3.59<br>2.98  | 6.26<br>5.49<br>5.79<br>6.04<br>5.32<br>5.34 | 5.56<br>6.73<br>4.85<br>6.14<br>5.72<br>5.51 | 95.22  |
| 1.0    | 4.64<br>5.48<br>4.75<br>5.42<br>6.52<br>5.97* | 6.95<br>7.23<br>5.53<br>7.82<br>6.89<br>8.22 | 5.73<br>5.97<br>7.16<br>5.89<br>5.33<br>6.64 | 111.94 |
| 2.0    | 5.62<br>5.82<br>7.48<br>6.11<br>5.38<br>6.80  | 6.39<br>4.57<br>6.89<br>4.27<br>4.76<br>3.86 | 7.12<br>5.02<br>5.59<br>3.14<br>3.84<br>3.04 | 95.70  |
| Totals | 96.46   | 107.42                                       | 98.98  | 302.86 |

CELL MEANS:

| TIME | TEMPERATURE |       |       |
|------|-------------|-------|-------|
|      | 350         | 375   | 400   |
| 0.5  | 4.412       | 5.707 | 5.752 |
| 1.0  | 5.463       | 7.073 | 6.120 |
| 2.0  | 6.202       | 5.123 | 4.625 |

$$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon_{(ij)k}$$

TOTALS

Temperature ( $T_i$ )

Time ( $H_j$ )

|                           |                            |
|---------------------------|----------------------------|
| T <sub>1..</sub> = 96.46  | T <sub>11..</sub> = 95.22  |
| T <sub>2..</sub> = 107.42 | T <sub>21..</sub> = 111.94 |
| T <sub>3..</sub> = 98.98  | T <sub>31..</sub> = 95.70  |
| 302.86                    | 302.86                     |

Temperature x Time (TH)<sub>ij</sub>

|                           |
|---------------------------|
| T <sub>11..</sub> = 26.47 |
| T <sub>12..</sub> = 32.78 |
| T <sub>13..</sub> = 37.21 |

|                           |
|---------------------------|
| T <sub>21..</sub> = 34.24 |
| T <sub>22..</sub> = 42.44 |
| T <sub>23..</sub> = 30.74 |

|                           |
|---------------------------|
| T <sub>31..</sub> = 34.51 |
| T <sub>32..</sub> = 36.72 |
| T <sub>33..</sub> = 27.75 |
| 302.86                    |

|                           |
|---------------------------|
| T <sub>...</sub> = 302.86 |
| $\Sigma Y_{ijk}$ =        |

TABLE F-3  
ANOVA  
FORCE - DUCTILITY ENERGY (N-M)  
All Data

(0)  $CT = (T \dots)^2 \div n = (302.86)^2 \div (54) = 1698.595919$

(1)  $SS (Temp.) = \frac{1}{(18)} [(96.46)^2 + \dots + (98.98)^2] - CT = 3.661$

(2)  $SS (Time) = \frac{1}{(18)} [(95.22)^2 + \dots + (95.70)^2] - CT = 10.065$

(3)  $SS (H \times T) = \frac{1}{(6)} [(26.47)^2 + \dots + (27.75)^2] - CT - SS (T) - SS (H)$   
 $= \frac{18.946}{74.916}$

(4)  $SS (Total) = \sum \sum x_{ijk}^2 - CT = 74.916$

(5)  $SS (Error) = SS (Total) - SS (T) - SS (H) - SS (T \times H)$   
 $= (4) - (1) - (2) - (3) = 42.244$

| ANALYSIS OF VARIANCE |    |        |       |      |      |
|----------------------|----|--------|-------|------|------|
| Source               | df | SS     | MS    | F    | F.05 |
| T <sub>i</sub>       | 2  | 3.661  | 1.831 | 1.95 | 3.21 |
| H <sub>j</sub>       | 2  | 10.065 | 5.033 | 5.36 | 3.21 |
| (TH) <sub>ij</sub>   | 4  | 18.946 | 4.737 | 5.05 | 2.59 |
| Error                | 45 | 42.244 | 0.939 |      |      |
| Total                | 53 | 74.916 |       |      |      |

TABLE F-4  
FORCE - DUCTILITY ENERGY (N-M)  
ALL DATA  
Newman-Keuls

Cell Means, F-1; Ms(Error), F-3  
 $S \sqrt{Y_{ij}} = \sqrt{4.737/6} = 0.8885$

| $\bar{Y}$ | (k, df) | $\frac{R_k - \bar{Y}}{k}$ | $\bar{Y}$ | (k, df) | $\frac{R_k - \bar{Y}}{k}$ |
|-----------|---------|---------------------------|-----------|---------|---------------------------|
| 9         | 5, 95   | 4.971                     | 5         | 4, 756  | 4.226                     |
| 8         | 5, 432  | 4.827                     | 4         | 4, 415  | 3.923                     |
| 7         | 5, 244  | 4.659                     | 3         | 3, 949  | 3.509                     |
| 6         | 5, 024  | 4.464                     | 2         | 3, 199  | 2.842                     |

| RANK  | 1     | 2     | 3     | 4     | 5     | 6     | 7     | 8     | 9     |
|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| (i,j) | 2,2   | 1,3   | 3,2   | 3,1   | 2,1   | 1,2   | 2,3   | 3,3   | 1,1   |
| Mean  | 7.073 | 6.202 | 6.120 | 5.752 | 5.707 | 5.463 | 5.123 | 4.625 | 4.412 |

| RANK | SIGNIFICANT RANGE |       |       |       |       |       |       |       |       |
|------|-------------------|-------|-------|-------|-------|-------|-------|-------|-------|
|      | k                 | k-1   | k-2   | k-3   | k-4   | k-5   | k-6   | k-7   | k-8   |
| 1    | 497               | 2.661 | 2.448 | 1.950 | 1.610 | 1.366 | 1.321 | 0.953 | 0.871 |
| 2    | 483               | 1.790 | 1.577 | 1.079 | 0.739 | 0.495 | 0.450 | 0.082 | 0     |
| 3    | 466               | 1.708 | 1.495 | 0.997 | 0.657 | 0.413 | 0.368 | 0     |       |
| 4    | 446               | 1.340 | 1.127 | 0.629 | 0.289 | 0.045 | 0     |       |       |
| 5    | 423               | 1.295 | 1.082 | 0.584 | 0.244 | 0     |       |       |       |
| 6    | 392               | 1.051 | 0.838 | 0.340 | 0     |       |       |       |       |
| 7    | 351               | 0.711 | 0.498 | 0     |       |       |       |       |       |
| 8    | 284               | 0.213 | 0     |       |       |       |       |       |       |

No Significant Differences

FORCE - DUCTILITY ENERGY (N-M)  
All Data, Cell Means

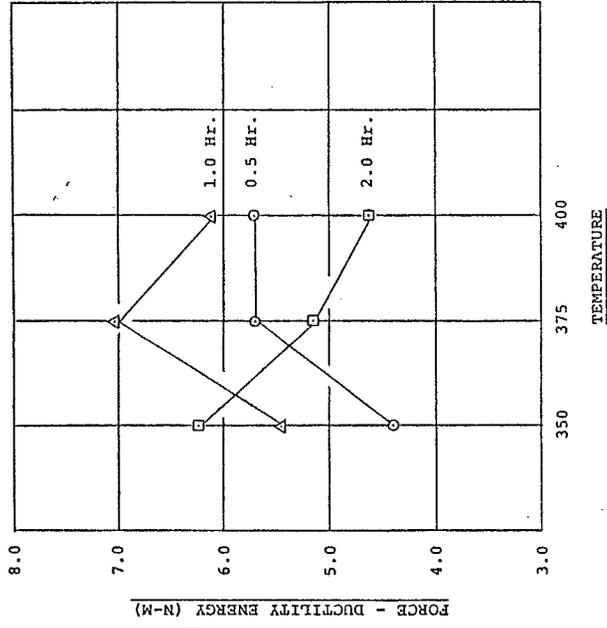


FIGURE F-2  
FORCE - DUCTILITY ENERGY  
VS  
TEMPERATURE

FORCE - DUCTILITY ENERGY (N-M)  
All Data, Cell Means

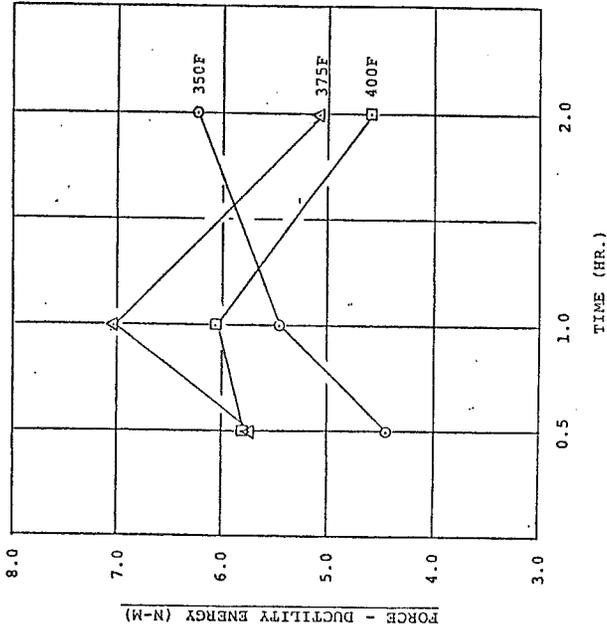


FIGURE F-1  
FORCE - DUCTILITY ENERGY  
VS  
TIME

TABLE F-5  
 FORCE-DUCTILITY ENERGY  
 HOLDING TIME  
 NEWMAN-KEULS

One-way Anova of time ( $q = 0.365$  Iq crit.)

| ANALYSIS OF VARIANCE |    |       |      |      |      |      |
|----------------------|----|-------|------|------|------|------|
| Source               | df | SS    | MS   | F    | F.05 | F.01 |
| Treat.               | 2  | 10.07 | 5.03 | 3.96 | 3.19 | 5.07 |
| Error                | 51 | 64.85 | 1.27 |      |      |      |
| Total                | 53 | 74.92 | -    |      |      |      |

$$S\bar{Y} = \sqrt{\frac{1.27}{18}} = 0.2656$$

| RANK | 1    | 2    | 3    |
|------|------|------|------|
| (i)  | 2    | 1    | 3    |
| Mean | 6.22 | 5.32 | 5.29 |

| Rank | ISR   |      |      |
|------|-------|------|------|
|      | k     | k-1  | 2    |
| 1    | 0.906 | 0.93 | 0.90 |
| 2    | 0.755 | 0.03 | 0    |

No significant differences

FIGURE G-1  
SCHWEYER RHEOMETER

IDENTIFICATION: 22R

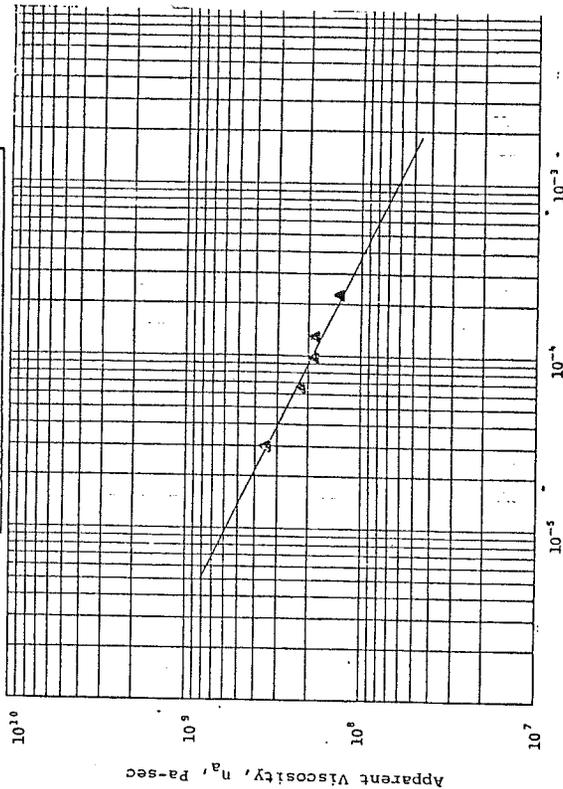
| CHART NO.                        | 5A       | TEMP.    | 4C       | TUBE     | 4G       |
|----------------------------------|----------|----------|----------|----------|----------|
| (L), LVDT Chart Dsp., mm.        | 4.2      | 9.0      | 13.4     | 17.0     | 30.0     |
| (T), Time Chart Dsp., mm         | 150.0    | 150.0    | 150.0    | 150.0    | 150.0    |
| (CS), Chart Speed, cm/min        | 1        | 1        | 1        | 1        | 1        |
| (P), Pressure, KPa               | 15       | 20       | 25       | 30       | 40       |
| $\tau$ Pa                        | 1.1E04   | 1.4E04   | 1.8E04   | 2.2E04   | 2.9E04   |
| $\dot{\gamma}$ mm/sec            | 3.72E-05 | 7.98E-05 | 1.19E-04 | 1.51E-04 | 2.66E-04 |
| $\dot{\gamma}$ sec <sup>-1</sup> | 3.07E-05 | 6.58E-05 | 9.80E-05 | 1.24E-04 | 2.19E-04 |
| $\eta_a$ Pa-sec                  | 3.5E08   | 2.2E08   | 1.8E08   | 1.8E08   | 1.3E08   |

Regression ( $\eta_a = a\dot{\gamma}^b$ ):  $a = 1.961E06$   $b = -4.956E-01$   $R^2 = 0.982$

Rheologic Constants:  $\eta_{a,5}$  8.9E06  $\eta_{a,10}$  2.0E06  $c = 5.1E-01$

Plotting Points:

| $\tau$  | $\dot{\gamma}$ |
|---------|----------------|
| 1.0E-05 | 1.0E-04        |
| 5.9E08  | 1.9E08         |
| 1.9E08  | 6.1E07         |



Shear Rate,  $\dot{\gamma}$ , sec<sup>-1</sup>

Table G-1  
Schweyer Rheometer  
Asphalt Cements

| MATERIAL   | TEMP. | TUBE | a      | b        | c    | R <sup>2</sup> | $\eta_{.05}$ | $\eta_{.10}$ |
|------------|-------|------|--------|----------|------|----------------|--------------|--------------|
| 375 1 Hr.  | 60F   | E    | 6.0E06 | -1.5E02  | 0.98 | 0.94           | 6.3E06       | 6.9E06       |
| 375 1 Hr.  | 60F   | F    | 2.1E08 | 6.2E-01  | 1.60 | 0.26           | 3.4E07       | 2.1E08       |
| 375 1 Hr.  | 30F   | E    | 1.0E08 | -3.4E-01 | 0.66 | 0.31           | 2.8E08       | 1.0E08       |
| 375 1 Hr.  | 30F   | F    | 3.5E08 | -1.4E-01 | 0.86 | 0.21           | 5.4E08       | 3.5E08       |
| 375 1 Hr.  | 30F   | G    | 1.6E07 | -4.9E-01 | 0.51 | 0.91           | 6.9E07       | 1.6E07       |
| Stock      |       |      |        |          |      |                |              |              |
| AR-1000    | 39.2F | D    | 8.5E09 | 1.0E00   | 2.00 | 0.86           | 4.1E08       | 8.5E09       |
| Stock      |       |      |        |          |      |                |              |              |
| AR-1000    | 39.2F | E    | 7.1E06 | -4.0E-01 | 0.60 | 0.02           | 2.4E07       | 7.1E06       |
| Stock      |       |      |        |          |      |                |              |              |
| AR-1000    | 39.2F | F    | 6.7E07 | 8.2E-02  | 1.10 | 0.06           | 5.2E07       | 6.7E07       |
| AR-1000    |       |      |        |          |      |                |              |              |
| Aged @ 375 |       |      |        |          |      |                |              |              |
| AR-1000    | 30F   | D    | 9.9E06 | -5.6E-01 | 0.44 | 0.08           | 5.2E07       | 9.9E06       |
| 1 Hr.      |       |      |        |          |      |                |              |              |
| Aged @ 375 |       |      |        |          |      |                |              |              |
| AR-1000    | 30F   | E    | 1.7E07 | -5.1E-01 | 0.49 | 0.34           | 7.9E07       | 1.7E07       |
| 1 Hr.      |       |      |        |          |      |                |              |              |
| Aged @ 375 |       |      |        |          |      |                |              |              |
| AR-1000    | 30F   | F    | 6.5E08 | -1.3E-01 | 0.87 | 0.90           | 9.5E08       | 6.5E08       |
| 1 Hr.      |       |      |        |          |      |                |              |              |
| AR-1000    |       |      |        |          |      |                |              |              |
| Aged @ 375 |       |      |        |          |      |                |              |              |
| AR-1000    | 60F   | C    | 1.7E07 | 4.5E-01  | 1.50 | 0.29           | 4.3E06       | 1.7E07       |
| 1 Hr.      |       |      |        |          |      |                |              |              |
| Aged @ 375 |       |      |        |          |      |                |              |              |
| AR-1000    | 60F   | D    | 3.1E06 | 4.0E-02  | 1.00 | 0.17           | 2.8E06       | 3.1E06       |
| 1 Hr.      |       |      |        |          |      |                |              |              |
| Aged @ 375 |       |      |        |          |      |                |              |              |
| AR-1000    | 60F   | E    | 7.9E05 | -2.3E-01 | 0.77 | 0.52           | 1.6E06       | 7.9E05       |
| 1 Hr.      |       |      |        |          |      |                |              |              |
| Aged @ 375 |       |      |        |          |      |                |              |              |
| AR-1000    | 39.2F | D    | 3.6E08 | 1.4E-01  | 1.10 | 0.22           | 2.3E08       | 3.6E08       |
| 1 Hr.      |       |      |        |          |      |                |              |              |
| Aged @ 375 |       |      |        |          |      |                |              |              |
| AR-1000    | 39.2F | E    | 1.7E09 | 3.8E-01  | 1.40 | 0.75           | 5.4E08       | 1.7E09       |
| 1 Hr.      |       |      |        |          |      |                |              |              |
| Aged @ 375 |       |      |        |          |      |                |              |              |
| AR-1000    | 39.2F | F    | 6.5E08 | 1.7E-01  | 1.20 | 0.77           | 3.9E08       | 6.5E08       |
| 1 Hr.      |       |      |        |          |      |                |              |              |
| Stock      |       |      |        |          |      |                |              |              |
| AR-1000    | 60F   | C    | 1.0E06 | 1.3E-01  | 1.10 | 0.81           | 6.9E05       | 1.0E06       |
| Stock      |       |      |        |          |      |                |              |              |
| AR-1000    | 60F   | D    | 5.7E05 | -3.5E-02 | 0.97 | 0.14           | 6.3E05       | 5.7E05       |
| Stock      |       |      |        |          |      |                |              |              |
| AR-1000    | 60F   | B    | 2.8E06 | 7.7E-01  | 1.8  | 0.72           | 2.8E05       | 2.8E06       |
| Stock      |       |      |        |          |      |                |              |              |
| AR-1000    | 30F   | D    | 9.1E09 | 6.7E-01  | 1.7  | 0.82           | 1.2E09       | 9.1E09       |
| Stock      |       |      |        |          |      |                |              |              |
| AR-1000    | 30F   | E    | 5.7E10 | 7.4E-01  | 1.7  | 0.62           | 6.3E09       | 5.7E10       |
| Stock      |       |      |        |          |      |                |              |              |
| AR-1000    | 30F   | F    | 4.0E11 | 9.4E-01  | 1.9  | 0.94           | 2.4E10       | 4.0E11       |

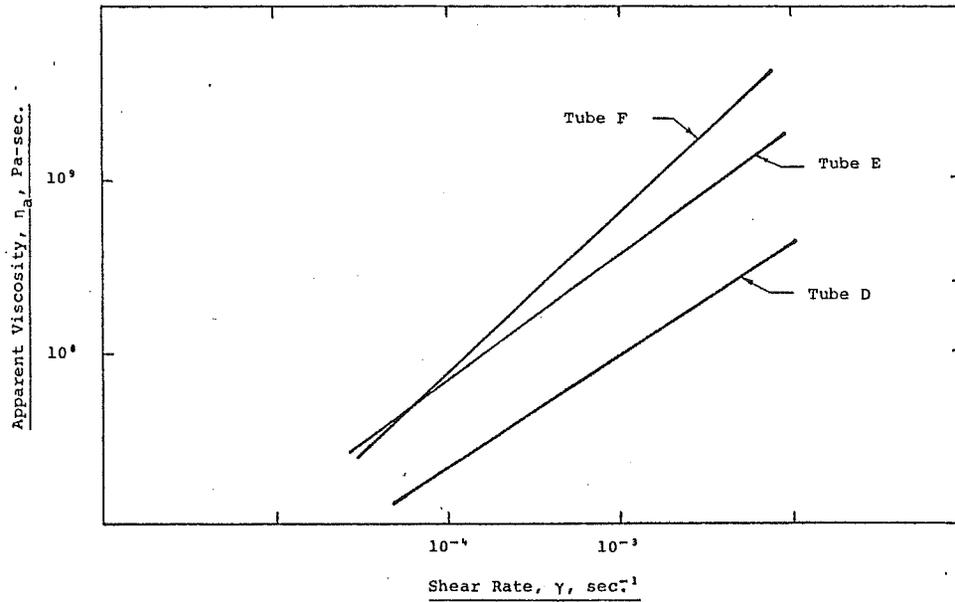


Figure G-2 - AR-1000 Unaged  
Test Temperature, 30F

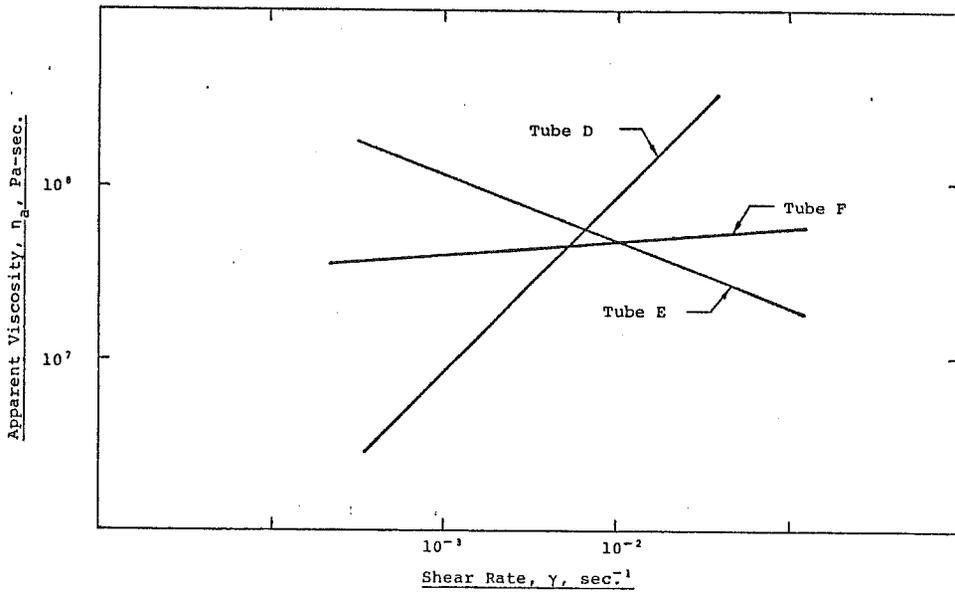


Figure G-1 - AR-1000 Unaged  
Test Temperature, 39.2F

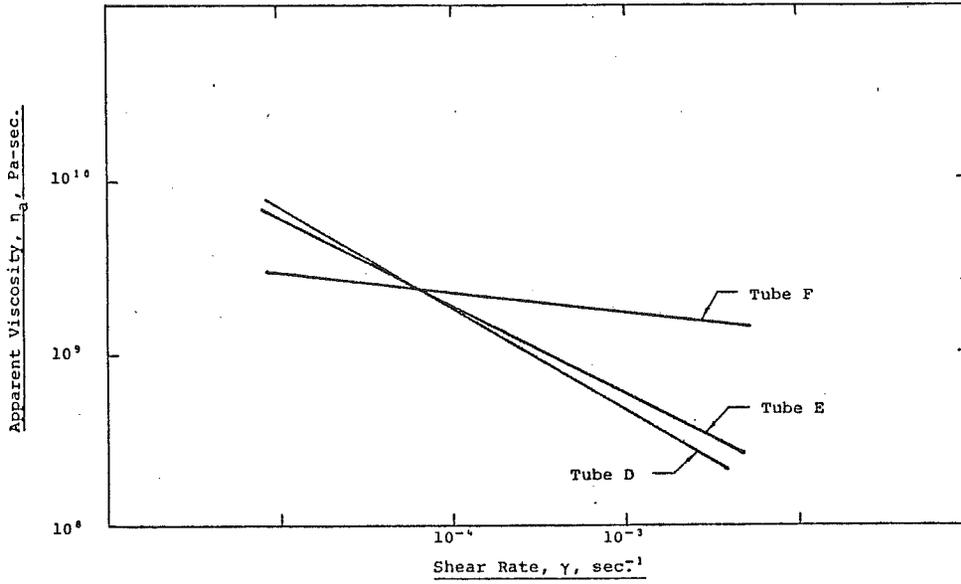


Figure G-4 - AR-1000 Aged 1 Hr. @ 375F  
Test Temperature, 30F

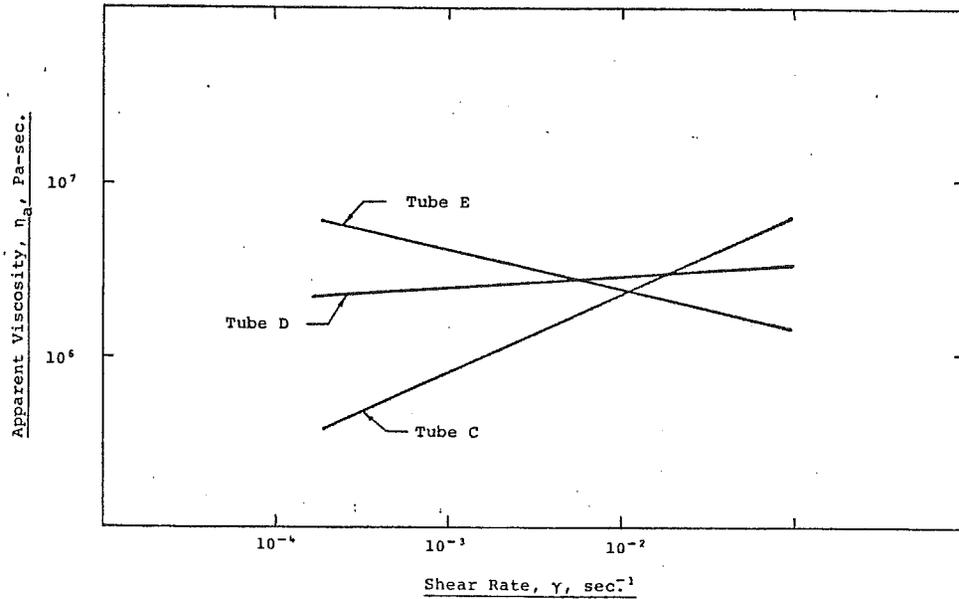


Figure G-3 - AR-1000 Aged 1 Hr. @ 375F  
Test Temperature, 60F

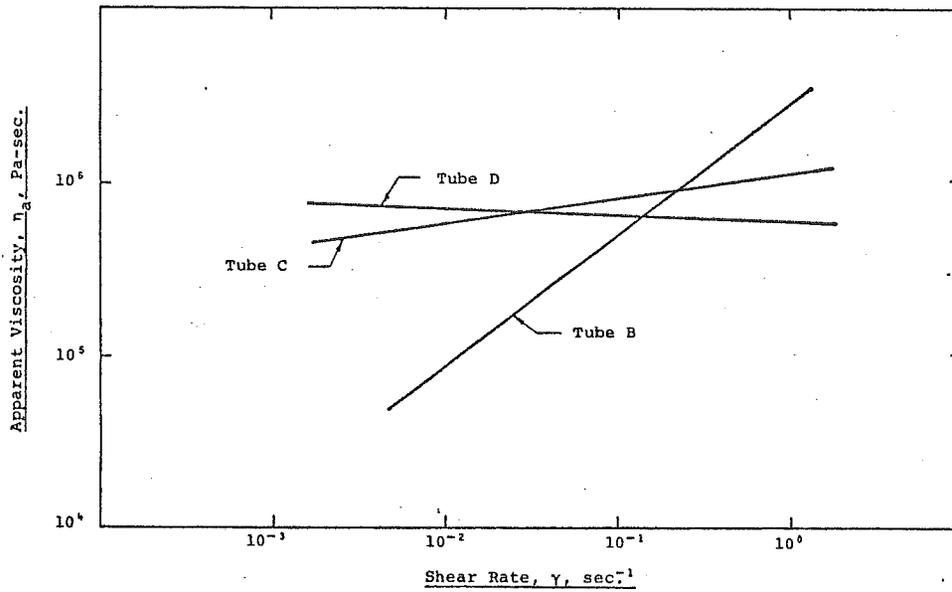


Figure G-6 - AR-1000 - Unaged  
Test Temperature, 60F

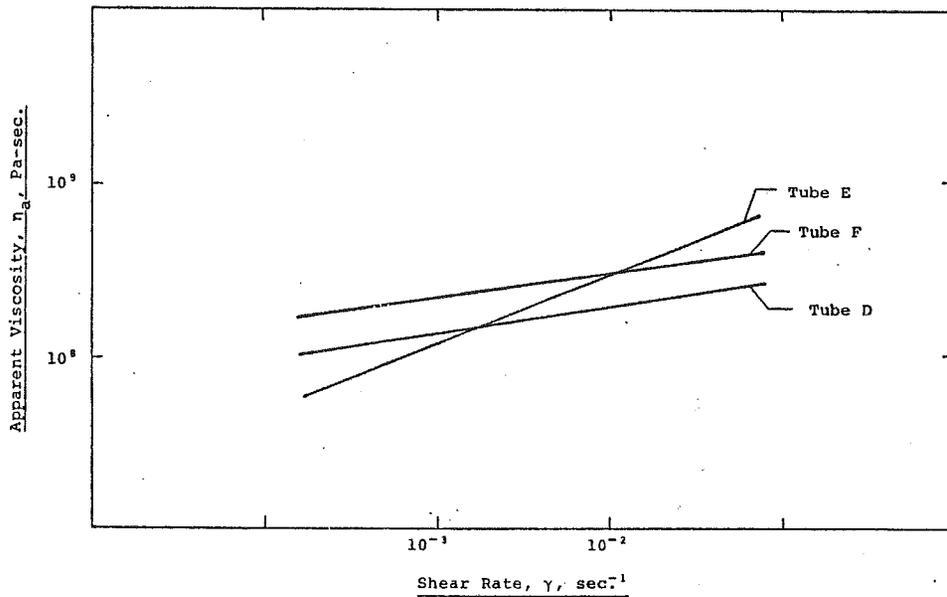


Figure G-5 - AR-1000 Aged 1 Hr. @ 375F  
Test Temperature, 39.2F

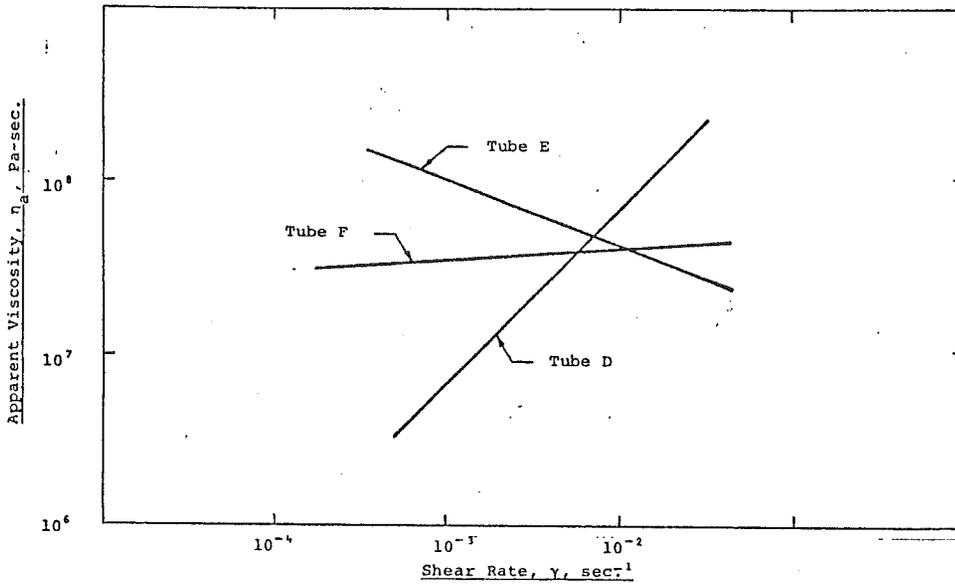


Figure G-8 - AR-1000 Unaged  
Test Temperature = 39.2F

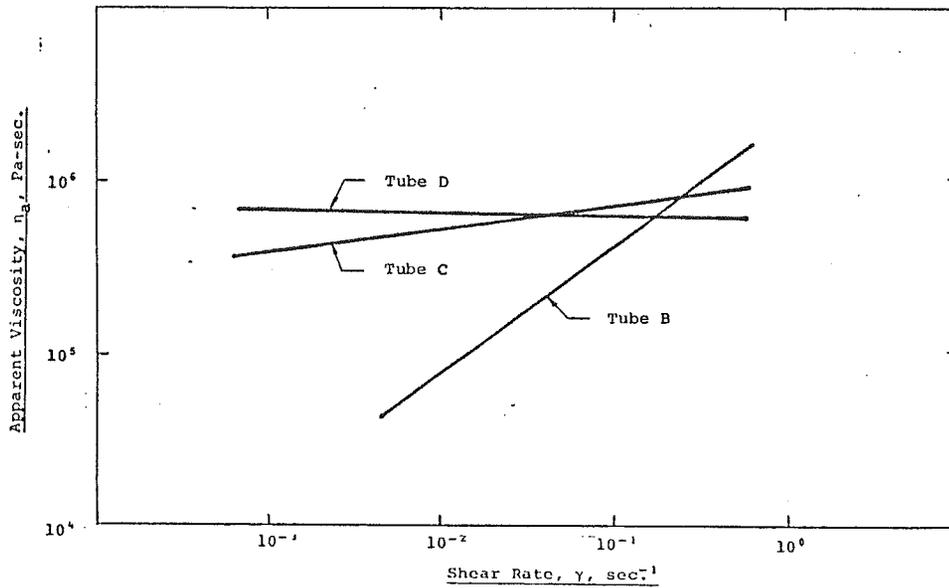


Figure G-7 - AR-1000 - Unaged  
Test Temperature = 60F

Apparent Viscosity,  $\eta_a$ , Pa-sec.

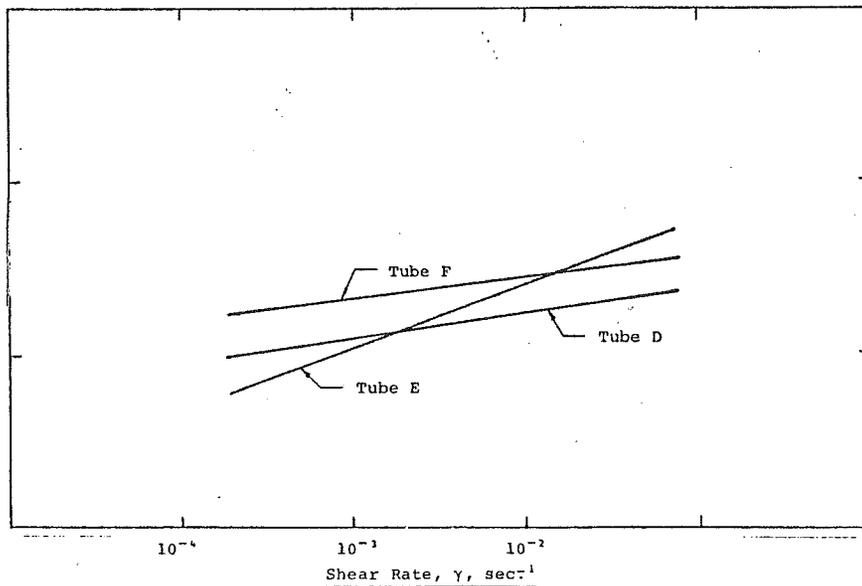


Figure G-9 - AR-1000 Aged at 375F for 1 Hour  
Test Temperature = 39.2F

TABLE G-2  
ANOVA  
Schweyer  
No. 05

|        | Holding Time ( $H_c$ ) |            |             | TOTALS      |
|--------|------------------------|------------|-------------|-------------|
|        | 350                    | 375        | 400         |             |
| 0.5    | E                      | 3.869 E+07 | 0.3134 E+07 | 4.714 E+07  |
|        |                        | 10.86 E+07 | 13.75 E+07  | 17.81 E+07  |
|        | F                      | 3.982 E+07 | 39.95 E+07  | 91.40 E+07  |
| 1.0    |                        | 6.072 E+07 | 94.05 E+07  | 9.312 E+07  |
|        | G                      | 1.239 E+07 | 18.25 E+07  | 10.06 E+07  |
|        |                        | 3.335 E+07 | 0.8452 E+07 | 1.254 E+07  |
| 1.5    | E                      | 24.15 E+07 | 57.47 E+07  | 18.58 E+07  |
|        |                        | 86.01 E+07 | 17.11 E+07  | 43.93 E+07  |
|        | F                      | 15.49 E+07 | 124.0 E+07  | 56.70 E+07  |
| 2.0    |                        | 79.17 E+07 | 6.474 E+07  | 66.76 E+07  |
|        | G                      | 4.219 E+07 | 5.389 E+07  | 11.26 E+07  |
|        |                        | 1.145 E+07 | 4.875 E+07  | 14.67 E+07  |
| 2.5    | E                      | 15.78 E+07 | 10.45 E+07  | 135.5 E+07  |
|        |                        | 28.03 E+07 | 6.674 E+07  | 7.126 E+07  |
|        | F                      | 230.6 E+07 | 13.68 E+07  | 15.84 E+07  |
| 3.0    |                        | 6.439 E+07 | 4.288 E+07  | 18.42 E+07  |
|        | G                      | 2.748 E+07 | 2.513 E+07  | 4.000 E+07  |
|        |                        | 1.092 E+07 | 0.8348 E+07 | 0.8877 E+07 |
| TOTALS |                        |            |             |             |

TABLE G-3

ANOVA  
Schweyer  
no. 05

TABLE G-3 (Cont.)

TOTALS - Continued

FOSTER & BURR q:

Matrix  $\Sigma_i$  \_\_\_\_\_ Matrix  $\Sigma_k^2$  \_\_\_\_\_  
 $v$  \_\_\_\_\_  $q$  0.287 \_\_\_\_\_  $q$ .99 0.198 \_\_\_\_\_  $q$ .999 0.265 \_\_\_\_\_

$Y_{ijk1} = \mu + T_i + C_j + H_k + (TC)_{ij} + (TH)_{ik} + (CH)_{jk} + (TCH)_{ijk} + \epsilon(ijk)1$

TOTALS

| Temperature ( $T_i$ ) | Tube ( $C_j$ )     | Time ( $H_j$ )    |
|-----------------------|--------------------|-------------------|
| $T_{1..}$ = _____     | $T_{.1..}$ = _____ | $T_{..1}$ = _____ |
| $T_{2..}$ = _____     | $T_{.2..}$ = _____ | $T_{..2}$ = _____ |
| $T_{3..}$ = _____     | $T_{.3..}$ = _____ | $T_{..3}$ = _____ |

Temperature x Tube (TC)<sub>ij</sub>

|                    |                    |                    |
|--------------------|--------------------|--------------------|
| $T_{11..}$ = _____ | $T_{21..}$ = _____ | $T_{31..}$ = _____ |
| $T_{12..}$ = _____ | $T_{22..}$ = _____ | $T_{32..}$ = _____ |
| $T_{13..}$ = _____ | $T_{23..}$ = _____ | $T_{33..}$ = _____ |

Temperature x Time (TH)<sub>jk</sub>

|                    |                    |                    |
|--------------------|--------------------|--------------------|
| $T_{1.1.}$ = _____ | $T_{2.1.}$ = _____ | $T_{3.1.}$ = _____ |
| $T_{1.2.}$ = _____ | $T_{2.2.}$ = _____ | $T_{3.2.}$ = _____ |
| $T_{1.3.}$ = _____ | $T_{2.3.}$ = _____ | $T_{3.3.}$ = _____ |

Tube x Time (CH)<sub>jk</sub>

|                    |                    |                    |
|--------------------|--------------------|--------------------|
| $T_{.11.}$ = _____ | $T_{.12.}$ = _____ | $T_{.13.}$ = _____ |
| $T_{.21.}$ = _____ | $T_{.22.}$ = _____ | $T_{.23.}$ = _____ |
| $T_{.31.}$ = _____ | $T_{.32.}$ = _____ | $T_{.33.}$ = _____ |

Temperature x Tube x Time (TCH)<sub>ijk</sub>

|                          |                          |                          |
|--------------------------|--------------------------|--------------------------|
| $T_{111.}$ = 147.29 E+06 | $T_{112.}$ = 1101.6 E+06 | $T_{113.}$ = 438.10 E+06 |
| $T_{121.}$ = 100.54 E+06 | $T_{122.}$ = 946.50 E+06 | $T_{123.}$ = 2370.4 E+06 |
| $T_{131.}$ = 45.740 E+06 | $T_{132.}$ = 53.540 E+06 | $T_{133.}$ = 38.40 E+06  |
| $T_{211.}$ = 140.63 E+06 | $T_{212.}$ = 745.80 E+06 | $T_{213.}$ = 171.24 E+06 |
| $T_{221.}$ = 1340.0 E+06 | $T_{222.}$ = 1304.7 E+06 | $T_{223.}$ = 179.68 E+06 |
| $T_{231.}$ = 190.95 E+06 | $T_{232.}$ = 102.54 E+06 | $T_{233.}$ = 33.478 E+06 |
| $T_{311.}$ = 225.24 E+06 | $T_{312.}$ = 625.10 E+06 | $T_{313.}$ = 1426.3 E+06 |
| $T_{321.}$ = 1007.1 E+06 | $T_{322.}$ = 1234.6 E+06 | $T_{323.}$ = 342.60 E+06 |
| $T_{331.}$ = 113.14 E+06 | $T_{332.}$ = 259.30 E+06 | $T_{333.}$ = 48.877 E+06 |

TABLE G-4

ANOVA  
Schweyer  
Log<sub>10</sub> (no. 05 ÷ 1.00 E+07)

|        | Holding Time (H <sub>ε</sub> ) |         | 400     |
|--------|--------------------------------|---------|---------|
|        | 350                            | 375     |         |
| E      | 0.5876                         | -0.5039 | 0.6734  |
|        | 1.0358                         | 1.1383  | 1.2507  |
|        | 0.6001                         | 1.6015  | 1.9609  |
| F      | 0.7833                         | 1.9734  | 0.9690  |
|        | 0.0931                         | 1.2613  | 1.0026  |
|        | 0.5231                         | -0.0730 | 0.0983  |
| G      | 1.3829                         | 1.7594  | 1.2690  |
|        | 1.9345                         | 1.2333  | 1.6428  |
|        | 1.9101                         | 2.0934  | 1.7536  |
| 1.0    | 1.8986                         | 0.8112  | 1.8245  |
|        | 0.6252                         | 0.7315  | 1.0515  |
|        | 0.0588                         | 0.6880  | 1.1664  |
| E      | 1.1981                         | 1.0191  | 2.1319  |
|        | 1.4476                         | 0.8244  | 0.8528  |
|        | 2.3629                         | 1.1361  | 1.1988  |
| 2.0    | 0.8088                         | 0.6323  | 1.2653  |
|        | 0.4390                         | 0.4002  | 0.6021  |
|        | 0.0382                         | -0.0784 | -0.0517 |
| TOTALS |                                |         |         |

TABLE G-5

ANOVA  
Schweyer  
Log<sub>10</sub> (no. 05 ÷ 1.00 E+07)

FOSTER & BURR q:  
Matrix Lx<sub>i</sub> 5.503.770 000 E-01      Matrix Lx<sub>i</sub> 7.945.572 551 E+01  
v 1.000      q 0.106      q<sub>199</sub> 0.198      q<sub>1999</sub> 0.265

| Y <sub>ijk1</sub> = μ + T <sub>i</sub> + C <sub>j</sub> + H <sub>k</sub> + (TC) <sub>ij</sub> + (TH) <sub>ik</sub> + (CH) <sub>jk</sub> + (TCH) <sub>ijk</sub> + ε <sub>(ijk)1</sub> |                             |                             |
|--|-----------------------------|-----------------------------|
| TOTALS   |                             |                             |
| Temperature (T <sub>i</sub> )  | Tube (C <sub>j</sub> )      |                             |
| T <sub>1...</sub> = 17.7277  | T <sub>1...</sub> = 20.8777 | Time (H <sub>j</sub> )      |
| T <sub>2...</sub> = 16.6481  | T <sub>2...</sub> = 25.5838 | T <sub>11..</sub> = 14.9755 |
| T <sub>3...</sub> = 20.6619  | T <sub>3...</sub> = 8.5762  | T <sub>21..</sub> = 23.8347 |
| T <sub>4...</sub> = 55.0377  | T <sub>4...</sub> = 55.0377 | T <sub>31..</sub> = 16.2275 |
| Temperature x Tube (TC) <sub>ij</sub>  |                             |                             |
| T <sub>11..</sub> = 7.5865   | T <sub>21..</sub> = 5.4706  | T <sub>11..</sub> = 7.8206  |
| T <sub>12..</sub> = 8.3638   | T <sub>22..</sub> = 8.2479  | T <sub>21..</sub> = 8.9721  |
| T <sub>13..</sub> = 1.7774   | T <sub>23..</sub> = 2.9296  | T <sub>31..</sub> = 3.8692  |
| T <sub>14..</sub> = 17.7277  | T <sub>24..</sub> = 16.6481 | T <sub>32..</sub> = 20.6619 |
| Temperature x Time (TH) <sub>ik</sub>  |                             |                             |
| T <sub>11..</sub> = 3.6230   | T <sub>21..</sub> = 5.3976  | T <sub>11..</sub> = 5.9549  |
| T <sub>12..</sub> = 7.8101   | T <sub>22..</sub> = 7.3168  | T <sub>21..</sub> = 8.7078  |
| T <sub>13..</sub> = 6.2946   | T <sub>23..</sub> = 3.9337  | T <sub>31..</sub> = 5.9992  |
| T <sub>14..</sub> = 17.7277  | T <sub>24..</sub> = 16.6481 | T <sub>32..</sub> = 20.6619 |
| Tube x Time (CH) <sub>jk</sub>   |                             |                             |
| T <sub>11..</sub> = 4.1819   | T <sub>12..</sub> = 9.2219  | T <sub>11..</sub> = 7.4739  |
| T <sub>21..</sub> = 7.8882   | T <sub>22..</sub> = 10.2914 | T <sub>21..</sub> = 7.4042  |
| T <sub>31..</sub> = 2.9054   | T <sub>32..</sub> = 4.3214  | T <sub>31..</sub> = 1.3494  |
| T <sub>41..</sub> = 14.9755  | T <sub>42..</sub> = 23.8347 | T <sub>41..</sub> = 16.2275 |

TABLE G-5 (Cont.)

TOTALS - Continued

Temperature x Tube x Time (TCH)<sub>ijk</sub>

|                  |   |        |
|------------------|---|--------|
| T <sub>111</sub> | = | 1.6234 |
| T <sub>121</sub> | = | 1.3834 |
| T <sub>131</sub> | = | 0.6162 |
| T <sub>211</sub> | = | 3.6230 |
| T <sub>221</sub> | = | 0.6344 |
| T <sub>231</sub> | = | 3.5749 |
| T <sub>311</sub> | = | 1.1883 |
| T <sub>321</sub> | = | 5.3976 |
| T <sub>112</sub> | = | 3.3174 |
| T <sub>122</sub> | = | 3.8087 |
| T <sub>132</sub> | = | 0.6840 |
| T <sub>212</sub> | = | 7.8101 |
| T <sub>222</sub> | = | 2.9927 |
| T <sub>232</sub> | = | 2.9046 |
| T <sub>312</sub> | = | 1.4195 |
| T <sub>322</sub> | = | 7.3168 |
| T <sub>113</sub> | = | 2.9118 |
| T <sub>123</sub> | = | 2.9299 |
| T <sub>133</sub> | = | 3.5781 |
| T <sub>213</sub> | = | 1.1009 |
| T <sub>223</sub> | = | 5.9549 |
| T <sub>233</sub> | = | 8.7078 |

|                  |   |        |
|------------------|---|--------|
| T <sub>113</sub> | = | 2.6457 |
| T <sub>123</sub> | = | 3.1717 |
| T <sub>133</sub> | = | 0.4772 |
| T <sub>213</sub> | = | 6.2946 |
| T <sub>223</sub> | = | 1.8435 |
| T <sub>233</sub> | = | 1.7684 |
| T <sub>313</sub> | = | 0.3218 |
| T <sub>323</sub> | = | 3.9337 |
| T <sub>333</sub> | = | 2.9847 |
| T <sub>333</sub> | = | 2.4641 |
| T <sub>333</sub> | = | 0.5504 |
| T <sub>333</sub> | = | 5.9992 |

TABLE G-6

ANOVA  
Schweyer  
Log<sub>10</sub> (no. 05 ÷ 1.00 E+07)

|                            |   |   |   |                               |   |                    |
|----------------------------|---|---|---|-------------------------------|---|--------------------|
| (0) CT                     | = | (T <sub>111</sub> ...) <sup>2</sup> ÷ n   | = | ( 55.0377 ) <sup>2</sup> ÷ 54 | = | 5.609 534 113 E+01 |
| (1) SS(T <sub>1</sub> )    | = | $\frac{1}{18} [( 17.7277 )^2 + \dots + ( 20.6619 )^2] - (0) - CT$   |   |                               |   |                    |
|                            |   |   |   | 4.793 640 400 E-01            |   |                    |
| (2) SS(C <sub>j</sub> )    | = | $\frac{1}{18} [( 20.8777 )^2 + \dots + ( 8.5762 )^2] - (0) - CT$  |   |                               |   |                    |
|                            |   |   |   | 8.569 124 760 E+00            |   |                    |
| (3) SS(H <sub>k</sub> )    | = | $\frac{1}{18} [( 14.9755 )^2 + \dots + ( .16.2275 )^2] - (0) - CT$  |   |                               |   |                    |
|                            |   |   |   | 2.554 118 930 E+00            |   |                    |
| (4) SS(TC) <sub>ij</sub>   | = | $\frac{1}{6} [( 7.5865 )^2 + \dots + ( 3.8692 )^2] - (0) - SS(T1) - SS(Cj)$   |   |                               |   |                    |
|                            |   |   |   | 4.955 417 200 E-01            |   |                    |
| (5) SS(TH) <sub>ik</sub>   | = | $\frac{1}{6} [( 3.6230 )^2 + \dots + ( 5.9992 )^2] - (0) - SS(T1) - SS(Hk)$   |   |                               |   |                    |
|                            |   |   |   | 7.322 491 200 E-01            |   |                    |
| (6) SS(CH) <sub>jk</sub>   | = | $\frac{1}{6} [( 4.1819 )^2 + \dots + ( 1.3494 )^2] - (0) - SS(T1) - SS(Cj) - SS(Hk)$  |   |                               |   |                    |
|                            |   |   |   | 1.162 486 310 E+00            |   |                    |
| (7) SS(TCH) <sub>ijk</sub> | = | $\frac{1}{2} [( 1.6234 )^2 + \dots + ( 0.5504 )^2] - (0) - CT - SS(T1) - SS(Cj) - SS(Hk) - SS(TH)ik - SS(CH)jk$   |   |                               |   |                    |
|                            |   |   |   | 1.816 263 090 E+00            |   |                    |
| (8) SS(Total)              | = | $\sum \sum \sum x_{ijk}^2 - (0) - CT$   |   |                               |   | 2.336 038 438 E+01 |
| (9) SS(Error)              | = | SS(Total) - SS(T <sub>1</sub> ) - SS(C <sub>j</sub> ) - SS(H <sub>k</sub> ) - SS(TC) <sub>ij</sub> - SS(TH) <sub>ik</sub> - SS(CH) <sub>jk</sub> - SS(TCH) <sub>ijk</sub> |   |                               |   |                    |
|                            |   |   |   |                               |   | 7.551 236 410 E+00 |

TABLE G-7

ANOVA  
Schweyer  
Log<sub>10</sub> (no. 05 ÷ 1.00 E+07)

| SOURCE               | df | SS      | MS     | F     | F.05 | F.01 |
|----------------------|----|---------|--------|-------|------|------|
| T <sub>i</sub>       | 2  | 0.4794  | 0.2397 | 0.86  | 3.35 | 5.49 |
| C <sub>j</sub>       | 2  | 8.5691  | 4.2846 | 15.32 | 3.35 | 5.49 |
| H <sub>k</sub>       | 2  | 2.5541  | 1.2771 | 4.57  | 3.35 | 5.49 |
| (TC) <sub>ij</sub>   | 4  | 0.4955  | 0.1239 | 0.44  | 2.73 | 4.11 |
| (TH) <sub>ik</sub>   | 4  | 0.7322  | 0.1831 | 0.65  | 2.73 | 4.11 |
| (CH) <sub>jk</sub>   | 4  | 1.1625  | 0.2906 | 1.04  | 2.73 | 4.11 |
| (TCH) <sub>ijk</sub> | 8  | 1.8163  | 0.2270 | 0.81  | 2.31 | 3.26 |
| Error                | 27 | 7.5512  | 0.2797 |       |      |      |
| Total                | 53 | 23.3604 |        |       |      |      |

TABLE G-8

ANOVA  
Schweyer  
Shear Susceptibility Index, C

|        | Holding Time (H <sub>e</sub> ) |       |       | TOTALS |
|--------|--------------------------------|-------|-------|--------|
|        | 350                            | 375   | 400   |        |
| 0.5    | E                              | 1.003 | 0.468 | 0.516  |
|        | F                              | 0.911 | 0.890 | 1.087  |
|        | G                              | 0.668 | 0.996 | 1.042  |
| 1.0    | E                              | 0.715 | 0.723 | 0.801  |
|        | F                              | 0.450 | 0.393 | 0.674  |
|        | G                              | 0.618 | 0.391 | 0.410  |
| 2.0    | E                              | 0.846 | 1.014 | 0.730  |
|        | F                              | 1.071 | 0.890 | 0.938  |
|        | G                              | 0.755 | 1.021 | 0.896  |
| TOTALS | E                              | 0.969 | 0.682 | 1.030  |
|        | F                              | 0.699 | 0.450 | 0.797  |
|        | G                              | 0.299 | 0.500 | 0.722  |
| TOTALS | E                              | 1.108 | 0.665 | 1.485  |
|        | F                              | 1.105 | 0.987 | 0.974  |
|        | G                              | 1.036 | 0.699 | 0.834  |
| TOTALS | E                              | 0.739 | 0.759 | 1.013  |
|        | F                              | 0.567 | 0.456 | 0.570  |
|        | G                              | 0.374 | 0.506 | 0.508  |

TABLE G-9

ANOVA  
Schweyer  
Shear Susceptibility Index, C

FOSTER & BURR q:

Matrix  $Ix_i$  41.419 Matrix  $Ix_j$  3.506 341.700  $E+01$   
 $v$  1.00  $q$  0.092  $q_{.99}$  0.198  $q_{.999}$  0.265

$$Y_{ijk1} = \mu + T_i + C_j + H_k + (TC)_{ij} + (TH)_{ik} + (CH)_{jk} + (TCH)_{ijk} + \epsilon_{(ijk)1}$$

TOTALS

| Temperature ( $T_i$ ) | Tube ( $C_j$ )      | Time ( $H_j$ )      |
|-----------------------|---------------------|---------------------|
| $T_{1..}$ = 13.933    | $T_{.1..}$ = 16.657 | $T_{...1}$ = 12.756 |
| $T_{2..}$ = 12.409    | $T_{.2..}$ = 15.378 | $T_{...2}$ = 14.278 |
| $T_{3..}$ = 15.077    | $T_{.3..}$ = 9.384  | $T_{...3}$ = 14.385 |
| 41.419                | 41.419              | 41.419              |

Temperature x Tube (TC)<sub>ij</sub>

|                    |                    |                    |
|--------------------|--------------------|--------------------|
| $T_{11..}$ = 6.044 | $T_{21..}$ = 4.833 | $T_{31..}$ = 5.780 |
| $T_{12..}$ = 4.882 | $T_{22..}$ = 4.880 | $T_{32..}$ = 5.616 |
| $T_{13..}$ = 3.007 | $T_{23..}$ = 2.696 | $T_{33..}$ = 3.681 |
| 13.933             | 12.409             | 15.077             |

Temperature x Time (TH)<sub>ik</sub>

|                    |                    |                    |
|--------------------|--------------------|--------------------|
| $T_{1.1.}$ = 4.365 | $T_{2.1.}$ = 3.961 | $T_{3.1.}$ = 4.530 |
| $T_{1.2.}$ = 4.639 | $T_{2.2.}$ = 4.476 | $T_{3.2.}$ = 5.163 |
| $T_{1.3.}$ = 4.929 | $T_{2.3.}$ = 4.072 | $T_{3.3.}$ = 5.384 |
| 13.933             | 12.409             | 15.077             |

Tube x Time (CH)<sub>jk</sub>

|                    |                    |                    |
|--------------------|--------------------|--------------------|
| $T_{.11.}$ = 4.875 | $T_{.12.}$ = 5.458 | $T_{.13.}$ = 6.324 |
| $T_{.21.}$ = 4.945 | $T_{.22.}$ = 5.353 | $T_{.23.}$ = 5.080 |
| $T_{.31.}$ = 2.936 | $T_{.32.}$ = 3.467 | $T_{.33.}$ = 2.981 |
| 12.756             | 14.278             | 14.385             |

TABLE G-9 (Cont.)

TOTALS - Continued

Temperature x Tube x Time (TCH)<sub>ijk</sub>

|                    |                    |                    |
|--------------------|--------------------|--------------------|
| $T_{111.}$ = 1.914 | $T_{112.}$ = 1.917 | $T_{113.}$ = 2.213 |
| $T_{121.}$ = 1.383 | $T_{122.}$ = 1.724 | $T_{123.}$ = 1.775 |
| $T_{131.}$ = 1.068 | $T_{132.}$ = 0.998 | $T_{133.}$ = 0.941 |
| 4.365              | 4.639              | 4.929              |
| $T_{211.}$ = 1.358 | $T_{212.}$ = 1.823 | $T_{213.}$ = 1.652 |
| $T_{221.}$ = 1.719 | $T_{222.}$ = 1.703 | $T_{223.}$ = 1.458 |
| $T_{231.}$ = 0.784 | $T_{232.}$ = 0.950 | $T_{233.}$ = 0.952 |
| 3.861              | 4.476              | 4.072              |
| $T_{311.}$ = 1.603 | $T_{312.}$ = 1.718 | $T_{313.}$ = 2.459 |
| $T_{321.}$ = 1.843 | $T_{322.}$ = 1.926 | $T_{323.}$ = 1.847 |
| $T_{331.}$ = 1.084 | $T_{332.}$ = 1.519 | $T_{333.}$ = 1.078 |
| 4.530              | 5.163              | 5.384              |

TABLE G-10

ANOVA  
Schweyer  
Shear Susceptibility Index, C

$$(0) CT = (T \dots)^2 \div n = (41.419)^2 \div 54 = 3.176\ 914\ 002\ E+01$$

$$(1) SS(T_i) = \frac{1}{18} [(13.933)^2 + \dots + (15.077)^2] - (0) = 1.990\ 654\ 800\ E-01$$

$$(2) SS(C_j) = \frac{1}{18} [(16.657)^2 + \dots + (9.384)^2] - (0) = 1.657\ 192\ 700\ E+00$$

$$(3) SS(H_k) = \frac{1}{18} [(12.756)^2 + \dots + (14.385)^2] - (0) = 9.225\ 137\ 000\ E-02$$

$$(4) SS(TC)_{ij} = \frac{1}{6} [(6.044)^2 + \dots + (3.681)^2] - (0) - SS(T_i) - SS(C_j) = 8.064\ 030\ 000\ E-02$$

$$(5) SS(TH)_{ik} = \frac{1}{6} [(4.365)^2 + \dots + (5.384)^2] - (0) - SS(T_i) - SS(C_j) = 3.230\ 863\ 000\ E-02$$

$$(6) SS(CH)_{jk} = \frac{1}{6} [(4.875)^2 + \dots + (2.981)^2] - (0) - SS(C_j) - SS(H_k) = 1.282\ 400\ 800\ E-01$$

$$(7) SS(TCH)_{ijk} = \frac{1}{2} [(1.914)^2 + \dots + (1.078)^2] - (0) - SS(T_i) - SS(C_j) - SS(H_k) - SS(TC)_{ij} - SS(TH)_{ik} - SS(CH)_{jk} = 1.947\ 779\ 200\ E-01$$

$$(8) SS(\text{Total}) = \sum \sum \sum x_{ijk}^2 - (0) = 3.294\ 276\ 980\ E+00$$

$$(9) SS(\text{Error}) = SS(\text{Total}) - SS(T_i) - SS(C_j) - SS(H_k) - SS(TC)_{ij} - SS(TH)_{ik} - SS(CH)_{jk} - SS(TCH)_{ijk} = (8) - (1) - (2) - (3) - (4) - (5) - (6) - (7) = 8.918\ 005\ 000\ E-01$$

TABLE G-11

ANOVA  
Schweyer  
Shear Susceptibility Index, C

| SOURCE                   | df | SS     | MS     | F     | F.05 | F.01 |
|--------------------------|----|--------|--------|-------|------|------|
| (1) T <sub>i</sub>       | 2  | 0.1991 | 0.0995 | 3.01  | 3.35 | 5.49 |
| (2) C <sub>j</sub>       | 2  | 1.6752 | 0.8376 | 25.36 | 3.35 | 5.49 |
| (3) H <sub>k</sub>       | 2  | 0.0223 | 0.0461 | 1.40  | 3.35 | 5.49 |
| (4) (TC) <sub>ij</sub>   | 4  | 0.0806 | 0.0202 | 0.61  | 2.73 | 4.11 |
| (5) (TH) <sub>ik</sub>   | 4  | 0.0323 | 0.0081 | 0.24  | 2.73 | 4.11 |
| (6) (CH) <sub>jk</sub>   | 4  | 0.1282 | 0.0321 | 0.97  | 2.73 | 4.11 |
| (7) (TCH) <sub>ijk</sub> | 8  | 0.1948 | 0.0243 | 99.74 | 2.31 | 3.26 |
| (9) Error                | 27 | 0.8218 | 0.0330 | -     | -    | -    |
| (8) Total                | 53 | 3.2943 | -      | -     | -    | -    |

TABLE G-13  
ANOVA  
Schweyer  
a Parameter

FOSTER & BURR q:

Matrix  $\Sigma x_i$  \_\_\_\_\_ Matrix  $\Sigma x_i^2$  \_\_\_\_\_  
 $v$  \_\_\_\_\_  $q$  0.782 \_\_\_\_\_  $q_{.99}$  0.198 \_\_\_\_\_  $q_{.999}$  0.265 \_\_\_\_\_

$$Y_{ijk1} = \mu + T_i + C_j + H_k + (TC)_{ij} + (TH)_{ik} + (CH)_{jk} + \epsilon_{(ijk)1}$$

TOTALS

Temperature ( $T_i$ )

Tube ( $C_j$ )

Time ( $H_k$ )

|                  |                  |                  |
|------------------|------------------|------------------|
| T <sub>1..</sub> | T <sub>1..</sub> | T <sub>1..</sub> |
| T <sub>2..</sub> | T <sub>2..</sub> | T <sub>2..</sub> |
| T <sub>3..</sub> | T <sub>3..</sub> | T <sub>3..</sub> |

Temperature x Tube (TC)<sub>ij</sub>

|                   |                   |                   |
|-------------------|-------------------|-------------------|
| T <sub>11..</sub> | T <sub>21..</sub> | T <sub>31..</sub> |
| T <sub>12..</sub> | T <sub>22..</sub> | T <sub>32..</sub> |
| T <sub>13..</sub> | T <sub>23..</sub> | T <sub>33..</sub> |

Temperature x Time (TH)<sub>ik</sub>

|                   |                   |                   |
|-------------------|-------------------|-------------------|
| T <sub>1.1.</sub> | T <sub>2.1.</sub> | T <sub>3.1.</sub> |
| T <sub>1.2.</sub> | T <sub>2.2.</sub> | T <sub>3.2.</sub> |
| T <sub>1.3.</sub> | T <sub>2.3.</sub> | T <sub>3.3.</sub> |

Tube x Time (CH)<sub>jk</sub>

|                   |                   |                   |
|-------------------|-------------------|-------------------|
| T <sub>.11.</sub> | T <sub>.12.</sub> | T <sub>.13.</sub> |
| T <sub>.21.</sub> | T <sub>.22.</sub> | T <sub>.23.</sub> |
| T <sub>.31.</sub> | T <sub>.32.</sub> | T <sub>.33.</sub> |

TABLE G-12  
ANOVA  
Schweyer  
a Parameter

|        | Holding Time ( $H_k$ ) |                       |              |             |
|--------|------------------------|-----------------------|--------------|-------------|
|        | Tube ( $C_j$ )         | Temperature ( $T_i$ ) |              |             |
|        | 350                    | 375                   | 400          |             |
| 0.5    | E                      | 39.01 E+06            | 0.03851 E+06 | 11.05 E+06  |
|        | F                      | 83.10 E+06            | 39.53 E+06   | 231.1 E+06  |
|        | G                      | 14.75 E+06            | 2.964 E+06   | 1036.0 E+06 |
| 1.0    | E                      | 25.82 E+06            | 48.93 E+06   | 51.31 E+06  |
|        | F                      | 2.382 E+06            | 41.05 E+06   | 37.91 E+06  |
|        | G                      | 10.63 E+06            | 1.442 E+06   | 2.145 E+06  |
| 2.0    | E                      | 152.5 E+06            | 59.85 E+06   | 88.70 E+06  |
|        | F                      | 1064.0 E+06           | 1022.0 E+06  | 423.5 E+06  |
|        | G                      | 74.35 E+06            | 10.37 E+06   | 415.0 E+06  |
| TOTALS | E                      | 722.0 E+06            | 96.54 E+06   | 724.5 E+06  |
|        | F                      | 17.12 E+06            | 24.95 E+06   | 61.34 E+06  |
|        | G                      | 1.403 E+06            | 10.91 E+06   | 63.71 E+06  |
| 0.5    | E                      | 21.79 E+06            | 38.35 E+06   | 5796.0 E+06 |
|        | F                      | 383.3 E+06            | 55.51 E+06   | 96.20 E+06  |
|        | G                      | 256.6 E+06            | 4.927 E+06   | 11.02 E+06  |
| 1.0    | E                      | 29.48 E+06            | 64.14 E+06   | 65.84 E+06  |
|        | F                      | 7.654 E+06            | 20.81 E+06   | 191.7 E+06  |
|        | G                      | 1.676 E+06            | 1.900 E+06   | 2.031 E+06  |
| TOTALS | E                      | 1.900 E+06            | 1.900 E+06   | 2.031 E+06  |

TABLE G-13 (Cont.)

TOTALS - Continued

Temperature x Tube x Time (TCH)<sub>ijk</sub>

|                  |   |       |      |                  |   |       |      |
|------------------|---|-------|------|------------------|---|-------|------|
| T <sub>111</sub> | = | 122.1 | E+06 | T <sub>113</sub> | = | 405.1 | E+06 |
| T <sub>121</sub> | = | 40.57 | E+06 | T <sub>123</sub> | = | 286.1 | E+06 |
| T <sub>131</sub> | = | 13.01 | E+06 | T <sub>133</sub> | = | 9.330 | E+06 |
|                  |   |       |      |                  |   |       |      |
| T <sub>211</sub> | = | 39.57 | E+06 | T <sub>213</sub> | = | 93.86 | E+06 |
| T <sub>221</sub> | = | 51.89 | E+06 | T <sub>223</sub> | = | 69.07 | E+06 |
| T <sub>231</sub> | = | 42.49 | E+06 | T <sub>233</sub> | = | 22.71 | E+06 |
|                  |   |       |      |                  |   |       |      |
| T <sub>311</sub> | = | 242.2 | E+06 | T <sub>313</sub> | = | 5892  | E+06 |
| T <sub>321</sub> | = | 1087  | E+06 | T <sub>323</sub> | = | 76.86 | E+06 |
| T <sub>331</sub> | = | 40.06 | E+06 | T <sub>333</sub> | = | 193.7 | E+06 |

TABLE G-14

ANOVA  
Schweyer  
Log<sub>10</sub> a

Holding Time (H<sub>c</sub>)  
Tube (C<sub>j</sub>)  
Temperature (T<sub>i</sub>)

|        | 350 | 375    | 400    | TOTALS |
|--------|-----|--------|--------|--------|
| 0.5    | E   | 7.5912 | 4.5856 | 7.0434 |
|        | F   | 7.9196 | 7.5969 | 8.3638 |
|        | G   | 7.1688 | 6.4719 | 9.0154 |
| 1.0    | E   | 7.4120 | 7.6896 | 7.7102 |
|        | F   | 6.3769 | 7.6133 | 7.5788 |
|        | G   | 7.0265 | 6.1596 | 6.3314 |
| 2.0    | E   | 8.1833 | 7.7771 | 7.9175 |
|        | F   | 9.0269 | 9.1212 | 8.6269 |
|        | G   | 7.8713 | 7.0158 | 8.6180 |
| TOTALS |     | 8.8585 | 7.9847 | 8.8600 |
|        |     | 7.2335 | 7.3971 | 7.7877 |
|        |     | 6.1471 | 7.0378 | 7.8042 |
| TOTALS |     | 7.3383 | 7.5838 | 9.7631 |
|        |     | 8.5835 | 7.7444 | 7.9832 |
|        |     | 8.4093 | 6.6926 | 7.0422 |
| TOTALS |     | 7.4595 | 7.8071 | 7.8185 |
|        |     | 6.8839 | 7.3183 | 8.2826 |
|        |     | 6.2243 | 6.2788 | 6.3077 |

TABLE G-15  
ANOVA  
Schweyer  
Log<sub>10</sub> a

FOSTER & BURR a:  
 Matrix  $\Sigma x_i$  408.4546      Matrix  $\Sigma x_i^2$  3.135.966.880 E+03  
 $v$  1.000       $q$  0.097       $q_{.99}$  0.198       $q_{.999}$  0.265  
 $Y_{ijk1} = \mu + T_i + C_j + H_k + (TC)_{ij} + (TH)_{ik} + (CH)_{jk} + (TCH)_{ijk} + \epsilon(ijk)1$

TOTALS

| Temperature (T <sub>i</sub> ) | Tube (C <sub>j</sub> )      | Time (H <sub>k</sub> )      |
|-------------------------------|-----------------------------|-----------------------------|
| T <sub>1...</sub> = 135.7246  | T <sub>1..</sub> = 142.7497 | T <sub>..1</sub> = 129.6549 |
| T <sub>2...</sub> = 129.8756  | T <sub>2..</sub> = 139.9154 | T <sub>..2</sub> = 143.2686 |
| T <sub>3...</sub> = 143.8548  | T <sub>3..</sub> = 125.7895 | T <sub>..3</sub> = 135.5311 |
| 408.4550                      | 408.4546                    | 408.4546                    |

Temperature x Tube (TC)<sub>ij</sub>

|                             |                             |                             |
|-----------------------------|-----------------------------|-----------------------------|
| T <sub>11..</sub> = 43.1005 | T <sub>21..</sub> = 50.6529 | T <sub>31..</sub> = 48.9963 |
| T <sub>12..</sub> = 45.4679 | T <sub>22..</sub> = 49.2083 | T <sub>32..</sub> = 45.2392 |
| T <sub>13..</sub> = 41.0865 | T <sub>23..</sub> = 43.4074 | T <sub>33..</sub> = 41.2956 |
| 129.6549                    | 143.2686                    | 135.5311                    |

Temperature x Time (TH)<sub>ik</sub>

|                             |                             |                             |
|-----------------------------|-----------------------------|-----------------------------|
| T <sub>1.1.</sub> = 43.4950 | T <sub>2.1.</sub> = 40.1169 | T <sub>3.1.</sub> = 46.0420 |
| T <sub>1.2.</sub> = 47.3206 | T <sub>2.2.</sub> = 46.3337 | T <sub>3.2.</sub> = 49.6143 |
| T <sub>1.3.</sub> = 44.9088 | T <sub>2.3.</sub> = 43.4250 | T <sub>3.3.</sub> = 47.1973 |
| 135.7244                    | 129.8756                    | 142.8546                    |

Tube x Time (CH)<sub>jk</sub>

|                             |                             |                             |
|-----------------------------|-----------------------------|-----------------------------|
| T <sub>.11.</sub> = 43.1005 | T <sub>.12.</sub> = 50.6529 | T <sub>.13.</sub> = 48.9963 |
| T <sub>.21.</sub> = 45.4679 | T <sub>.22.</sub> = 49.2083 | T <sub>.23.</sub> = 45.2392 |
| T <sub>.31.</sub> = 41.0865 | T <sub>.32.</sub> = 43.4072 | T <sub>.33.</sub> = 41.2956 |
| 129.6549                    | 143.2686                    | 135.5311                    |

TABLE G-15 (Cont.)

TOTALS - Continued

Temperature x Tube x Time (TCH)<sub>ijk</sub>

|                             |                             |                              |
|-----------------------------|-----------------------------|------------------------------|
| T <sub>111.</sub> = 15.5108 | T <sub>112.</sub> = 17.2102 | T <sub>113.</sub> = 15.9218  |
| T <sub>121.</sub> = 14.5807 | T <sub>122.</sub> = 16.7298 | T <sub>123.</sub> = 15.8788  |
| T <sub>131.</sub> = 13.4035 | T <sub>132.</sub> = 13.3806 | T <sub>133.</sub> = 13.1082  |
| 43.4950                     | 47.3206                     | 44.9088                      |
| T <sub>211.</sub> = 12.1825 | T <sub>212.</sub> = 16.8983 | T <sub>213.</sub> = 15.3281  |
| T <sub>221.</sub> = 14.1615 | T <sub>222.</sub> = 15.0005 | T <sub>223.</sub> = 14.4997  |
| T <sub>231.</sub> = 13.7729 | T <sub>232.</sub> = 14.4349 | T <sub>233.</sub> = 13.5970  |
| 40.1169                     | 46.3337                     | 43.4248                      |
| T <sub>311.</sub> = 15.4072 | T <sub>312.</sub> = 16.5444 | T <sub>313.</sub> = 17.7463  |
| T <sub>321.</sub> = 16.7256 | T <sub>322.</sub> = 17.4781 | T <sub>323.</sub> = 14.8607  |
| T <sub>331.</sub> = 13.9102 | T <sub>332.</sub> = 15.5920 | T <sub>333.</sub> = 14.5903  |
| 46.0430                     | 49.6145                     | 47.1973                      |
|                             |                             | T <sub>3... = 142.8546</sub> |

TABLE G-16

ANOVA  
Schweyer  
Logis a

$$\begin{aligned}
 (0) \quad CT &= (T \dots)^2 \div n = (408.4546)^2 \div 54 = 3.089540006 \text{ E}+03 \\
 (1) \quad SS(T_i) &= \frac{1}{18} [(135.7246)^2 + \dots + (142.8548)^2] - \frac{(0)}{CT} = \\
 &= \underline{4.700583000 \text{ E}+00} \\
 (2) \quad SS(C_j) &= \frac{1}{18} [(142.7497)^2 + \dots + (125.7895)^2] - \frac{(0)}{CT} = \\
 &= \underline{9.170790000 \text{ E}+00} \\
 (3) \quad SS(H_k) &= \frac{1}{18} [(29.6549)^2 + \dots + (.135.5311)^2] - \frac{(0)}{CT} = \\
 &= \underline{5.180211000 \text{ E}+00} \\
 (4) \quad SS(TC)_{ij} &= \frac{1}{6} [(43.1005)^2 + \dots + (41.2956)^2] - \frac{(0)}{CT} - \frac{(1)}{SS(T_i)} - \\
 &= \frac{(2)}{SS(C_j)} = \underline{2.756549000 \text{ E}+00} \\
 (5) \quad SS(TH)_{ik} &= \frac{1}{6} [(43.4950)^2 + \dots + (47.1973)^2] - \frac{(0)}{CT} - \frac{(1)}{SS(T_i)} - \\
 &= \frac{(3)}{SS(H_k)} = \underline{3.931520000 \text{ E}+01} \\
 (6) \quad SS(CH)_{jk} &= \frac{1}{6} [(43.1005)^2 + \dots + (41.2956)^2] - \frac{(0)}{CT} - \\
 &= \frac{(3)}{SS(C_j)} - \frac{(3)}{SS(H_k)} = \underline{2.277021000 \text{ E}+00} \\
 (7) \quad SS(TCH)_{ijk} &= \frac{1}{2} [(15.5108)^2 + \dots + (14.5903)^2] \\
 &= \frac{(0)}{CT} - \frac{(1)}{SS(T_i)} - \frac{(2)}{SS(C_j)} - \frac{(3)}{SS(H_k)} \\
 &= \frac{(4)}{SS(TC)_{ij}} - \frac{(5)}{SS(TH)_{ik}} - \frac{(6)}{SS(CH)_{jk}} \\
 &= \underline{3.310444000 \text{ E}+00} \\
 (8) \quad SS(\text{Total}) &= \sum \sum \sum x_{ijk}^2 - \frac{(0)}{CT} = \underline{4.642687400 \text{ E}+01} \\
 (9) \quad SS(\text{Error}) &= SS(\text{Total}) - SS(T_i) - SS(C_j) - SS(H_k) \\
 &= SS(TC)_{ij} - SS(TH)_{ik} - SS(CH)_{jk} - SS(TCH)_{ijk} \\
 &= (8) - (1) - (2) - (3) - (4) - (5) - (6) - (7) \\
 &= \underline{1.863802400 \text{ E}+01}
 \end{aligned}$$

TABLE G-17

ANOVA  
Schweyer  
Logis a

| SOURCE                   | df | SS     | MS    | F    | F.05 | F.01 |
|--------------------------|----|--------|-------|------|------|------|
| (1) T <sub>i</sub>       | 2  | 4.701  | 2.350 | 3.40 | 3.35 | 5.49 |
| (2) C <sub>j</sub>       | 2  | 9.171  | 4.585 | 6.64 | 3.35 | 5.49 |
| (3) H <sub>k</sub>       | 2  | 5.180  | 2.590 | 3.75 | 3.35 | 5.49 |
| (4) (TC) <sub>ij</sub>   | 4  | 2.757  | 0.689 | 1.00 | 2.73 | 4.11 |
| (5) (TH) <sub>ik</sub>   | 4  | 0.993  | 0.098 | 0.14 | 2.73 | 4.11 |
| (6) (CH) <sub>jk</sub>   | 4  | 2.277  | 0.569 | 0.82 | 2.73 | 4.11 |
| (7) (TCH) <sub>ijk</sub> | 8  | 3.310  | 0.414 | 4.80 | 2.31 | 3.26 |
| (9) Error                | 27 | 18.638 | 0.690 | -    | -    | -    |
| (8) Total                | 53 | 46.427 | -     | -    | -    | -    |

TABLE G-18

ANOVA  
Schweyer  
 $R^2 (\eta_a = a \{b\})$

|        | Holding Time ( $H_c$ ) |                       | 375    | 400    |
|--------|------------------------|-----------------------|--------|--------|
|        | Tube ( $C_j$ )         | Temperature ( $T_i$ ) |        |        |
| E      | 350                    | 392                   | 0.755  | 0.834  |
|        | E-04                   | E-04                  | 0.184  | 0.0577 |
| F      | 350                    | 392                   | 4.20   | 0.018  |
|        | E-04                   | E-04                  | 0.593  | 0.280  |
| G      | 350                    | 392                   | 0.961  | 0.567  |
|        | E-04                   | E-04                  | 0.971  | 0.926  |
| E      | 350                    | 392                   | 2.00   | 0.255  |
|        | E-04                   | E-04                  | 0.223  | 4.03   |
| F      | 350                    | 392                   | 3.20   | 0.0869 |
|        | E-04                   | E-04                  | 0.845  | 50.6   |
| G      | 350                    | 392                   | 0.959  | 0.863  |
|        | E-04                   | E-04                  | 0.795  | 0.646  |
| E      | 350                    | 392                   | 0.684  | 0.288  |
|        | E-04                   | E-04                  | 0.0142 | 57.2   |
| F      | 350                    | 392                   | 0.273  | 0.671  |
|        | E-04                   | E-04                  | 0.804  | 31.3   |
| G      | 350                    | 392                   | 0.875  | 0.671  |
|        | E-04                   | E-04                  | 0.972  | 0.986  |
| TOTALS |                        |                       |        |        |

TABLE G-19

ANOVA  
Schweyer  
 $R^2$

FOSTER & BURR g:

Matrix  $I \times_i$  26.103 Matrix  $I \times_i^2$  2.078 522 622 E-01  
 $v$  1.000  $q$  0.092  $q$  0.198  $q$  0.999 0.265

$Y_{ijkl} = \mu + T_i + C_j + H_k + (TC)_{ij} + (TH)_{ik} + (CH)_{jk} + (TCH)_{ijk} + \epsilon_{(ijk)l}$

TOTALS

| Temperature ( $T_i$ ) | Tube ( $C_j$ )      | Time ( $H_k$ )     |
|-----------------------|---------------------|--------------------|
| $T_{1..}$ = 9.030     | $T_{.1..}$ = 3.795  | $T_{...}$ = 9.500  |
| $T_{2..}$ = 9.909     | $T_{.2..}$ = 6.785  | $T_{...}$ = 7.333  |
| $T_{3..}$ = 7.164     | $T_{.3..}$ = 15.523 | $T_{...}$ = 9.270  |
| $T_{...}$ = 26.103    | $T_{...}$ = 26.103  | $T_{...}$ = 26.103 |

Temperature x Tube (TC)  $_{ij}$

|                    |                    |                    |
|--------------------|--------------------|--------------------|
| $T_{11..}$ = 0.493 | $T_{21..}$ = 1.860 | $T_{31..}$ = 1.441 |
| $T_{12..}$ = 3.205 | $T_{22..}$ = 2.516 | $T_{32..}$ = 1.064 |
| $T_{13..}$ = 5.331 | $T_{23..}$ = 5.532 | $T_{33..}$ = 4.659 |
| $T_{...}$ = 9.029  | $T_{...}$ = 9.908  | $T_{...}$ = 7.164  |

Temperature x Time (TH)  $_{ik}$

|                   |                   |                   |
|-------------------|-------------------|-------------------|
| $T_{11.}$ = 3.353 | $T_{21.}$ = 3.464 | $T_{31.}$ = 2.683 |
| $T_{12.}$ = 2.654 | $T_{22.}$ = 2.823 | $T_{32.}$ = 1.856 |
| $T_{13.}$ = 3.023 | $T_{23.}$ = 3.622 | $T_{33.}$ = 2.625 |
| $T_{...}$ = 9.029 | $T_{...}$ = 9.909 | $T_{...}$ = 7.164 |

Tube x Time (CH)  $_{jk}$

|                    |                    |                    |
|--------------------|--------------------|--------------------|
| $T_{.11.}$ = 1.870 | $T_{.12.}$ = 0.717 | $T_{.13.}$ = 1.208 |
| $T_{.21.}$ = 2.298 | $T_{.22.}$ = 1.735 | $T_{.23.}$ = 2.751 |
| $T_{.31.}$ = 5.332 | $T_{.32.}$ = 4.880 | $T_{.33.}$ = 5.311 |
| $T_{...}$ = 9.500  | $T_{...}$ = 7.332  | $T_{...}$ = 9.270  |

TABLE G-19 (Cont.)

TOTALS - Continued

Temperature x Tube x Time (TCH)<sub>ijk</sub>

|                  |         |                  |         |
|------------------|---------|------------------|---------|
| T <sub>111</sub> | = 0.039 | T <sub>112</sub> | = 0.238 |
| T <sub>121</sub> | = 1.407 | T <sub>122</sub> | = 0.798 |
| T <sub>131</sub> | = 1.907 | T <sub>132</sub> | = 1.617 |
|                  | 3.353   |                  | 2.653   |
| T <sub>211</sub> | = 0.939 | T <sub>212</sub> | = 0.223 |
| T <sub>221</sub> | = 0.593 | T <sub>222</sub> | = 0.845 |
| T <sub>231</sub> | = 1.932 | T <sub>232</sub> | = 1.754 |
|                  | 3.464   |                  | 2.822   |
| T <sub>311</sub> | = 0.892 | T <sub>312</sub> | = 0.255 |
| T <sub>321</sub> | = 0.298 | T <sub>322</sub> | = 0.092 |
| T <sub>331</sub> | = 1.493 |                  | 1.509   |
|                  | 2.683   |                  | 1.856   |

|                  |         |
|------------------|---------|
| T <sub>113</sub> | = 0.216 |
| T <sub>223</sub> | = 1.000 |
| T <sub>333</sub> | = 1.807 |
|                  | 3.023   |
| T <sub>213</sub> | = 0.698 |
| T <sub>223</sub> | = 1.077 |
| T <sub>233</sub> | = 1.847 |
|                  | 3.622   |
| T <sub>313</sub> | = 0.294 |
| T <sub>323</sub> | = 0.674 |
| T <sub>333</sub> | = 1.657 |
|                  | 2.625   |

TABLE G-20

ANOVA  
Schweyer  
R<sup>2</sup>

- (0)  $CT = (T_{\dots})^2 + n = (26.103)^2 \div 54 = 1.261790017 E+01$
- (1)  $SS(T_i) = \frac{1}{18} [(9.030)^2 + \dots + (7.164)^2] - (0) = 2.183263300 E-01$
- (2)  $SS(C_j) = \frac{1}{18} [(3.795)^2 + \dots + (15.523)^2] - (0) = 4.126643110 E+00$
- (3)  $SS(H_k) = \frac{1}{18} [(9.500)^2 + \dots + (9.270)^2] - (0) = 1.549776100 E-01$
- (4)  $SS(TC)_{ij} = \frac{1}{6} [(0.493)^2 + \dots + (4.659)^2] - (0) - SS(T_i) - SS(C_j)$   
 $SS(C_j) = 4.108592200 E-01$
- (5)  $SS(TH)_{ik} = \frac{1}{6} [(3.353)^2 + \dots + (2.625)^2] - (0) - SS(T_i) - SS(H_k)$   
 $SS(H_k) = 1.649806000 E-02$
- (6)  $SS(CH)_{jk} = \frac{1}{6} [(1.870)^2 + \dots + (5.311)^2] - (0) - SS(C_j) - SS(H_k)$   
 $SS(C_j) = 6.391711000 E-02$
- (7)  $SS(TCH)_{ijk} = \frac{1}{2} [(0.039)^2 + \dots + (1.657)^2] - (0) - SS(T_i) - SS(C_j) - SS(H_k)$   
 $SS(TCH)_{ijk} = 3.174358900 E-01$
- (8)  $SS(Total) = \sum_{ijk} T_{ijk}^2 - (0) = 8.167326050 E+00$
- (9)  $SS(Error) = SS(Total) - SS(T_i) - SS(C_j) - SS(H_k) - SS(TC)_{ij} - SS(TH)_{ik} - SS(CH)_{jk} - SS(TCH)_{ijk}$   
 $= (8) - (1) - (2) - (3) - (4) - (5) - (6) - (7) = 2.858568720 E+00$

TABLE G-21

ANOVA  
Schweyer  
R<sub>2</sub>

| SOURCE             | df | SS    | MS    | F     | F.05 | F.01 |
|--------------------|----|-------|-------|-------|------|------|
| (1) T <sub>i</sub> | 2  | 0.218 | 0.109 | 1.03  | 3.35 | 5.49 |
| (2) C <sub>j</sub> | 2  | 4.127 | 2.063 | 19.49 | 3.35 | 5.49 |
| (3) R <sub>k</sub> | 2  | 0.155 | 0.077 | 0.73  | 3.35 | 5.49 |
| (4) (TC) ij        | 4  | 0.411 | 0.103 | 0.97  | 2.73 | 4.11 |
| (5) (TH) ik        | 4  | 0.016 | 0.004 | 0.04  | 2.73 | 4.11 |
| (6) (CH) jk        | 4  | 0.064 | 0.016 | 0.15  | 2.73 | 4.11 |
| (7) (TCH) ijk      | 8  | 0.317 | 0.040 | 0.38  | 2.31 | 3.26 |
| (9) ERROR          | 27 | 2.859 | 0.106 |       |      |      |
| (8) Total          | 53 |       |       |       |      |      |

TABLE G-22

TUBE E  
Schweyer  
Logu a

|     | Time Temperature |        |        |
|-----|------------------|--------|--------|
|     | 350              | 375    | 400    |
| 0.5 | 7.5912           | 4.5856 | 7.0434 |
|     | 7.9196           | 7.5969 | 8.3638 |
| 1.0 | 8.1833           | 7.7771 | 7.9175 |
|     | 9.0269           | 9.1212 | 8.6269 |
| 2.0 | 7.3383           | 7.5838 | 9.7631 |
|     | 8.5835           | 7.7444 | 7.9832 |

TABLE G-23

FOSTER & BURR g: TUBE E  
Schweyer Log<sub>10</sub> a

Matrix  $\Sigma x_i$  142.7497  
Matrix  $\Sigma x_i^2$  1.151 957 216 E+03  
v 1.000  
g 0.291 0.576 0.999 0.750

$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon(ijk)$

TOTALS

Temperature (T<sub>i</sub>)

T<sub>1..</sub> = 48.6428  
T<sub>2..</sub> = 44.4090  
T<sub>3..</sub> = 49.6979  
142.7497  
Time (H<sub>j</sub>)  
T<sub>.1.</sub> = 43.1005  
T<sub>.2.</sub> = 50.6529  
T<sub>.3.</sub> = 48.9953  
142.7497

Temperature x Time (TH)<sub>ij</sub>

T<sub>11.</sub> = 15.5108  
T<sub>12.</sub> = 17.2102 48.6428  
T<sub>13.</sub> = 15.9218  
T<sub>21.</sub> = 12.1825  
T<sub>22.</sub> = 16.8983 44.4090  
T<sub>23.</sub> = 15.3282  
T<sub>31.</sub> = 15.4072  
T<sub>32.</sub> = 16.5444 49.6979  
T<sub>33.</sub> = 17.7463

T... = 142.7497

$\Sigma \Sigma Y_{ijk}$

TABLE G-24

TUBE E  
Schweyer Log<sub>10</sub> a

(0) CT = (T...)<sup>2</sup> ÷ n = (142.7497)<sup>2</sup> ÷ ( 18 ) = 1.132 082 047 E+03  
(1) SS (Temp.) =  $\frac{1}{(6)} [(48.6428)^2 + \dots + (49.6979)^2] - CT = 2.611 709 000 E+00$   
(2) SS (Time) =  $\frac{1}{(6)} [(43.1005)^2 + \dots + (48.9953)^2] - CT = 5.252 418 000 E+00$   
(3) SS (H x T) =  $\frac{1}{(2)} [(05.5108)^2 + \dots + (07.7463)^2] - CT - SS (T) - SS (H)$   
= 2.658 490 000 E+00

(4) SS (Total) =  $\Sigma \Sigma x_{ijk}^2 - CT = 1.987 516 900 E+01$

(5) SS (Error) = SS (Total) - SS (T) - SS (H) - SS (T x H)

= (4) - (1) - (2) - (3) = 9.342 552 000 E+00

| Source                 | df | ANALYSIS OF VARIANCE |        |      |      |
|------------------------|----|----------------------|--------|------|------|
|                        |    | SS                   | MS     | F    | F.05 |
| (1) T <sub>i</sub>     | 2  | 2.6117               | 1.3059 | 1.26 | 4.26 |
| (2) H <sub>j</sub>     | 2  | 5.2524               | 2.6262 | 2.53 | 4.26 |
| (3) (TH) <sub>ij</sub> | 4  | 2.6685               | 0.6671 | 0.64 | 3.63 |
| (5) Error              | 9  | 9.3426               | 1.0381 |      |      |
| (4) Total              | 17 | 19.8752              |        |      |      |

TABLE G-26

Tube F

Schweyer Log<sub>10</sub> a

FOSTER & BURR q:

Matrix Ex<sub>i</sub> = 139.9154

Matrix Ex<sub>i</sub><sup>2</sup> = 1.097 219 275 E+03

v = 1.000

q = 0.152      q<sub>0.99</sub> = 0.576      q<sub>0.999</sub> = 0.750

Time  
Temperature

|     |        |        |        |
|-----|--------|--------|--------|
|     | 350    | 375    | 400    |
| 0.5 | 7.1688 | 6.4719 | 9.0154 |
|     | 7.4120 | 7.6896 | 7.7102 |
| 1.0 | 7.8713 | 7.0158 | 8.6180 |
|     | 8.8585 | 7.9847 | 8.8600 |
| 2.0 | 8.4093 | 6.6926 | 7.0422 |
|     | 7.4695 | 7.8071 | 7.8185 |

$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon_{(ijk)}$

TOTALS

Temperature (T<sub>i</sub>)

T<sub>1..</sub> = 47.1894

T<sub>2..</sub> = 43.6617

T<sub>3..</sub> = 49.0643

T<sub>...</sub>  = 139.9154

Time (H<sub>j</sub>)

T<sub>.1.</sub> = 45.4679

T<sub>.2.</sub> = 49.2083

T<sub>.3.</sub> = 45.2392

T<sub>...</sub>  = 139.9154

Temperature x Time (TH)<sub>ij</sub>

T<sub>11.</sub> = 14.5808

T<sub>12.</sub> = 16.7298

T<sub>13.</sub> = 15.8788

T<sub>21.</sub> = 14.1615

T<sub>22.</sub> = 15.0005

T<sub>23.</sub> = 14.4997

T<sub>31.</sub> = 16.7256

T<sub>32.</sub> = 17.4780

T<sub>33.</sub> = 14.8607

T<sub>...</sub>  =

TH<sub>ijk</sub> =

TABLE G-27

ANOVA

Tube F

Schweyer Log<sub>10</sub> a

- (0)  $CT = (T \dots)^2 \div n = (139.954)^2 \div (18) = 1.087\ 573\ 287\ E+03$
- (1)  $SS\ (Temp.) = \frac{1}{6} [(47.1894)^2 + \dots + (49.0643)^2] - CT = 2.508\ 222\ 000\ E+03$
- (2)  $SS\ (Time) = \frac{1}{6} [(45.4679)^2 + \dots + (45.2392)^2] - CT = 1.555\ 369\ 000\ E+03$
- (3)  $SS\ (H \times T) = \frac{1}{2} [(14.5808)^2 + \dots + (14.8607)^2] - CT - SS\ (T) - SS\ (H)$   
 $= 1.509\ 719\ 000\ E+00$
- (4)  $SS\ (Total) = \sum \sum x_{ijk}^2 - CT = 9.645\ 988\ 000\ E+00$
- (5)  $SS\ (Error) = SS\ (Total) - SS\ (T) - SS\ (H) - SS\ (T \times H)$   
 $= (4) - (1) - (2) - (3) = 3.972\ 678\ 000\ E+00$

| ANALYSIS OF VARIANCE |    |        |        |      |      |      |
|----------------------|----|--------|--------|------|------|------|
| Source               | df | SS     | MS     | F    | F.05 | F.01 |
| T <sub>i</sub>       | 2  | 2.5082 | 1.2541 | 2.84 | 4.26 | 8.02 |
| H <sub>j</sub>       | 2  | 1.5554 | 0.8277 | 1.88 | 4.26 | 8.02 |
| (TH) <sub>ij</sub>   | 4  | 1.5097 | 0.3774 | 0.86 | 3.63 | 6.42 |
| Error                | 9  | 3.9727 | 0.4414 |      |      |      |
| Total                | 17 | 9.5460 |        |      |      |      |

TABLE G-28

TUBE G  
Schweyer  
Log<sub>10</sub> a

|     | Time Temperature |        |        |
|-----|------------------|--------|--------|
|     | 350              | 375    | 400    |
| 0.5 | 6.3769           | 7.6133 | 7.5788 |
|     | 7.0265           | 6.1596 | 6.3314 |
| 1.0 | 7.2335           | 7.3971 | 7.7877 |
|     | 6.1471           | 7.0378 | 7.8042 |
| 2.0 | 6.8839           | 7.3183 | 8.2826 |
|     | 6.2243           | 6.2788 | 6.3077 |

TABLE G-30

Tube G

Schweyer Log<sub>10</sub> a

- (0)  $CT = (T_{...})^2 \div n = (125.7895)^2 \div (18) = 8.790\ 554\ 617\ E+02$
- (1)  $SS\ (Temp.) = \frac{1}{(6)} [(99.8922)^2 + \dots + (44.0924)^2] - CT = 1.474\ 042\ 100\ E+$
- (2)  $SS\ (Time) = \frac{1}{(6)} [(41.0865)^2 + \dots + (41.2956)^2] - CT = 5.494\ 443\ 000\ E-01$
- (3)  $SS\ (H \times T) = \frac{1}{(2)} [(13.4034)^2 + \dots + (14.5903)^2] - CT - SS\ (T) - SS\ (H)$   
 $= 3.883\ 519\ 000\ E-01$

(4)  $SS\ (Total) = \sum \sum x_{ijk}^2 - CT = 7.820\ 201\ 600\ E+00$

(5)  $SS\ (Error) = SS\ (Total) - SS\ (T) - SS\ (H) - SS\ (T \times H)$

$= (4) - (1) - (2) - (3) = 5.408\ 363\ 300\ E+00$

TABLE G-29

Tube G

Schweyer Log<sub>10</sub> a

FOSTER & BURR c:

|                     |                           |              |              |
|---------------------|---------------------------|--------------|--------------|
| Matrix $\sum x_i$   | <u>125.7895</u>           |              |              |
| Matrix $\sum x_i^2$ | <u>8.868 756 633 E+02</u> |              |              |
| $v$                 | <u>1.000</u>              |              |              |
| $q$                 | <u>0.214</u>              | <u>0.576</u> | <u>0.750</u> |
|                     | $q_{0...}$                | $q_{...}$    | $q_{...}$    |

$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon_{(ij)k}$

TOTALS

Temperature ( $T_i$ )

|           |                 |                |                     |
|-----------|-----------------|----------------|---------------------|
| $T_{1..}$ | = 39.8922       | Time ( $H_j$ ) | $T_{.1.}$ = 41.0865 |
| $T_{2..}$ | = 41.8049       |                | $T_{.2.}$ = 43.4074 |
| $T_{3..}$ | = 44.0924       |                | $T_{.3.}$ = 41.2956 |
|           | <u>125.7895</u> |                | <u>125.7895</u>     |

Temperature x Time (TH)  $_{ij}$

|           |           |
|-----------|-----------|
| $T_{11.}$ | = 13.4034 |
| $T_{12.}$ | = 13.3806 |
| $T_{13.}$ | = 13.1082 |
| $T_{21.}$ | = 13.7729 |
| $T_{22.}$ | = 14.4349 |
| $T_{23.}$ | = 13.5971 |
| $T_{31.}$ | = 13.9102 |
| $T_{32.}$ | = 15.5919 |
| $T_{33.}$ | = 14.5903 |

T...

$\sum \sum Y_{ijk}$

| ANALYSIS OF VARIANCE |    |        |        |      |      |      |
|----------------------|----|--------|--------|------|------|------|
| Source               | df | SS     | MS     | F    | F.05 | F.01 |
| (1) $T_i$            | 2  | 1.4740 | 0.7370 | 1.23 | 4.26 | 8.02 |
| (2) $H_j$            | 2  | 0.5494 | 0.2747 | 0.46 | 4.26 | 8.02 |
| (3) $(TH)_{ij}$      | 4  | 0.3884 | 0.0971 | 0.16 | 3.63 | 6.42 |
| (5) Error            | 9  | 5.4084 | 0.6009 |      |      |      |
| (4) Total            | 17 | 7.8202 |        |      |      |      |

TABLE G-31

TUBE E  
Schweyer C

| Time | Temperature |       |       |
|------|-------------|-------|-------|
|      | 350         | 375   | 400   |
| 0.5  | 1.003       | 0.468 | 0.516 |
|      | 0.911       | 0.890 | 1.087 |
| 1.0  | 0.846       | 1.014 | 0.730 |
|      | 1.071       | 0.809 | 0.988 |
| 2.0  | 1.108       | 0.665 | 1.485 |
|      | 1.105       | 0.987 | 0.974 |

TABLE G-32

Tube E  
Schweyer c

FOSTER & BURR q:

Matrix  $Ex_i$  = 16.657

Matrix  $Ex_i^2$  = 1.636 834 100 E+01

$v$  = 1.000

$q$  = 0.210

$q_{0.99}$  = 0.576

$q_{0.99}$  = 0.750

$$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon_{(ijk)}$$

TOTALS

Temperature ( $T_i$ )

$T_{1..}$  = 6.044  
 $T_{2..}$  = 4.833  
 $T_{3..}$  = 5.780  
 = 16.657

Time ( $H_j$ )

$T_{.1.}$  = 4.875  
 $T_{.2.}$  = 5.458  
 $T_{.3.}$  = 6.324  
 = 16.657

Temperature x Time (TH)  $ij$

$T_{11.}$  = 1.914  
 $T_{12.}$  = 1.917  
 $T_{13.}$  = 2.213

$T_{21.}$  = 1.358  
 $T_{22.}$  = 1.823  
 $T_{23.}$  = 1.652

$T_{31.}$  = 1.603  
 $T_{32.}$  = 1.718  
 $T_{33.}$  = 2.459

$T_{...}$  =

$EXY_{ijk}$  =

TABLE G-33

ANOVA  
Tube E  
Schweyer c

- (0)  $CT = (T \dots)^2 \div n = (16.657)^2 \div (18) = 1.541420272 E+01$
- (1)  $SS (Temp.) = \frac{1}{(6)} [(6.044)^2 + \dots + (5.780)^2] - CT = 1.351581100 E$
- (2)  $SS (Time) = \frac{1}{(6)} [(4.875)^2 + \dots + (6.324)^2] - CT = 1.771914500 E-0$
- (3)  $SS (H \times T) = \frac{1}{(2)} [(1.914)^2 + \dots + (2.459)^2] - CT - SS (T) - SS (H)$   
 $= 1.234702200 E-01$
- (4)  $SS (Total) = \sum \sum x_{ijk}^2 - CT = 9.541382800 E-01$
- (5)  $SS (Error) = SS (Total) - SS (T) - SS (H) - SS (T \times H)$   
 $= (4) - (1) - (2) - (3) = 5.183085000 E-01$

TABLE G-34

TUBE F  
Schweyer C

|     | Time Temperature |       |       |
|-----|------------------|-------|-------|
|     | 350              | 375   | 400   |
| 0.5 | 0.568            | 0.996 | 1.042 |
| 1.0 | 0.715            | 0.723 | 0.801 |
| 2.0 | 0.755            | 1.021 | 0.896 |
|     | 0.969            | 0.682 | 1.030 |
|     | 1.036            | 0.699 | 0.834 |
|     | 0.739            | 0.759 | 1.013 |

| ANALYSIS OF VARIANCE |    |        |        |      |      |      |
|----------------------|----|--------|--------|------|------|------|
| Source               | df | SS     | MS     | F    | F.05 | F.01 |
| (1) $T_i$            | 2  | 0.1352 | 0.0676 | 1.17 | 4.26 | 8.02 |
| (2) $H_j$            | 2  | 0.1772 | 0.0886 | 1.54 | 4.26 | 8.02 |
| (3) $(TH)_{ij}$      | 4  | 0.1235 | 0.0309 | 0.54 | 3.63 | 6.42 |
| (5) Error            | 9  | 0.5183 | 0.0576 |      |      |      |
| (4) Total            | 17 | 0.9541 |        |      |      |      |

TABLE G-35

Tube F  
Schweyer c

FOSTER & BURR q:

Matrix  $\Sigma x_i$  15.378

Matrix  $\Sigma x_i^2$  1.348 565 000 E+01

v 1.000

q 0.175  $q_{0.99}$  0.576  $q_{0.999}$  0.750

$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon_{(ij)k}$

TOTALS

Temperature ( $T_i$ )

- $T_{1..} = 4.882$
- $T_{2..} = 4.880$
- $T_{3..} = 5.616$
- 15.378

Time ( $H_j$ )

- $T_{.1.} = 4.945$
- $T_{.2.} = 5.353$
- $T_{.3.} = 5.080$
- 15.378

Temperature x Time (TH)  $_{ij}$

- $T_{11.} = 1.383$
- $T_{12.} = 1.724$
- $T_{13.} = 1.775$
- $T_{21.} = 1.719$
- $T_{22.} = 1.703$
- $T_{23.} = 1.458$
- $T_{31.} = 1.843$
- $T_{32.} = 1.926$
- $T_{33.} = 1.847$

$T_{...} =$

$\Sigma \Sigma Y_{ijk} =$

TABLE G-36

ANOVA  
Tube F  
Schweyer c

(0)  $CT = (T_{...})^2 \div n = (15.378)^2 \div (18) = 1.313 793 800 E+01$

(1)  $SS (\text{Temp.}) = \frac{1}{(6)} [(4.882)^2 + \dots + (5.616)^2] - CT = 6.002 533 000 E-$

(2)  $SS (\text{Time}) = \frac{1}{(6)} [(4.945)^2 + \dots + (5.080)^2] - CT = 1.440 100 000 E-0:$

(3)  $SS (H \times T) = \frac{1}{(2)} [(1.383)^2 + \dots + (1.847)^2] - CT - SS (T) - SS (H)$   
 $= 5.461 467 000 E-02$

(4)  $SS (\text{Total}) = \Sigma \Sigma x_{ijk}^2 - CT = 3.477 120 000 E-01$

(5)  $SS (\text{Error}) = SS (\text{Total}) - SS (T) - SS (H) - SS (T \times H)$   
 $= (4) - (1) - (2) - (3) = 2.186 710 000 E-01$

| ANALYSIS OF VARIANCE |    |        |        |      |      |      |
|----------------------|----|--------|--------|------|------|------|
| Source               | df | SS     | MS     | F    | F.05 | F.01 |
| (1) $T_i$            | 2  | 0.0600 | 0.0300 | 1.24 | 4.26 | 8.02 |
| (2) $H_j$            | 2  | 0.0144 | 0.0072 | 0.30 | 4.26 | 8.02 |
| (3) $(TH)_{ij}$      | 4  | 0.0546 | 0.0137 | 0.56 | 3.63 | 6.42 |
| (5) Error            | 9  | 0.2187 | 0.0243 |      |      |      |
| (4) Total            | 17 | 0.3477 |        |      |      |      |

TABLE G-37

TUBE G  
Schweyer C

| Time | Temperature |       |       |
|------|-------------|-------|-------|
|      | 350         | 375   | 400   |
| 0.5  | 0.450       | 0.393 | 0.674 |
|      | 0.618       | 0.391 | 0.410 |
| 1.0  | 0.699       | 0.450 | 0.797 |
|      | 0.299       | 0.500 | 0.722 |
| 2.0  | 0.567       | 0.456 | 0.570 |
|      | 0.374       | 0.506 | 0.508 |

TABLE G-38

Tube G  
Schweyer c

FOSTER & BURR q:

|                       |                          |
|-----------------------|--------------------------|
| Matrix $\Sigma x_i$   | 9.384                    |
| Matrix $\Sigma x_i^2$ | 5.209 426 000 E+00       |
| v                     | 1.000                    |
| q                     | 0.341                    |
|                       | q <sub>0.999</sub> 0.576 |
|                       | q <sub>0.999</sub> 0.750 |

$$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon_{(ij)k}$$

TOTALS

Temperature (T<sub>i</sub>)

|                  |   |       |
|------------------|---|-------|
| T <sub>1..</sub> | = | 3.007 |
| T <sub>2..</sub> | = | 2.696 |
| T <sub>3..</sub> | = | 3.681 |
|                  | = | 9.384 |

Time (H<sub>j</sub>)

|                  |   |       |
|------------------|---|-------|
| T <sub>.1.</sub> | = | 2.936 |
| T <sub>.2.</sub> | = | 3.467 |
| T <sub>.3.</sub> | = | 2.981 |
|                  | = | 9.384 |

Temperature x Time (TH)<sub>ij</sub>

|                  |   |       |
|------------------|---|-------|
| T <sub>11.</sub> | = | 1.068 |
| T <sub>12.</sub> | = | 0.998 |
| T <sub>13.</sub> | = | 0.941 |

|                  |   |       |
|------------------|---|-------|
| T <sub>21.</sub> | = | 0.784 |
| T <sub>22.</sub> | = | 0.950 |
| T <sub>23.</sub> | = | 0.962 |

|                  |   |       |
|------------------|---|-------|
| T <sub>31.</sub> | = | 1.084 |
| T <sub>32.</sub> | = | 1.519 |
| T <sub>33.</sub> | = | 1.078 |

T... =  
 $\Sigma \Sigma Y_{ijk}$  =

TABLE G-39  
ANOVA  
Tube G  
Schweyer c

$$(0) CT = (T...)^2 \div n = (9.384)^2 \div (18) = 4.892192000E+00$$

$$(1) SS (Temp.) = \frac{1}{(6)} [(3.007)^2 + \dots + (3.681)^2] - CT = 8.451233300E02$$

$$(2) SS (Time) = \frac{1}{(6)} [(2.936)^2 + \dots + (2.981)^2] - CT = 2.889900000E02$$

$$(3) SS (H \times T) = \frac{1}{(2)} [(1.068)^2 + \dots + (1.078)^2] - CT - SS (T) - SS (H) = 4.900166700E-02$$

$$(4) SS (Total) = \sum \sum x_{ijk}^2 - CT = 3.172340000E-01$$

$$(5) SS (Error) = SS (Total) - SS (T) - SS (H) - SS (T \times H) = (4) - (1) - (2) - (3) = 1.548210000E-012$$

TABLE G-40

TUBE E  
Schweyer R<sup>2</sup>

|     | Time Temperature |          |          |
|-----|------------------|----------|----------|
|     | 350              | 375      | 400      |
| 0.5 | 0.000147         | 0.755    | 0.834    |
|     | 0.000392         | 0.194    | 0.0577   |
| 1.0 | 0.228            | 0.000200 | 0.255    |
|     | 0.0104           | 0.223    | 0.000403 |
| 2.0 | 0.213            | 0.684    | 0.288    |
|     | 0.00262          | 0.0142   | 0.000572 |

| ANALYSIS OF VARIANCE   |    |        |        |      |      |      |
|------------------------|----|--------|--------|------|------|------|
| Source                 | df | SS     | MS     | F    | F.05 | F.01 |
| (1) T <sub>i</sub>     | 2  | 0.0845 | 0.0423 | 2.46 | 4.26 | 8.02 |
| (2) H <sub>j</sub>     | 2  | 0.0289 | 0.0144 | 0.84 | 4.26 | 8.02 |
| (3) (TH) <sub>ij</sub> | 4  | 0.0490 | 0.0123 | 0.71 | 3.63 | 6.42 |
| (5) Error              | 9  | 0.1548 | 0.0172 |      |      |      |
| (4) Total              | 17 | 0.3172 |        |      |      |      |

TABLE G-41  
Tube E  
Schweyer R

FOSTER & BURR q:

Matrix  $Ex_1$  3.750 634 000 E+00  
 Matrix  $Ex_1^2$  2.065 990 649 E+00  
 $v$  1.000  
 $q$  0.248  $q_{0.99}$  0.576  $q_{0.999}$  0.750

$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon_{(ijk)}$

TOTALS

Temperature ( $T_i$ )  
 $T_{1..} = 0.455$   
 $T_{2..} = 1.860$   
 $T_{3..} = 1.436$   
 $T_{...} = 3.751$

Time ( $H_j$ )  
 $T_{.1.} = 1.831$   
 $T_{.2.} = 0.717$   
 $T_{.3.} = 1.202$   
 $T_{.3.} = 3.751$

Temperature x Time ( $TH$ )<sub>ij</sub>

$T_{11.} = 0.000539$   
 $T_{12.} = 0.238$   
 $T_{13.} = 0.216$   
 $T_{21.} = 0.939$   
 $T_{22.} = 0.223$   
 $T_{23.} = 0.698$   
 $T_{31.} = 0.892$   
 $T_{32.} = 0.255$   
 $T_{33.} = 0.289$

$T_{...} =$   
 $TH_{ijk} =$

TABLE G-42  
ANOVA  
Tube E  
Schweyer R

(0)  $CT = (T_{...})^2 \div n = (3.750 634 000 E+00)^2 \div (18) = 7.815 141 889 E-0$   
 (1)  $SS (Temp.) = \frac{1}{6} [(0.455)^2 + \dots + (1.436)^2] - CT = 1.732 736 444 E-0$   
 (2)  $SS (Time) = \frac{1}{6} [(1.831)^2 + \dots + (1.202)^2] - CT = 1.037 281 444 E-0$   
 (3)  $SS (H \times T) = \frac{1}{2} [(0.000 539)^2 + \dots + (0.289)^2] - CT - SS (T) -$

$SS (H) = 1.745 671 683 E-01$

(4)  $SS (Total) = \sum \sum x_{ijk}^2 - CT = 1.284 476 470 E+00$

(5)  $SS (Error) = SS (Total) - SS (T) - SS (H) - SS (T \times H)$

$= (4) - (1) - (2) - (3) = 8.329 085 137 E-01$

| ANALYSIS OF VARIANCE |    |        |        |      |      |      |
|----------------------|----|--------|--------|------|------|------|
| Source               | df | SS     | MS     | F    | F.05 | F.01 |
| (1) $T_i$            | 2  | 0.1733 | 0.0866 | 0.94 | 4.26 | 8.02 |
| (2) $H_j$            | 2  | 0.1037 | 0.0519 | 0.56 | 4.26 | 8.02 |
| (3) $(TH)_{ij}$      | 4  | 0.1746 | 0.0436 | 0.47 | 3.63 | 6.42 |
| (5) Error            | 9  | 0.8329 | 0.0925 |      |      |      |
| Total                | 17 | 1.2845 |        |      |      |      |

TABLE G-43

TUBE F  
Schweyer R<sup>2</sup>

| Time | Temperature | 350      | 375     | 400   |
|------|-------------|----------|---------|-------|
| 0.5  | 0.884       | 0.000420 | 0.018   | 0.018 |
|      | 0.513       | 0.593    | 0.280   |       |
| 1.0  | 0.795       | 0.000320 | 0.0869  |       |
|      | 0.00311     | 0.845    | 0.00506 |       |
| 2.0  | 0.0606      | 0.273    | 0.671   |       |
|      | 0.994       | 0.804    | 0.00313 |       |

TABLE G-44

Tube F  
Schweyer R<sup>2</sup>

FOSTER & BURR q:

Matrix  $\Sigma x_i$  = 6.839 540.000 E+00  
 Matrix  $\Sigma x_i^2$  = 5.009 319 321 E+00  
 $\bar{v}$  = 1.000  
 $\bar{q}$  = 0.169  
 $q_{0.99}$  = 0.576  
 $q_{0.999}$  = 0.750

$$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon(ij)k$$

TOTALS

Temperature (T<sub>i</sub>)

T<sub>1..</sub> = 3.260  
 T<sub>2..</sub> = 2.516  
 T<sub>3..</sub> = 1.064  
 = 6.840

Time (H<sub>j</sub>)

T<sub>.1.</sub> = 2.298  
 T<sub>.2.</sub> = 1.735  
 T<sub>.3.</sub> = 2.806  
 = 6.840

Temperature x Time (TH)<sub>ij</sub>

T<sub>11.</sub> = 1.407  
 T<sub>12.</sub> = 0.798  
 T<sub>13.</sub> = 1.055

T<sub>21.</sub> = 0.593  
 T<sub>22.</sub> = 0.845  
 T<sub>23.</sub> = 1.077

T<sub>31.</sub> = 0.298  
 T<sub>32.</sub> = 0.092  
 T<sub>33.</sub> = 0.674

T... =  
 THY<sub>ijk</sub> =

TABLE G-45  
ANOVA  
Tube F  
Schweyer R<sup>2</sup>

- (0)  $CT = (T \dots)^2 \div n = (6.839 \ 540 \ 000 \ E+00 \ )^2 \div (18 \ ) = 2.598 \ 850 \ 412 \ E+0$
- (1)  $SS \ (Temp.) = \frac{1}{(6)} [(3.260)^2 + \dots + (1.064)^2] - CT = 4.161 \ 415 \ 880 \ E-$
- (2)  $SS \ (Time) = \frac{1}{(6)} [(2.298)^2 + \dots + (2.806)^2] - CT = 2.526 \ 042 \ 100 \ E-0$
- (3)  $SS \ (H \times T) = \frac{1}{(2)} [(1.407)^2 + \dots + (0.674)^2] - CT - SS \ (T) -$   
 $SS \ (H) = 1.430 \ 600 \ 790 \ E-01$
- (4)  $SS \ (Total) = \sum \sum X_{ijk}^2 - CT = 2.410 \ 476 \ 909 \ E+00$
- (5)  $SS \ (Error) = SS \ (Total) - SS \ (T) - SS \ (H) - SS \ (T \times H)$   
 $= (4) - (1) - (2) - (3) = 1.756 \ 006 \ 821 \ E+00$

| ANALYSIS OF VARIANCE   |    |        |        |      |      |      |
|------------------------|----|--------|--------|------|------|------|
| Source                 | df | SS     | MS     | F    | F.05 | F.01 |
| (1) T <sub>i</sub>     | 2  | 0.4161 | 0.2081 | 1.07 | 4.26 | 8.02 |
| (2) H <sub>j</sub>     | 2  | 0.0953 | 0.0476 | 0.24 | 4.26 | 8.02 |
| (3) (TH) <sub>ij</sub> | 4  | 0.1431 | 0.0358 | 0.18 | 3.63 | 6.42 |
| (5) Error              | 9  | 1.7560 | 0.1951 |      |      |      |
| (4) Total              | 17 | 2.4105 |        |      |      |      |

TABLE G-46

TUBE G  
Schweyer R<sup>2</sup>

|     | Time Temperature |       |       |
|-----|------------------|-------|-------|
|     | 350              | 375   | 400   |
| 0.5 | 1.000            | 0.961 | 0.567 |
| 1.0 | 0.907            | 0.971 | 0.926 |
| 1.0 | 0.644            | 0.959 | 0.863 |
| 2.0 | 0.973            | 0.795 | 0.646 |
| 2.0 | 0.863            | 0.875 | 0.671 |
|     | 0.944            | 0.972 | 0.986 |

TABLE G-47  
Tube G  
Schweyer R

FOSTER & BURR q:

Matrix  $\Sigma x_i$  15.523  
 Matrix  $\Sigma x_i^2$  1.371 198 300 E+01  
 $v$  1.000  
 $q$  0.218  $q_{0.99}$  0.576  $q_{0.999}$  0.750

$$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \varepsilon(ij)k$$

TOTALS

Temperature  $(T_i)$

$T_{1..} = 5.331$   
 $T_{2..} = 5.533$   
 $T_{3..} = 4.659$   
 $T_{...} = 15.523$

Time  $(H_j)$

$T_{.1.} = 5.332$   
 $T_{.2.} = 4.880$   
 $T_{.3.} = 5.311$   
 $T_{...} = 15.523$

Temperature x Time  $(TH)_{ij}$

$T_{11.} = 1.907$   
 $T_{12.} = 1.617$   
 $T_{13.} = 1.807$

$T_{21.} = 1.932$   
 $T_{22.} = 1.754$   
 $T_{23.} = 1.847$

$T_{31.} = 1.493$   
 $T_{32.} = 1.509$   
 $T_{33.} = 1.657$

$T_{...} =$   
 $THY_{ijk} =$

TABLE G-48

ANOVA  
Tube G  
Schweyer R

(0)  $CT = (T_{...})^2 \div n = (15.523)^2 \div (18) = 1.338\ 686\ 272\ E+01$   
 (1)  $SS\ (Temp.) = \frac{1}{(6)} [(5.331)^2 + \dots + (4.659)^2] - CT = 6.979\ 245\ 000\ E-01$   
 (2)  $SS\ (Time) = \frac{1}{(6)} [(5.332)^2 + \dots + (5.311)^2] - CT = 2.169\ 478\ 000\ E-02$   
 (3)  $SS\ (H \times T) = \frac{1}{(2)} [(1.907)^2 + \dots + (1.657)^2] - CT - SS\ (T) - SS\ (H)$   
 $= 1.610\ 755\ 000\ E-02$

(4)  $SS\ (Total) = \Sigma \Sigma x_{ijk}^2 - CT = 3.251\ 202\ 800\ E-01$

(5)  $SS\ (Error) = SS\ (Total) - SS\ (T) - SS\ (H) - SS\ (T \times H)$   
 $= (4) - (1) - (2) - (3) = 2.175\ 255\ 000\ E-01$

| Source          | df | ANALYSIS OF VARIANCE |        |      |      |
|-----------------|----|----------------------|--------|------|------|
|                 |    | SS                   | MS     | F    | F.05 |
| (1) $T_i$       | 2  | 0.0698               | 0.0349 | 1.44 | 4.26 |
| (2) $H_j$       | 2  | 0.0217               | 0.0108 | 0.45 | 4.26 |
| (3) $(TH)_{ij}$ | 4  | 0.0161               | 0.0040 | 0.17 | 3.63 |
| (5) Error       | 9  | 0.2175               | 0.0242 |      |      |
| (4) Total       | 17 | 0.3251               |        |      |      |

TABLE G-49

TUBE E  
Schweyer

Log<sub>10</sub> (n<sub>0.05</sub> ÷ 1.00 E+07)

| Time        | 350    | 375    | 400    |
|-------------|--------|--------|--------|
| Temperature |        |        |        |
| 0.5         | 0.5876 | 0.5039 | 0.6734 |
|             | 1.0358 | 1.1383 | 1.2507 |
| 1.0         | 1.3829 | 1.7594 | 1.2690 |
|             | 1.9345 | 1.2333 | 1.6428 |
| 2.0         | 1.1981 | 1.0191 | 2.1319 |
|             | 1.4476 | 0.8244 | 0.8528 |

TABLE G-50

Tube E  
Schweyer

Log<sub>10</sub> (n<sub>0.05</sub> ÷ 1.00 E+07)

FOSTER & BURR q:

Matrix Ix<sub>1</sub> 20.8777

Matrix Ix<sub>2</sub> 3.008 734 329 E+01

v 1.000

q 0.318

q<sub>0.99</sub> 0.576

q<sub>0.999</sub> 0.750

$$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon(ijk)$$

TOTALS

Temperature (T<sub>i</sub>)

T<sub>1..</sub> = 7.5865  
 T<sub>2..</sub> = 5.4706  
 T<sub>3..</sub> = 7.8206  
 20.8777

Time (H<sub>j</sub>)

T<sub>.1.</sub> = 4.1819  
 T<sub>.2.</sub> = 9.2219  
 T<sub>.3.</sub> = 7.4739  
 20.8777

Temperature x Time (TH)<sub>ij</sub>

T<sub>11.</sub> = 1.6234  
 T<sub>12.</sub> = 3.3174  
 T<sub>13.</sub> = 2.6457

T<sub>21.</sub> = 0.6344  
 T<sub>22.</sub> = 2.9927  
 T<sub>23.</sub> = 1.8435

T<sub>31.</sub> = 1.9241  
 T<sub>32.</sub> = 2.9118  
 T<sub>33.</sub> = 2.9847

T... =

THY<sub>ijk</sub> =

TABLE G-50

ANOVA  
Tube E  
Schweyer

$$\text{Log}_{10} (\eta_{0.05} \div 1.00 \text{ E} + 07)$$

$$(0) \text{ CT} = (\text{T} \dots)^2 \div n = (20.8777)^2 \div (18) = 2.421546429 \text{ E}+01$$

$$(1) \text{ SS (Temp.)} = \frac{1}{(6)} [(7.5865)^2 + \dots + (7.8206)^2] - \text{CT} = 5.585742100 \text{ E}-0$$

$$(2) \text{ SS (Time)} = \frac{1}{(6)} [(4.1819)^2 + \dots + (7.4739)^2] - \text{CT} = 2.183020440 \text{ E}+0$$

$$(3) \text{ SS (H x T)} = \frac{1}{(2)} [(1.6234)^2 + \dots + (2.9847)^2] - \text{CT} - \text{SS (T)} - \text{SS (H)}$$

$$= 2.862819900 \text{ E}-01$$

$$(4) \text{ SS (Total)} = \sum \sum x_{ijk}^2 - \text{CT} = 5.871879000 \text{ E}+00$$

$$(5) \text{ SS (Error)} = \text{SS (Total)} - \text{SS (T)} - \text{SS (H)} - \text{SS (T x H)}$$

$$= (4) - (1) - (2) - (3) = 2.844002360 \text{ E}+00$$

TABLE G-51

TUBE F  
Schweyer  
 $\text{Log}_{10} (\eta_{0.05} \div 1.00 \text{ E}+07)$

Time  
Temperature

|     |        |        |        |
|-----|--------|--------|--------|
|     | 350    | 375    | 400    |
| 0.5 | 0.6001 | 1.6015 | 1.9609 |
|     | 0.7833 | 1.9734 | 0.9690 |
| 1.0 | 1.9101 | 2.0934 | 1.7536 |
|     | 1.8986 | 0.8112 | 1.8245 |
| 2.0 | 2.3629 | 1.1361 | 1.1988 |
|     | 0.8088 | 0.6323 | 1.2653 |

| ANALYSIS OF VARIANCE   |    |        |        |      |      |      |
|------------------------|----|--------|--------|------|------|------|
| Source                 | df | SS     | MS     | F    | F.05 | F.01 |
| (1) T <sub>i</sub>     | 2  | 0.5586 | 0.2793 | 0.88 | 4.26 | 8.02 |
| (2) H <sub>j</sub>     | 2  | 2.1830 | 1.0915 | 3.45 | 4.26 | 8.02 |
| (3) (TH) <sub>ij</sub> | 4  | 0.2863 | 0.0716 | 0.23 | 3.63 | 6.42 |
| (5) Error              | 9  | 2.8440 | 0.3160 |      |      |      |
| (4) Total              | 17 | 5.8719 |        |      |      |      |

TABLE G-52  
Tube F  
Schweyer

FOSTER & BURR g<sub>i</sub>      Log<sub>10</sub> (η<sub>0.05</sub> ± 1.00 E + 07)

|                                     |                    |
|-------------------------------------|--------------------|
| Matrix Lx <sub>i</sub>              | 25.5838            |
| Matrix Lx <sub>i</sub> <sup>2</sup> | 4.188 041 058 E+01 |
| v                                   | 1.000              |
| g                                   | 0.320              |
| g                                   | 0.576              |
| g                                   | 0.999              |
| g                                   | 0.750              |

Y<sub>ijk</sub> = μ + T<sub>i</sub> + H<sub>j</sub> + (TH)<sub>ij</sub> + ε<sub>(ij)k</sub>

TOTALS

Temperature (T<sub>i</sub>)

|                  |           |
|------------------|-----------|
| T <sub>1..</sub> | = 8.3638  |
| T <sub>2..</sub> | = 8.2479  |
| T <sub>3..</sub> | = 8.9721  |
| T <sub>...</sub> | = 25.5838 |

Time (H<sub>j</sub>)

|                  |           |
|------------------|-----------|
| T <sub>.1.</sub> | = 7.8882  |
| T <sub>.2.</sub> | = 10.2914 |
| T <sub>.3.</sub> | = 7.4042  |
| T <sub>...</sub> | = 25.5838 |

Temperature x Time (TH)<sub>ij</sub>

|                  |          |
|------------------|----------|
| T <sub>11.</sub> | = 1.3834 |
| T <sub>12.</sub> | = 3.8087 |
| T <sub>13.</sub> | = 3.1717 |
| T <sub>21.</sub> | = 3.5749 |
| T <sub>22.</sub> | = 2.9046 |
| T <sub>23.</sub> | = 1.7684 |
| T <sub>31.</sub> | = 2.9299 |
| T <sub>32.</sub> | = 3.5781 |
| T <sub>33.</sub> | = 2.4641 |

T<sub>...</sub>  
T<sub>...</sub>

TABLE G-53  
ANOVA  
Tube F  
Schweyer

Log<sub>10</sub> (η<sub>0.05</sub> ± 1.00 E + 07)

- (0) CT = (T<sub>...</sub>)<sup>2</sup> ÷ n = (25.5838)<sup>2</sup> ÷ ( 18 ) = 3.638 282 347 E+01
- (1) SS (Temp.) =  $\frac{1}{(6)}$  [(8.3638)<sup>2</sup> + ... + (8.9721)<sup>2</sup>] - CT = 5.044 041 000 E-02
- (2) SS (Time) =  $\frac{1}{(6)}$  [(7.8882)<sup>2</sup> + ... + (7.4042)<sup>2</sup>] - CT = 7.969 750 000 E-01
- (3) SS (H x T) =  $\frac{1}{(2)}$  [(1.3834)<sup>2</sup> + ... + (2.4641)<sup>2</sup>] - CT - SS (T) - SS (H) = 1.930 973 270 E+00

(4) SS (Total) = ΣΣx<sub>ijk</sub><sup>2</sup> - CT = 5.517 587 110 E+00

(5) SS (Error) = SS (Total) - SS (T) - SS (H) - SS (T x H)

= (4) - (1) - (2) - (3) = 2.739 198 430 E+00

| ANALYSIS OF VARIANCE   |    |        |        |      |      |      |
|------------------------|----|--------|--------|------|------|------|
| Source                 | df | SS     | MS     | F    | F.05 | F.01 |
| (1) T <sub>i</sub>     | 2  | 0.0504 | 0.0252 | 0.08 | 4.26 | 8.02 |
| (2) H <sub>j</sub>     | 2  | 0.7970 | 0.3985 | 1.31 | 4.26 | 8.02 |
| (3) (TH) <sub>ij</sub> | 4  | 1.9310 | 0.4827 | 1.59 | 3.63 | 6.42 |
| (5) Error              | 9  | 2.7392 | 0.3044 |      |      |      |
| (4) Total              | 17 | 5.5176 |        |      |      |      |

TABLE G-54

TUBE G

Schweyer

$\text{Log}_{10} (n0.05 + 1.00 E+07)$

| Time |        | Temperature |         |     |
|------|--------|-------------|---------|-----|
|      |        | 350         | 375     | 400 |
| 0.5  | 0.0931 | 1.2613      | 1.0026  |     |
|      | 0.5231 | -0.0730     | 0.0983  |     |
| 1.0  | 0.6252 | 0.7315      | 1.0515  |     |
|      | 0.0588 | 0.6880      | 1.1664  |     |
| 2.0  | 0.4390 | 0.4002      | 0.6021  |     |
|      | 0.0382 | -0.0784     | -0.0517 |     |

TABLE G-55

Tube G

Schweyer

$\text{Log}_{10} (n_{0.05} \div 1.00 E + 07)$

FOSTER & BURR  $q_1$

Matrix  $Lx_i$  8.5762

Matrix  $Lx_i^2$  7.487 971 640 E+00

$v$  1.000

$q$  0.273

$q_{0.99}$  0.576

$q_{0.999}$  0.750

$Y_{ijk} = \mu + T_i + H_j + (TH)_{ij} + \epsilon(ijk)$

TOTALS

Temperature ( $T_i$ )

$T_{1..} = 1.7774$

$T_{2..} = 2.9296$

$T_{3..} = 3.9692$

$T_{...} = 8.5762$

Time ( $H_j$ )

$T_{.1.} = 2.9054$

$T_{.2.} = 4.3214$

$T_{.3.} = 1.3494$

$T_{.3.} = 8.5782$

Temperature x Time ( $TH$ )  $ij$

$T_{11.} = 0.6162$

$T_{12.} = 0.6840$

$T_{13.} = 0.4772$

$T_{21.} = 1.1883$

$T_{22.} = 1.4195$

$T_{23.} = 0.3218$

$T_{31.} = 1.1009$

$T_{32.} = 2.2179$

$T_{33.} = 0.5504$

$T_{...} =$

$\epsilon\epsilon\epsilon_{ijk} =$

TABLE G-56

ANOVA

Schweyer

$$\log_{10} (n_{0.05} \pm 1.00 E + 07)$$

- (0)  $CT = (T \dots)^2 \div n = (8.5762)^2 \div ( 18 ) = 4.086 178 136 E+00$
- (1)  $SS (Temp.) = (\frac{1}{6}) [(1.7774)^2 + \dots + (3.8692)^2] - CT = 3.658 911240 E-0$
- (2)  $SS (Time) = (\frac{1}{6}) [(2.9054)^2 + \dots + (1.3494)^2] - CT = 7.366 097 770 E-0$
- (3)  $SS (H \times T) = (\frac{1}{2}) [(0.6162)^2 + \dots + (0.5504)^2] - CT - SS (T) - SS (H)$   
 $= 3.312 569 830 E-01$
- (4)  $SS (Total) = \sum \sum x_{ijk}^2 - CT = 3.401 793 504 E+00$
- (5)  $SS (Error) = SS (Total) - SS (T) - SS (H) - SS (T \times H)$   
 $= (4) - (1) - (2) - (3) = 1.968 035 620 E+00$

| ANALYSIS OF VARIANCE |    |        |        |      |      |      |
|----------------------|----|--------|--------|------|------|------|
| Source               | df | SS     | MS     | F    | F.05 | F.01 |
| T                    | 2  | 0.3659 | 0.1829 | 0.48 | 4.26 | 8.02 |
| H                    | 2  | 0.7366 | 0.3683 | 0.97 | 4.26 | 8.02 |
| (TH) ij              | 4  | 0.3313 | 0.0828 | 0.22 | 3.63 | 6.42 |
| Error                | 9  | 3.4018 | 0.3780 |      |      |      |
| Total                | 17 | 1.9680 |        |      |      |      |