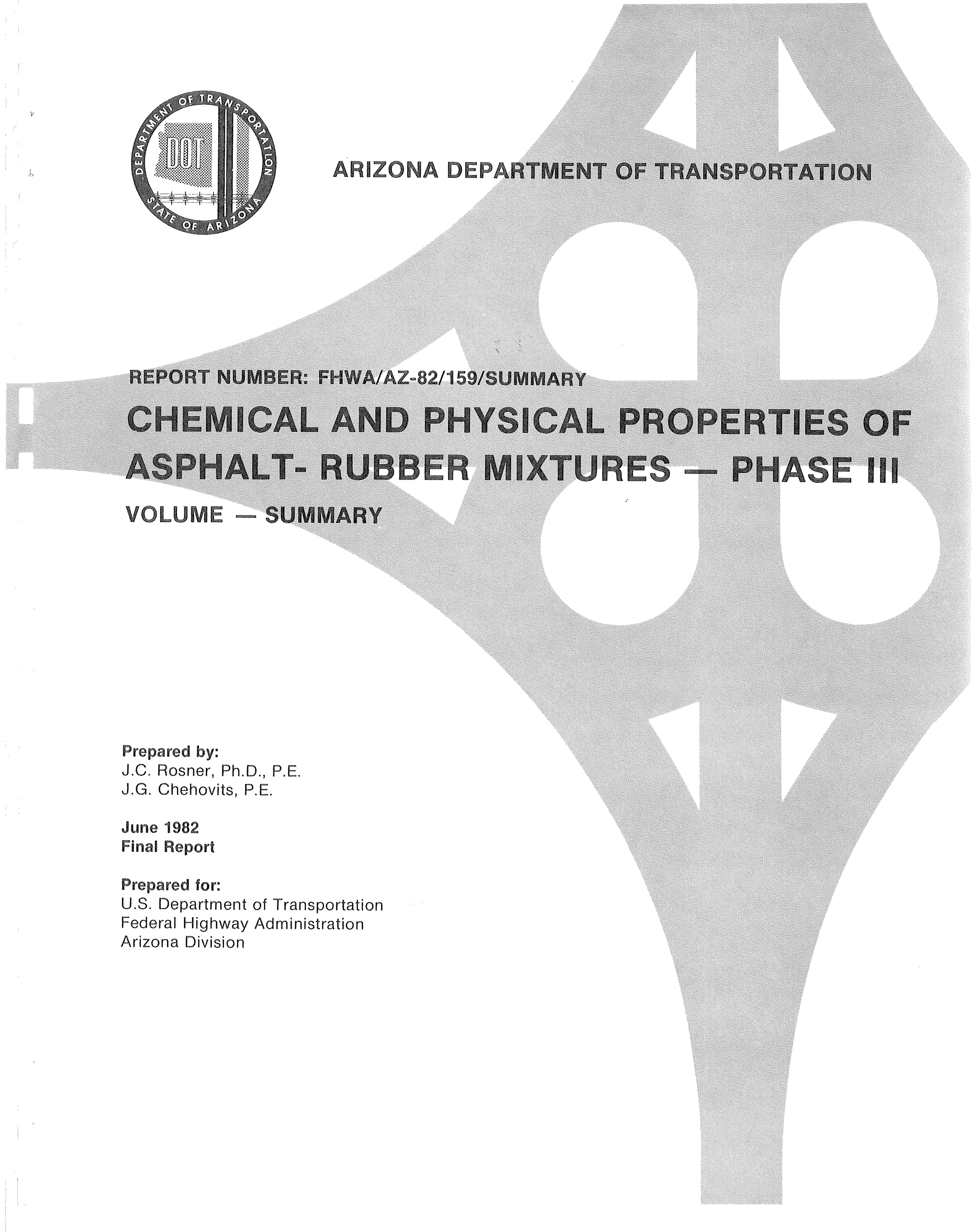

ARIZONA DEPARTMENT OF TRANSPORTATION

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CHEMICAL AND PHYSICAL PROPERTIES OF ASPHALT- RUBBER MIXTURES — PHASE III

VOLUME — SUMMARY

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16. Abstract

An extensive statistically designed laboratory investigation which had objectives of evaluating (1) effects of asphalt-rubber components on mixture properties, (2) effects of diluent on mixture properties, (3) effects of temperature on mixture properties, (4) applicability of several test procedures for asphalt-rubber mixtures, and (5) differences between field and lab produced asphalt-rubber mixtures was performed. Asphalt-rubber materials formulated with six rubber types from two different production processes, four rubber concentrations, four asphalts and four diluent concentrations were produced and tested. Field produced mixtures were obtained from experimental asphalt-rubber field projects in Arizona. Testing procedures included absolute viscosity, Schweyer rheometer, sliding plate microviscometer, force-ductility, and viscosity during mixing.

The study concluded that asphalt-rubber mixture properties vary depending on the rubber type, rubber concentration, asphalt type, diluent concentration, diluent cure time, and temperature. Temperature susceptibility of asphalt-rubber mixtures containing rubber from the two production processes studied varied. Mixtures produced in the laboratory were found to be stiffer than field produced mixtures. The force-ductility and sliding plate microviscometer tests were found to yield acceptable testing variability to permit their use in specifying asphalt-rubber physical properties.

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CHEMICAL AND PHYSICAL PROPERTIES
OF ASPHALT - RUBBER MIXTURES - PHASE III

SUMMARY REPORT

BY
J. C. ROSNER, J. G. CHEHOVITS

SUBMITTED TO

THE ARIZONA DEPARTMENT OF TRANSPORTATION
HIGHWAY DIVISION
PHOENIX, ARIZONA

FOR

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INDEX

1.0 PROJECT DESCRIPTION

1.1 The objectives of this project were to:

- Evaluate the effects of the components of asphalt-rubber mixtures on physical properties of the mixtures,
- Evaluate the effects of temperature on physical properties of asphalt-rubber mixtures,
- Determine physical properties of field-produced asphalt-rubber mixtures,
- Compare properties of field and lab produced asphalt-rubber mixtures, and
- Evaluate the feasibility of using testing procedures employed in this investigation for asphalt-rubber specification purposes.

1.2 The above objectives were accomplished by performing and analyzing results of several series of statistically designed laboratory experiments utilizing different asphalt-rubber mixtures.

1.3 The experiments performed considered asphalt-rubber mixtures formulated with:

- Six different types of rubber from two different production processes,
- Four different rubber concentrations,
- Four different asphalts, and
- Four different concentrations of diluent and five curing times.

1.4 Field produced mixtures which were characterized in the lab were obtained from an asphalt-rubber experimental project constructed in Arizona from October to December of 1978.

1.5 Material properties assessed in this study were:

- Absolute viscosity at 140F (60C)

- Apparent viscosity and shear rate sensitivity by the Schweyer rheometer at -20, 39.2, and 77F
- Stress, strain, and creep compliance properties at -20, 39.2, and 77F using force-ductility.
- Apparent viscosity, creep, strain recovery, rebound and rheological characteristics at 32F (0C) using the sliding plate microviscometer
- Ring-and-Ball Softening Point
- Fracture Temperature
- Viscosity during mixing at 375F (191C) using the Arizona Torque-Fork
- Viscosity during mixing at 375F (191C) using the Haake Rotational Viscometer.

1.6 Analytical techniques used included conventional one, two, three, and four-way analyses of variance, a means ranking procedure, and graphical plots.

1.7 Details of the experiments, results, and conclusions are reported in five separate volumes of the report, "Chemical and Physical Properties of Asphalt-Rubber Mixtures - Phase III" as follows:

Volume I	Effects of Rubber Type, Concentration, and Asphalt
Volume II	Effects of Asphalt
Volume III	Effects of Diluent
Volume IV	Physical Properties of Field-Mixed Asphalt-Rubber Mixtures and Comparison of Lab and Field-Mixed Asphalt-Rubbers
Volume V	Effects of Temperature

2.0 ASPHALT-RUBBER AS A PAVING MAINTENANCE MATERIAL

2.1 Introduction

Asphalt-rubber can be defined as a mixture of asphalt cement, granulated reclaimed tire rubber, and possibly certain additives. Asphalt-rubber is distinguished from a material generally termed rubberized asphalt in that asphalt-rubber contains a higher amount of rubber (15 to 25% by weight) than rubberized asphalt (1 to 3% by weight).

2.1.1 In recent years, asphalt-rubber materials have proven useful in pavement maintenance in several functional applications including:

- Asphalt concrete binders
- Surface treatments
- Stress absorbing membranes for reduction of reflective cracking
- Waterproofing membranes
- Crack and joint sealants

2.1.2 Presently, the major emphasis for usage of asphalt-rubber materials is in pavement maintenance applications - specifically, for use in reducing reflective cracking. Use of asphalt-rubber as a crack and joint sealer is becoming increasingly more popular.

2.1.3 The asphalt-rubber concept was developed by Charles H. McDonald while he was Engineering Supervisor for the City of Phoenix, Arizona in the early 1960's (1). The material was used mainly as a surface patching material for cracked pavement sections.

2.1.4 Since the mid 1960's, performance of asphalt-rubber materials has been studied in numerous field test projects. The major functional uses studied have been as stress absorbing membrane interlayers for prevention and control of reflective cracking and as seal coats.

2.2 Reaction Mechanism

2.2.1 When rubber and asphalt cement are mixed at high temperatures (250-400F), the rubber particles swell to several times their original volume (2). Swelling is postulated to occur due to physical and chemical interactions between rubber particles and asphalt. Swelling results in an increase in viscosity of the asphalt-rubber mixture, and is commonly referred to as the reaction between asphalt and rubber.

2.2.2 The reacted asphalt-rubber material has radically differing properties from the original asphalt cement and also from the unreacted asphalt-rubber material. Properly reacted asphalt-rubber materials have sufficient stress and strain tolerance such that the materials can bridge underlying pavement cracks and withstand deformations without fracturing. Additionally, properly reacted asphalt-rubber is extremely sticky having excellent adhesion to aggregate and pavement surfaces which is very important in paving applications.

2.3 Component Materials

Mixture properties of asphalt-rubber are influenced by the properties and characteristics, both physical and chemical, of component materials.

2.3.1 The specific asphalt cement used in asphalt-rubber will not only influence the viscosity of the mixture due to asphalt grade and consistency, but, its chemical compositional characteristics may influence the degree and rate of swelling of rubber particles thus influencing properties of the reacted asphalt-rubber material.

2.3.2 Rubber characteristics including particle size and shape, surface texture, rubber source and composition, and rubber concentration in the mixture have been noted to influence the properties of asphalt-rubber mixtures (3, 4). Particle size, shape and surface texture, and rubber source and composition can influence the degree and rate of swelling or reaction which occurs between rubber and asphalt. Rubber concentration in the mixture can influence the mass properties of reacted asphalt-rubber mixtures (3).

2.3.3 Diluents are sometimes added to commercial asphalt-rubber mixtures to reduce viscosity of the reacted product so that it can be sprayed through a distributer truck. It has been noted that diluents tend to inhibit rubber particle swelling (1).

2.4 Uses of Asphalt-Rubber

Major uses of asphalt-rubber in paving have been in maintenance related operations - chip seals for stress absorbing membranes and hot-poured joint sealers.

2.4.1 Chip seals using asphalt-rubber as the binder have been found to perform well as low-modulus stress-absorbing-membranes (SAM) or stress-absorbing-membrane-interlayers (SAMI) for aiding in reduction of reflective cracking. Numerous field test projects using asphalt-rubber in this application have been constructed and are well documented (5, 6, 7, 8, 9). Performance records indicate that asphalt-rubber chip seals used in either a SAM or SAMI functional application can aid in reduction of reflection of fatigue cracks but will not prevent reflection of thermally-induced transverse cracks (10).

2.4.2 Effective joint and crack sealers for asphalt and concrete pavements have been made from asphalt-rubber materials (11). Asphalt-rubber crack sealers have the ability to withstand differential crack movement, bond strongly to crack faces, and reheal or rebond to joint faces in warm weather if fracture or separation from crack faces occurs.

2.5 Recent Reseach Developments

Considerable amounts of research work related to performance of asphalt-rubber mixtures in varying applications has been performed in recent years.

2.5.1 Laboratory research related to asphalt-rubber mixtures has primarily been concerned with studying asphalt-rubber reactions, applicability of standard asphalt testing procedures, and unique characteristics of asphalt-rubber materials alone and in combination with aggregates.

2.5.1.1 The basic materials properties of asphalt-rubber were studied and reported by Green and Tolonen (1). Properties studied included rubber swelling characteristics in asphalt, viscosity, and strain recovery characteristics. Testing procedures for asphalt-rubber materials are recommended.

2.5.1.2 Oliver (3) studied the elastic behavior of asphalt-rubber mixtures using a sliding plate rheometer. It was noted that digestion (reaction) time and temperature can influence the elastic recovery of asphalt-rubber mixtures and that effects were different with different rubber types. It was also concluded that rubber concentration also influences elastic recovery, but particle size does not. Additionally, an asphalt-rubber produced in the laboratory was found to have similar elastic properties to field produced asphalt-rubber composed of the same materials and reacted at the same temperature for the same time period.

2.5.1.3 Frobel, Jimenez, and Cluff (4) studied properties of asphalt-rubber mixtures as related to use as a waterproof membrane. Conclusions reached during this study include:

- Permeability of asphalt-rubber materials is so low that they can be considered to be impermeable.
- Asphalt-rubber mixtures have decreased ductility when compared to that of the base asphalt and that finer rubber particles result in higher ductilities than larger particles.
- Toughness (resistance to deformation) of an asphalt-rubber mixture increases as the rubber particle size increases.
- Viscosity of asphalt is greatly increased by the addition of crumb rubber particles.

2.5.1.4 Pavlovich, Shuler and Rosner (12) reported on a study which investigated the effects of reaction time and temperature on a single asphalt-rubber mixture using several modified standard asphalt testing procedures and non-standard procedures.

The study concluded that:

- Modified standard tests studied (softening point, absolute viscosity, and ductility) are not applicable for testing asphalt-rubber without modification of testing method and precision statement.
- Force-ductility testing can detect differences in reaction time and temperature used in preparation of asphalt-rubber mixtures.
- The Schweyer Rheometer can be used to determine low temperature viscosity of asphalt-rubber materials.
- More research work related to testing procedures and asphalt-rubber reactions is required before test methods for specification purposes can be developed.

3.0 MATERIALS

3.1 Rubber. Six different types of granulated rubber were used in the experiments performed in this study. Rubber from two different production processes, ambient grind tread peel crumb and ambient grind high natural rubber content devulcanized crumb, were studied, each at three differing particle size distributions (gradations).

3.1.1 The ambient grind tread peel crumb rubber was produced by Atlos Rubber Reclaiming of Los Angeles, California.

3.1.1.1 The material consisted of ground (crumb) rubber produced by mechanically grinding passenger car tread peel at ambient temperatures. No additional processing or depolymerization was used on the ground rubber product to alter material properties prior to mixing with asphalt. The addition of a maximum of 4 percent calcium carbonate powder prior to packaging is permitted to prevent the rubber particles from sticking together.

3.1.1.2 Two Atlos rubber products which differed in gradation were studied. These products are designated by Atlos as TP044 and TP027. TP044 is composed mostly of rubber particles sized between the #8 (0.0937 in.) and #30 (0.0234 in.) mesh screens, while TP027 is mostly between the #30 (0.0234 in.) and #100 (0.0059 in.) mesh screens. Additionally, in this study, a 50/50 mixture by weight of TP044 and TP027 was studied to investigate effects of a more well-graded ambient grind tread peel crumb rubber product on asphalt rubber mixture properties.

3.1.1.3 Gradations of TP044, TP027, and the 50/50 mixture of TP044 and TP027 are tabulated in Table 1 and plotted in Figure 1.

3.1.2 Devulcanized ambient grind crumb rubber with a high natural rubber content produced by U.S. Rubber Reclaiming of Vicksburg, Mississippi was the second source of rubber studied in this project.

TABLE 1

GRADATIONS OF ATLOS TP044, TP027, AND A 50/50 MIXTURE (by weight) OF TP044 AND TP027

Sieve Size	% Passing		
	TP044	TP027	50% TP044 50% TP027
#8	100	100	100
#16	83	100	91
#30	7	94	50
#50	1	19	10
#100	0.5	6.5	3.5
#200	0.0	0.0	0.0

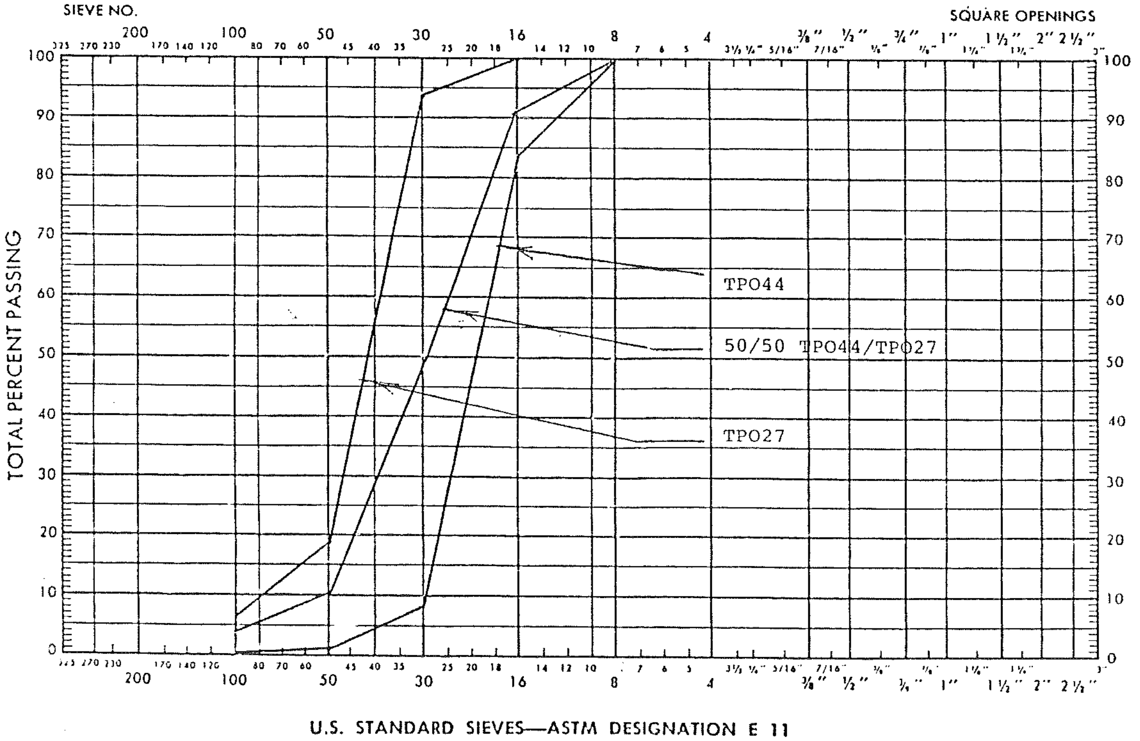


Figure 1 Gradations of Atlos TP044, TP027, and a 50/50 Mixture (by weight of TP044 and TP027

3.1.2.1 The material consists of a blend of 40 percent powdered reclaimed (devulcanized) rubber and 60 percent ambient ground vulcanized rubber which contains a high natural rubber content. The rubber is free from fabric, wire, or other contaminants except that up to 4 percent calcium carbonate may be added to prevent caking.

3.1.2.2 Two U.S. Rubber Reclaiming products which differed in gradation were studied. The first is designated as GT274 which is composed mostly of particles sized between the #16 (0.0469 in.) and #100 (0.0059 in.) mesh sieves. The second is a finer material obtained from grinding buffings produced during production of GT274. This material is designated as U.S. Rubber Fine (USRF) and is sized mostly between the #30 (0.0234 in.) and #100 (0.0059 in.) mesh sieves.

Additionally, a 50/50 mixture by weight of GT274 and USRF was studied to investigate effects of a more well-graded high natural rubber content devulcanized rubber product on asphalt-rubber mixture properties.

3.1.3 Each of the six rubber types described above (two production processes each at three gradations) was incorporated into asphalt-rubber mixtures at 15, 20, 25, and 30 percent by weight of total asphalt-rubber mixture.

3.1.4 Scanning election photomicrographs of TP044 and GT274 rubber particles are shown in Figure 3 at magnifications of 70 and 250 times. The smaller particle size of GT274 is noted and it can be seen that GT274 appears to have a rougher surface texture than TP044.

3.2 Asphalt Cement. Four different asphalt cements were used in asphalt-rubber mixtures for this study. Three of the asphalt cements used were a combination of an AR4000 and 3 different percentages of an extender oil. The fourth asphalt used was an AR1000.

TABLE 2

GRADATIONS OF U.S. RUBBER RECLAIMING GT274, U.S. RUBBER FINES, AND A 50/50 MIXTURE (by weight) OF GT274 AND U.S. RUBBER FINE

Sieve Size	% Passing		
	GT274	USRF	50% GT274 50% USRF
#8	100	100	100
#16	98	100	99
#30	76	95	86
#50	23	33	28
#100	8.0	12.2	10.1
#200	0.0	0.0	0.0

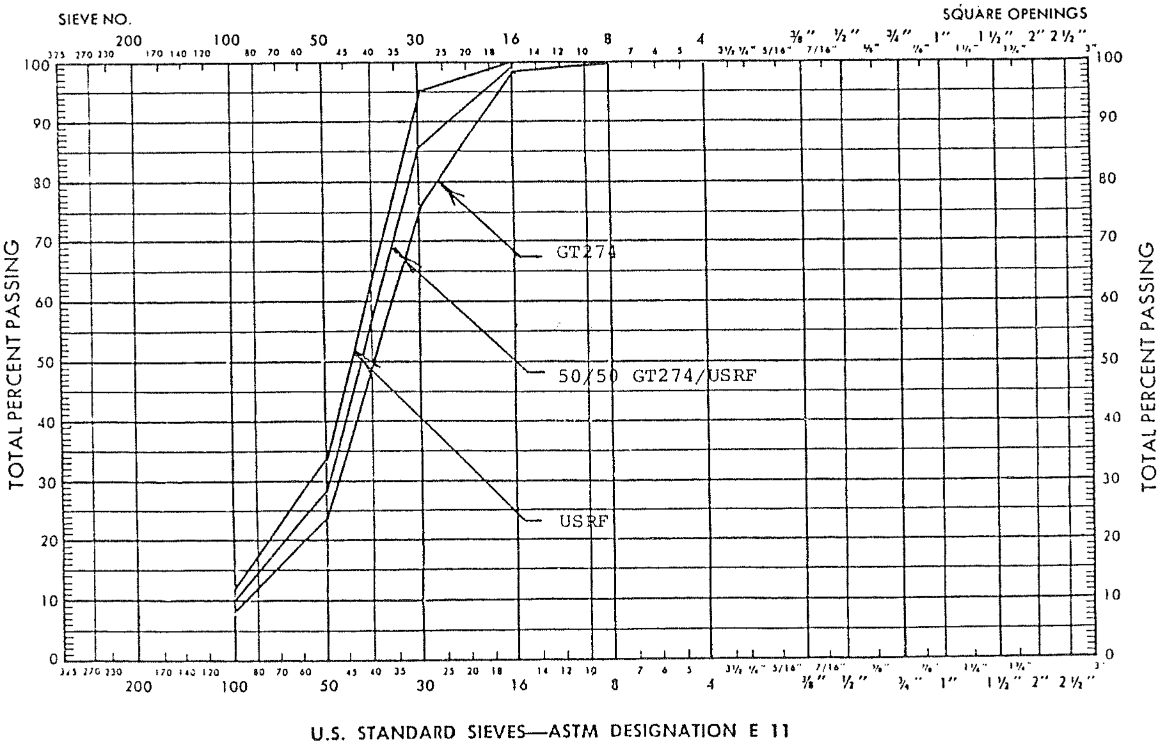
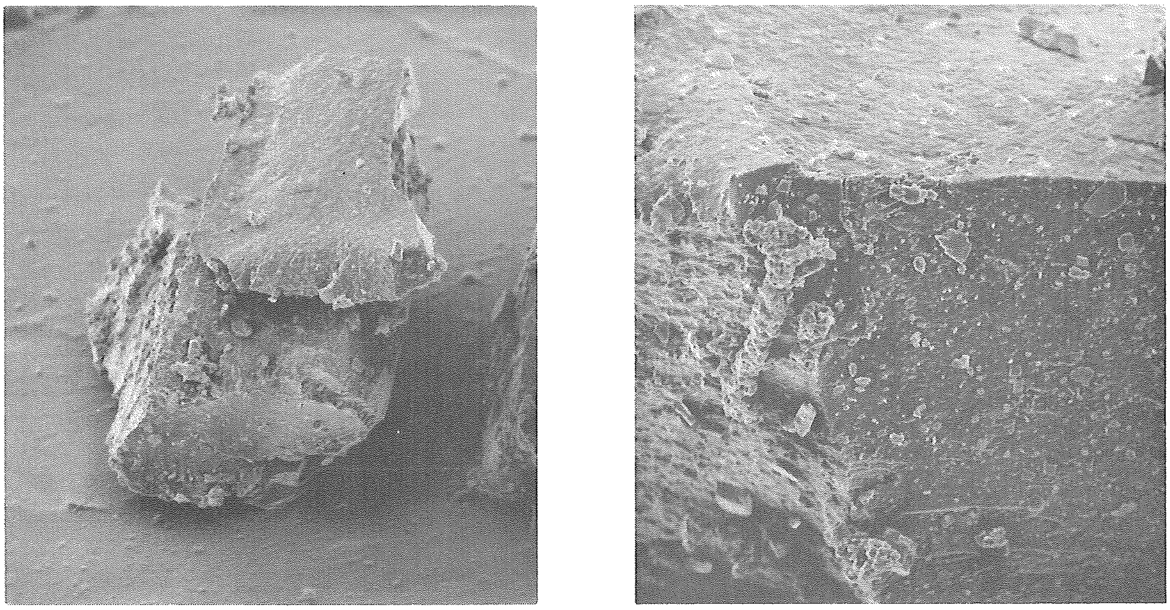
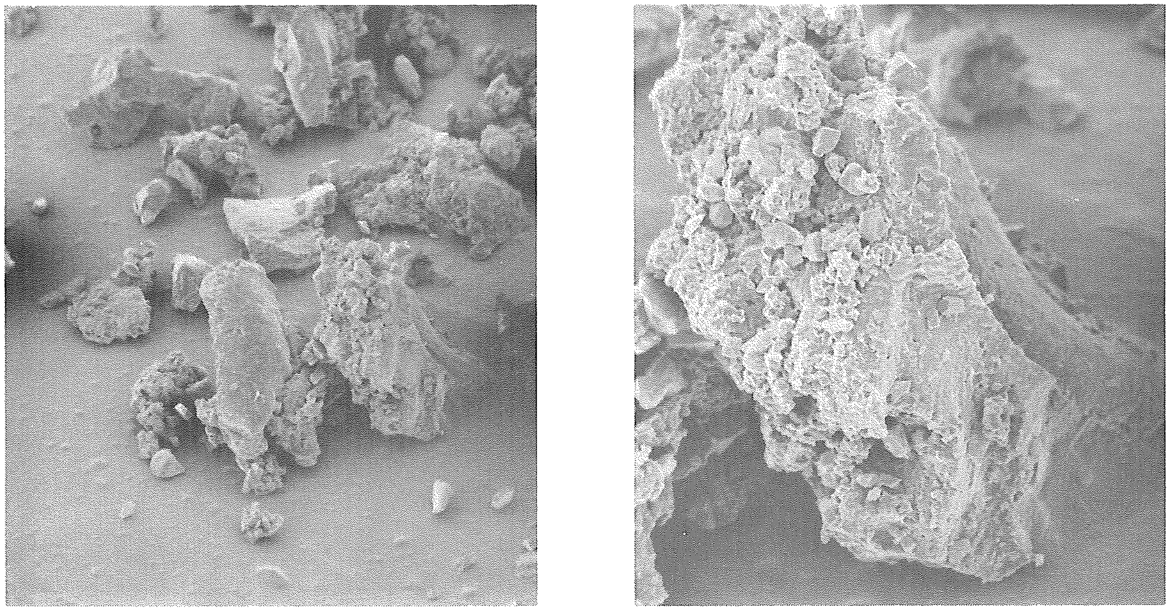


Figure 2 Gradations of U.S. Rubber Reclaiming GT274, U.S. Rubber Fines, and a 50/50 Mixture (by weight) of GT274 and U.S. Rubber Fine



TP044 70X

TP044 250X



GT274 70X

GT274 250X

Figure 3

3.2.1 The AR1000 used in this study was supplied by Sahuaro Petroleum and Asphalt Company of Phoenix, Arizona. This asphalt is commonly used in conjunction with approximately 25 percent Atlos TP044 rubber and diluents to produce commercial asphalt-rubber mixtures.

3.2.2 The AR4000 used in this study was supplied by Arizona Refining Company (ARCO) of Phoenix, Arizona. This asphalt is commonly used in conjunction with 20 percent U.S. Rubber Reclaiming GT274 rubber and 2 percent extender oil to produce commercial asphalt-rubber mixtures.

3.2.3 The extender oil used in this study was Califlux GP manufactured by the Golden Bear Division of Witco Chemical Corporation and supplied by Sahuaro Petroleum and Asphalt Company.

3.2.4 Penetration, softening point, absolute viscosity, kinematic viscosity, ductility, flash point and solubility tests were performed for both AR4000 and AR1000 asphalt cements. Test procedures used to characterize asphalts are tabulated in Table 3. Test results and ASTM D3381-76 (13) specification limits for viscosity-graded asphalt cements are tabulated in Tables 4 and 5.

3.2.4.1 The Sahuaro AR1000 meets ASTM D3381-76 requirements for an AR1000 asphalt cement.

3.2.4.2 The ARCO AR4000 meets ASTM D3381-76 requirements for an AR4000 asphalt cement.

3.2.5 Penetration, absolute viscosity, and kinematic viscosity tests were performed on the mixtures of unaged AR4000 with Califlux at 2, 4, 8, 15, and 25 percent by weight of asphalt. Test results, along with results for unaged AR4000 and AR1000 asphalts are tabulated in Table 6.

3.2.5.1 Based on test results in Table 5, two percentages of Califlux additions in the AR4000 were studied. A 15 percent addition was selected to produce an asphalt similar in characteristics to the AR1000, and a 6 percent addition was selected to produce a material with characteristics falling between the AR4000 with 2 percent Califlux and the AR4000 with 15 percent Califlux.

TABLE 3

TEST PROCEDURES USED TO CHARACTERIZE ASPHALT CEMENTS

Property	Testing Procedures
Penetration	ASTM D5-49, "Penetration of Bituminous Materials"
Softening Point	ASTM D36-76, "Softening Point of Bitumen (Ring-and-Ball Apparatus)"
Absolute Viscosity	ASTM D2171-78, "Viscosity of Asphalts by Vacuum Capillary Viscometer"
Kinematic Viscosity	ASTM D2170-76, "Kinematic Viscosity of Asphalts (Bitumens)"
Ductility	ASTM D113-79, "Ductility of Bituminous Materials"
Flash Point	ASTM D92-78, "Flash and Fire Points by Cleveland Open Cup"
Solubility	ASTM D2042-76, "Solubility of Asphalt Materials in Trichlorethylene"
Rolling Thin Film	ASTM D2872-80, "Effect of Heat and Air on a Moving Film of Asphalt (Rolling Thin Film Oven Test)"

TABLE 4

PHYSICAL PROPERTIES OF UNAGED SAHUARO AR1000, AGED RESIDUE AND ASTM D3381 SPECIFICATION LIMITS

Property	Test Results Unaged	 RTFOT[1] Aged Residue	Specification Limits
Penetration, 100g, 5 sec, 77F; 1/10 mm	127	86	65 min[2]
Softening Point; °C	41.0	46.0	--
Absolute Viscosity, 60C, 30 cmHg; Poise	662	1062	1000±250[2]
Kinematic Viscosity, 135C; cSt	178	233	140 min[2]
Ductility, 77F, 5cm/min; cm	-	100	100 min[2]
Flash Point, COC; °F	490	--	400 min
Solubility in Trichlorethylene; %	99.95	--	99.0 min
Rolling Thin Film Oven Loss; %		0.32	

Notes: [1] Rolling Thin Film Oven Test
[2] Tests on RTFOT Residue

TABLE 5

PHYSICAL PROPERTIES OF UNAGED ARCO AR4000, AGED RESIDUE, AND ASTM D3381 SPECIFICATION LIMITS

	Test Results		
Property	Unaged	RTFOT[1] Aged Residue	Specification Limits
Penetration, 100g, 5 sec, 77F; 1/10 mm	78	48	25 min[2]
Softening Point; °C	47.0	51.5	--
Absolute Viscosity, 60C, 30 cmHg; Poise	1612	3318	4000±1000[2]
Kinematic Viscosity, 135C; cSt	248	348	275 min[2]
Ductility, 77F, 5cm/min; cm	--	7100	75 min[2]
Flash Point, COC; °F	555	--	440 min
Solubility in Trichlorethylene; %	99.97	--	99.0 min
% Original Penetration, 77F	61		45 min
Rolling Thin Film Oven Loss; %	0.09		

Notes: [1] Rolling Thin Film Oven Test
[2] Tests on RTFOT Residue

TABLE 6

PENETRATION, ABSOLUTE VISCOSITY, AND KINEMATIC VISCOSITY OF AR1000, AR4000, AND AR4000 WITH 2, 4, 8, 15, AND 25 PERCENT CALIFLUX

Property	AR1000	AR4000 + 25% Califlux	AR4000 + 15% Califlux	AR4000 + 8% Califlux	AR4000 + 4% Califlux	AR4000 + 2% Califlux	AR4000
Penetration, 100g, 5 sec, 77F; 1/10 mm	127	245	154	102	79	58	78
Absolute Viscosity, 30 cm Hg, 140F; Poise	662	268	495	838	1166	1484	1612
Kinematic Viscosity, 275F, cSt	178	109	150	196	229	248	248

3.2.6 Compositional analysis of the AR1000 and AR4000 unaged asphalt cements, Califlux GP and mixtures of the AR4000 and Califlux were performed by Sahuaro Petroleum using a modification of the Rostler-Sternberg Procedure (14). Test results are tabulated in Table 7.

3.3 Diluent. The diluent (Kerosene) used in this study was termed 410-H and was obtained from Chevron, USA in Phoenix, Arizona.

TABLE 7

ROSTLER COMPOSITIONAL ANALYSIS OF CALIFLUX GP, AR1000, AR4000, AND AR4000 WITH 2, 6, AND 15 PERCENT CALIFLUX

	% By Weight					
Fraction	Califlux GP	AR1000	AR4000 + 15% Califlux	AR4000 + 6% Califlux	AR4000 + 2% Califlux	AR4000
Asphaltenes, A	1.8	14.4	10.8	11.4	12.7	11.1
Nitrogen Bases, N	21.8	28.6	22.3	24.5	23.7	25.0
First Acidefins, A_1	20.5	23.6	29.3	25.3	27.2	28.2
Second Acidefins, A_2	32.8	21.6	25.6	26.4	25.1	25.1
Parafins, P	23.7	11.2	11.6	12.3	11.3	10.6
Rostler Parameter[1]	0.74	1.59	1.26	1.29	1.40	1.49
Gotolski Parameter[2]	2.95	2.88	3.45	3.22	3.17	3.61

Note: [1] Rostler Parameter = $\frac{N + A_1}{P + A_2}$ [2] Gotolski Parameter = $\frac{A_1 + A_2 + N}{A + P}$

4.0 EXPERIMENTAL DESIGNS AND DATA ANALYSIS

4.1 Experiments performed during this study were designed as either sequentially or completely randomized fixed factorial models with two replications per cell. Data were analyzed using conventional analysis of variance techniques. Results were tested for significance at the 95 and 99 percent confidence levels.

4.2 Prior to performing analysis of variance (ANOVA), homogeneity of variance was tested by the Foster and Burr q-test (15). Appropriate data transformations were used when necessary to comply with variance homogeneity constraints required for analysis of variance.

4.3 For several of the experiments, following ANOVA, significant effects were ranked using the Newman-Keuls multiple range test (16).

4.4 One, two, three, and four-way designs were used in the study. Details of the analytical models used for each experiment are contained in Volumes I through V of this report.

5.0 ASPHALT-RUBBER MIXTURE PREPARATION

5.1 Individual asphalt-rubber formulations were prepared in 1000 gm batches using the Arizona Torque-Fork mixer. Details on components of the Torque-Fork are contained in Appendix A. Upon completion of mixing, the asphalt-rubber mixture was separated into 8 ounce sealed tins and stored at 0F until tested. Material used for testing was reheated only once after mixing.

5.1.1 Asphalt-rubber mixtures used for both replications of a specific test were obtained from the same mixture batch. This procedure was used to eliminate the mixing variable in test results.

5.2 The mixing procedure used was as follows:

A. Heat asphalt to 375F (191C) in the Torque-Fork mixing bowl with the stirring mechanism in operating at 200-300 rpm.

B. Increase mixer speed to 500 rpm after the asphalt has reached 375F (191C).

C. Introduce required amount of rubber into the asphalt as rapidly as possible while maintaining mixing speed at 500 rpm. The temperature will drop slightly, but do not adjust temperature controls.

D. Remove mixed asphalt-ruber from Torque-Fork mixer, transfer to storage tins, and place material into freezer after material has reached room temperature.

E. Record entire mixing cycle temperature and viscosity readings on a time chart.

5.3 To prepare a mixed asphalt-rubber mixture for testing, an 8 ounce tin of the specific mixture was removed from the freezer and heated to 275F on a hotplate while constantly being stirred. When the material reached 275F, it was ready for specimen fabrication.

6.0 TESTING PROCEDURES

6.1 Vacuum Capillary Absolute Viscosity at 140F (60C).

6.1.1 The test method used was a modification of the standard method of test ASTM D2171-66 (13), as outlined by Green and Tolonen (1). Modifications to the procedure include:

- Use of large bore capillary viscometers (Asphalt Institute No. 800 for example) to accommodate swollen rubber particles.
- Reduction of applied vacuum from the standard 30 cm Hg to 10 cm to produce slower flow times and prevent separation of asphalt and rubber during capillary flow.

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

$$K = \frac{M_H}{(H-h)} \qquad (1)$$

in which:

K = Instrument constant in poises/seconds 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 viscometer manufacturer.

6.1.3 Viscosity is flow time in seconds multiplied by the constant M_H for the applied vacuum.

6.1.4 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. The wood plunger snugly fits inside the syringe tube. Procedure for filling the viscometer tube as described by Green and Tolonen (1) is as follows:

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

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

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

6.1.4.4 Transfer of asphalt-rubber 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.

6.1.4.5 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.

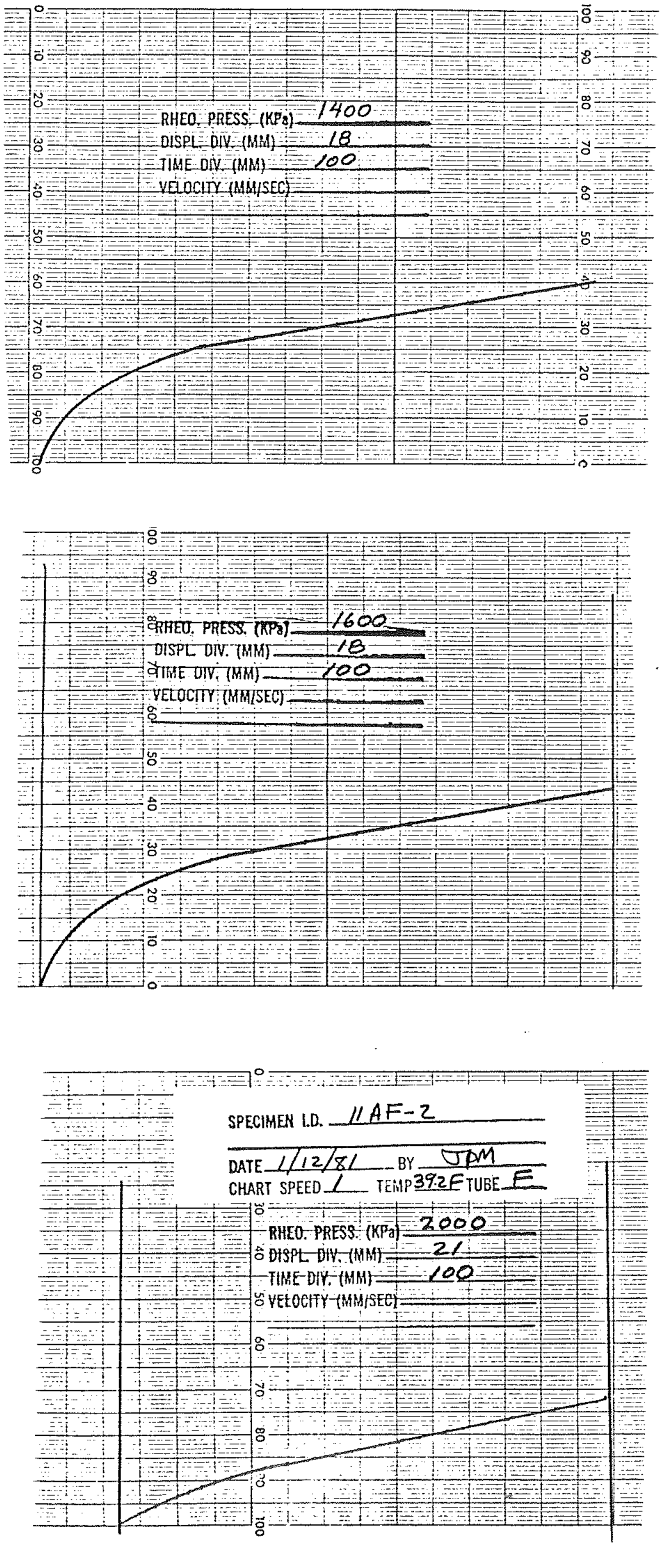
6.1.5 Data obtained from this testing procedure for analysis is the absolute viscosity of the asphalt-rubber at 140F (60C).

6.2 Schweyer Rheometer

6.2.1 The Schweyer rheometer (17) is described as a constant stress rheometer that produces a rheogram of apparent viscosity (η) versus shear rate ($\dot{\gamma}$).

6.2.2 Principle 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 linear variable displacement transducer (LVDT).

6.2.3 Plunger movement is recorded as a function of time on a strip chart. The corrected plunger movement is equated to specimen flow through the capillary tube. Figure 4 shows a typical output curve.



RHEO. PRESS. (KPa) 1400
DISPL. DIV. (MM) 18
TIME DIV. (MM) 100
VELOCITY (MM/SEC)

RHEO. PRESS. (KPa) 1600
DISPL. DIV. (MM) 18
TIME DIV. (MM) 100
VELOCITY (MM/SEC)

SPECIMEN I.D. 11AF-2
DATE 1/12/81 BY JPM
CHART SPEED 1 TEMP 392F TUBE E

RHEO. PRESS. (KPa) 2000
DISPL. DIV. (MM) 21
TIME DIV. (MM) 100
VELOCITY (MM/SEC)

Figure 4 Schweyer Output Curve

6.2.4 Movement of the plunger in the initial stage is nonlinear, but becomes linear when equilibrium (constant) flow rate is established. Velocity measurements are made on the linear portion.

6.2.5 Test procedure and calculations are as follows:

6.2.5.1 Pressure on the loading ram is read directly from a gauge on the front of the instrument. Pressure is supplied by pressurized gaseous nitrogen and can be controlled to suit test conditions and material characteristics.

6.2.5.2 Applied force is the product of pressure (P) and the machine constant (M).

6.2.5.3 Shear stress (τ) is the product of force and and the tube constant ($K_{\dot{\tau}}$).

$$\tau = (P \times M)\ (K_{\dot{\tau}}) \tag{2}$$

6.2.5.4 Velocity (V) is the slope of the linear portion of the movement versus time tracing obtained from the strip chart recorder.

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

$$\dot{\gamma} = VK_{\dot{\gamma}} \tag{3}$$

6.2.5.6 Apparent viscosity ($_a$) is given as:

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

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

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

6.2.5.8 Several runs are made and a rheogram is developed that is a plot of log of apparent viscosity (log η_a) versus log of shear rate (log $\dot{\gamma}$) where each point on the curve represents one test 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 a 0.05 sec.$^{-1}$ shear rate.

A typical rheogram and data used to generate it are shown in Figure 5.

6.2.6 Straight lines on a log log plot are represented by the power law equation:

$$y = a\,x^b \qquad (5)$$

or for the rheogram:

$$\eta_a = a(\dot{\gamma})^b \qquad (6)$$

where b is the slope of the straight line.

6.2.6.1 Materials with horizontal rheogram plots ($b = 0$) are Newtonian fluids and are not shear susceptible. In other words apparent viscosity is constant over a range of shear rates.

6.2.6.2 Materials with slopes down to the right ($b < 0$) are termed pseudoplastic. These materials are shear susceptible: as shear rate increases viscosity decreases.

6.2.6.3 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. This property may be of interest in the asphalt-rubber field since observations have been reported that performance in runway touchdown areas (high shear rate) 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.

IDENTIFICATION 2BA1

CHART NO. 2 TEMP. 4C TUBE F

(L), LVDT Chart Dsp., mm	24	14	11	6		
(T), Time Chart Dsp., mm	100	100	100	100		
(CS), Chart Speed, cm/min	1	1	1	1		
(P), Pressure, KPa	2000	1600	1200	800		
τ Pa	1.8×10^{6}	1.4×10^{6}	1.1×10^{6}	7.0×10^{5}		
V mm/sec	3.2×10^{-4}	1.9×10^{-4}	1.5×10^{-4}	8.0×10^{-5}		
$\dot{\gamma}$ sec^{-1}	2.4×10^{-3}	1.4×10^{-3}	1.1×10^{-3}	6.0×10^{-4}		
η_a Pa-sec	7.3×10^{8}	1.0×10^{9}	9.6×10^{8}	1.2×10^{9}		

Regression ($\eta_a = a\dot{\gamma}^b$): a = 1.1×10^{8} b = -3.2×10^{-1} R^2 = 0.89

Rheologic Constants : $\eta_{0.05}$ = 2.9×10^{8} $\eta_{1.0}$ 1.1×10^{8} c = 0.68

Plotting Points:

$\dot{\gamma}$	5.0×10^{-3}	5.0×10^{-4}	
η_a	6.1×10^{8}	1.3×10^{9}	

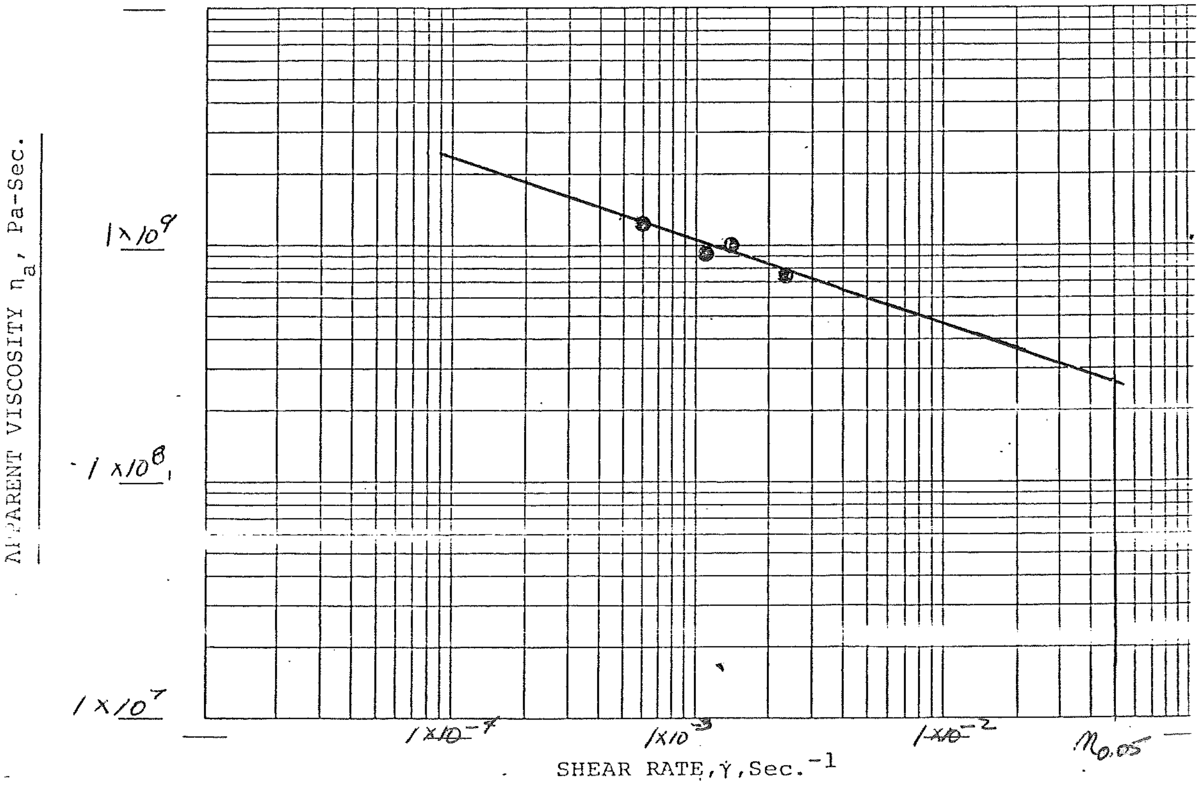


Figure 5 Schweyer Rheogram

6.2.7 Shear susceptibility 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 and the power law parameters a and b (slope).

6.2.7.1 Shear susceptibility in this study is reported by the parameter C. The shear susceptibility index is defined as:

$$C = 1 + \text{slope} = 1 + b \qquad (7)$$

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

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

6.2.7.4 Dilatant materials ($b > 0$) have shear susceptibility indices greater than 1.

6.2.8 Specimen tubes used for the constant stress rheometer consist of a specimen or flow tube of 9.47 mm (approximately 3/8 inches) in diameter with a capillary threaded onto the end of the flow tube which is smaller in diameter than the flow tube. Selection of capillary size is based on relative viscosity of the material under test conditions. Low viscosity materials require smaller capillaries than high viscosity materials.

6.2.8.1 The pressurized ram or plunger forces the large plug of material being tested down the flow tube and through the capillary. The capillary dictates flow rate and hence shear rate and shear stress. There is some question as to size effects of the capillary on measured viscosity. It should be expected that, for a given material under constant environmental conditions, different tube sizes would give the same viscosity if capillary size effects were not present. For asphalt-rubber, the possibility of particle interference needs to be considered as rubber particles may clog capillary openings and interfere with flow. If interference exists, measurements could be misleading since, instead of representing asphalt-rubber mass viscosity, elasticity of rubber or flow of asphalt around rubber particles could be measured.

6.2.9 Schweyer Rheometer tests at -20F (-29C), 39.2F (4C), and 77F (25C) were performed in this study using two different capillary tube sizes -"F" (4.650 mm diameter) and "G" (9.700 mm diameter). Apparent viscosity at a shear rate of 0.05 sec^{-1} and shear susceptibility constants C were calculated and analyzed using both F and G tubes.

6.3 Force-Ductility

6.3.1 The force-ductility test used during this project is a modificaton of the standard ductility test (ASTM D113-77, "Ductility of Bituminous Materials") and is similar to the procedure developed by Anderson and Wiley (18). Basically, the test is described as follows:

6.3.1.1 A modified ductility briquet is stretched at a constant rate in a constant temperature bath. The load required to stretch the specimen is continuously monitored with a load cell connected in series with the test specimen. Specimen elongation is also monitored during the test.

6.3.1.2 Asphalt-rubber test specimens are cast in a modified ductility mold as shown in Figure 6. This mold geometry provides a constant cross section so that the compliance of the material being tested can be evaluated.

6.3.1.3 Testing was performed at -20F (-29C), 39.2F (4C), and 77F (25C) using an extension rate of 1 cm per minute.

6.3.2 During the test, the following measurements are taken:

6.3.2.1 Initial gauge length (approximately 50 mm) between two gauge marks placed at each end of the specimen constant cross section portion.

6.3.2.2 Load obtained from the load cell output at 2 minute intervals.

6.3.2.3 Length between guage marks at the same time load readings are taken.

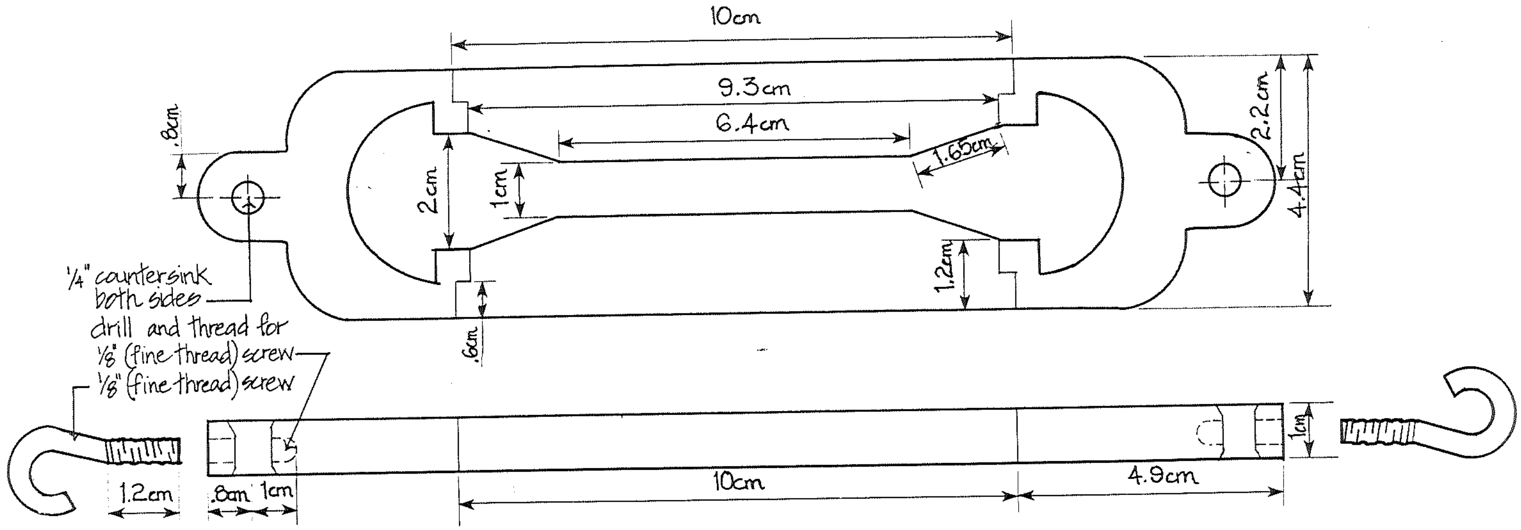


Figure 6 Modified Ductility Mold

6.3.3 From the above measurements, ten parameters are calculated and reported.

6.3.3.1 Load at Failure - reported in pounds.

6.3.3.2 Elongation at Failure - calculated as the length between gauge marks at failure minus initial gauge length in millimeters.

6.3.3.3 Engineering Stress at Failure - calculated as the load at failure per unit of original specimen cross section as follows:

$$\sigma_e = \frac{P}{A} \qquad (8)$$

in which:

σ_e = Engineering stress at failure, psi
P = Load at Failure, pounds
A = Original specimen cross section, in^2

6.3.3.4 Engineering Strain at Failure - calculated as follows:

$$\varepsilon_e = \frac{F - I}{F} \qquad (9)$$

in which:

ε_e = Engineering strain at failure, mm/mm
F = Gauge length at failure, mm
I = Initial gauge length, mm

6.3.3.5 True Stress at Failure - based on the specimen cross section at failure calculated assuming a constant specimen volume between gauge marks. True stress at failure was calculated using the following formula:

$$\sigma_t = \frac{P}{A_c} \qquad (10)$$

in which:

σ_t = True stress at failure, psi
P = Load at failure, pounds
A_c = Calculated specimen cross sectional area, in^2

6.3.3.6 True Strain at Failure - calculated as:

$$\varepsilon_t = (1 + \varepsilon_e) \quad (11)$$

in which:

ε_t = True strain at failure, mm/mm
ε_e = Engineering strain at failure, mm/mm

6.3.3.7 Engineering Creep Compliance at Failure - calculated as engineering strain at failure divided by engineering stress at failure. Units are psi^{-1}.

6.3.3.8 True Creep Compliance at Failure - calculated as true strain at failure divided by true stress at failure. Units are psi^{-1}.

6.3.3.9 Maximum True Creep Compliance - determined as the maximum true creep compliance value reached throughout the test duration.

6.3.3.10 Time to Maximum True Creep Compliance - the elapsed time in minutes to reach maximum true creep compliance.

6.4 Sliding Plate Microviscometer

6.4.1 Eight ounce portions of asphalt-rubber mixtures prepared at Western Technologies, Inc. during this study were shipped to Petroleum Sciences, Inc., Spokane, Washington for testing using the sliding plate microviscometer at 32F (0C).

6.4.2 The testing procedure used is a modification of the elastic rebound procedure described by Green and Tolonen (1). The procedure used is discussed in following sections.

6.4.2.1 Apparatus consists of:

- sliding plate microviscometer as designed by Shell Development Company as described by Fenijn (19).
- glass microviscosity plates 20 mm x 30 mm x 10 mm.

- spacers 2.54 mm in thickness.
- temperature control bath

6.4.2.2 A 20 x 25 mm specimen of asphalt-rubber which is 2.5 mm in thickness is pour formed between the glass plates.

6.4.2.3 The testing procedure used consists of:

- Placing the prepared specimen in the sliding plate microviscometer and lowering the specimen into a constant temperature bath until thermal equilibrium is reached at the desired testing temperature.
- When temperature equilibrium is reached, a 500 gram shearing load is applied to the plates for 30 minutes and resulting deflections monitored.
- During this 30 minute creep cycle, 22 deflection measurements are obtained as a function of time.
- At the end of the 30 minute creep cycle, the 500 gram shear load is removed permitting the specimen to rebound under a no shear stress condition. Resulting rebound or recovery is monitored for 30 minutes. Again, 22 deflection measurements are obtained as a function of time. This procedure completes first cycle testing.
- Following completion of the 30 minute recovery phase, the specimen is once again loaded with the 500 gram load and allowed to creep for 30 minutes. Deflection after 30 minutes of creep is recorded and the 500 gram load then removed and the specimen permitted to recover.
- At the end of 30 minute recovery period, deflection is recorded and the specimen allowed to recover for 20 hours. At the end of the 20 hour recovery period, the total deflection is measured. This completes the second cycle and is the end of the test.

6.4.3 The response of the samples to shear stress was separated into a non-recoverable apparent viscosity component and a recoverable viscoelastic component. A schematic diagram of the strain vs time curve for the entire test is shown in Figure 7. Apparent viscosity was calculated from the non-recoverable strain (after conditioning for 20 hours following completion of the test), and the 9807 Pa stress (500 gm shear load) which is applied to the sample for 60 minutes as follows:

$$\eta = \frac{\tau}{\dot{\gamma}} \qquad (12)$$

in which:

η = apparent viscosity
τ = applied shear stress (9807 Pa)
$\dot{\gamma}$ = shear strain rate calculated based on the measured non-recoverable flow, specimen thickness, and 3600 second loading time.

The viscoelastic creep component of the data was calculated by subtracting the strain resulting from viscous flow from the total strain at each time interval. Viscous effects are not present in the strain recovery portion of the test, thus the data, as measured, is representative of the viscoelastic strain recovery response.

The viscoelastic portions of the creep and strain recovery data were each fitted to the following mathematical model as proposed by Green and Tolonen (1):

$$S_v = S_m(1-e^{-bt^n}) \qquad (13)$$

in which:

S_v = elastic strain at any time interval

t = time

S_m = maximum elastic strain obtainable at $t = \infty$

b,n = constants

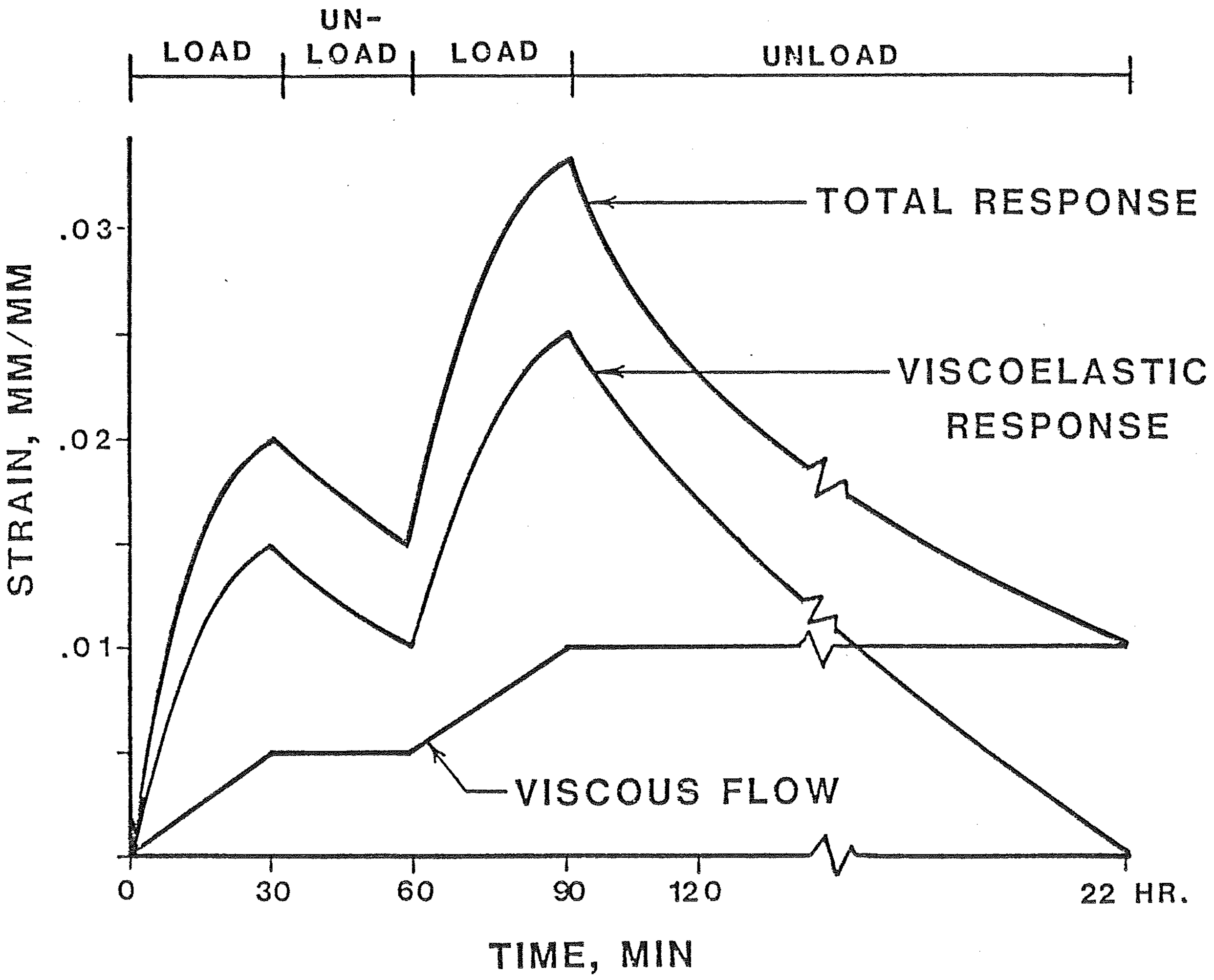


Figure 7 Strain as a Function of Time During the Creep-Strain Recovery Test

Equation 13 is not theoretically derived, but permits separation of viscous and elastic responses. In equation 13, S_m indicates the degree of elasticity of the asphalt-rubber while b and n are related to viscous characteristics.

6.4.4 For calculation purposes, equation 13 was rearranged to:

$$\frac{Sm}{S_m - S_v} = ebt^n \qquad (14)$$

The natural logarithm of the natural logarithm of equation 13 is then taken to yield:

$$\ln \ln \frac{Sm}{S_m - S_v} = n \ln t + \ln b \qquad (15)$$

Values for S_m were inserted into equation 15 on a trial and error basis and results compared to measured values until a maximum correlation coefficient was obtained. Correlation coefficients (r^2) were generally above 0.99. Coefficients b and n were determined as the intercept and slope of the $\ln \ln \frac{Sm}{S_m - S_v}$ vs. ln t plot (Figure 8) using the S_m value which gave the maximum correlation coefficient.

6.4.5 Parameters obtained from sliding plate microviscometer testing which are reported, analyzed and discussed are:

- Apparent viscosity
- First cycle 30 minute creep in microns.
- First cycle 30 minute recovery in microns.
- Second cycle 30 minute creep in microns.
- Second cycle 30 minute recovery in microns.
- Second cycle 20 hour recovery in microns.
- Second cycle 20 hour recovery minus second cycle 30 minute recovery in microns.

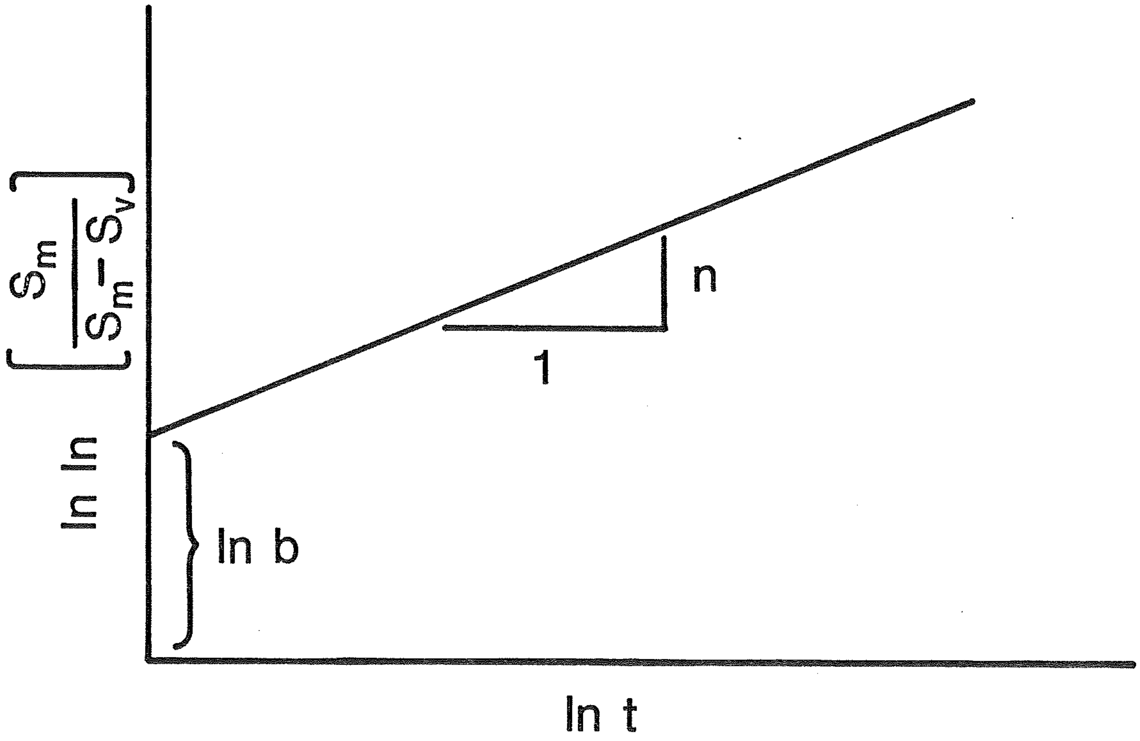


Figure 8 Ln Ln $(\frac{Sm}{S_m - S_v})$ vs. ln t Plot

- Percent first cycle recovery calculated as:

$$\frac{\text{First cycle recovery (30 min)}}{\text{First cycle creep (30 min)}} \times 100\%$$

- First cycle creep rheological constant S_m
- First cycle creep rheological constant b
- First cycle creep rheological constant n
- First cycle recovery rheological constant S_m
- First cycle recovery rheological constant b
- First cycle recovery rheological constant n

6.5 Viscosity During Mixing at 375F (191C) by the Arizona Torque Fork.

6.5.1 Viscosity during mixing at 375F (191C) by the Torque Fork was determined by correlation of millivolt readings from the servodyne unit with calibration readings obtained for S30000 Calibration oil obtained from Cannon Instrument Company. Details of construction and calibration of the Torque-Fork are contained in Appendix A.

6.5.2 During mixing Servodyne millivolt output readings were taken at 15 minutes and 1 hour following reestablishment of mixing temperature (375F) after introduction of rubber. Millivolt readings were transformed to viscosity in poise using techniques described in Appendix A..

6.6 Viscosity During Mixing at 375F (191C) by the Haake Rotational Viscometer.

6.6.1 Viscosity during mixing at 375F (191C) was determined at 15 minutes and 1 hour following reestablishment of mixing temperature (375F) after introduction of rubber by reading directly from the Haake viscometer. Viscosity readings are reported in poise.

6.7 Low temperature fracture tests were performed using the McDonald procedure, Method B as described in Appendix B.

6.8 Ring and ball softening point was determined in accordance with ASTM D2398-76, "Softening Point of Bitumen in Ethylene Glycol (Ring-and-Ball)" (13). ASTM D36-76 was not used due to the high softening point of several of the asphalt-rubber mixtures tested.

7.0 EFFECTS OF RUBBER TYPE, CONCENTRATION, AND ASPHALT

7.1 In order to evaluate the effects of rubber type, concentration, and asphalt on physical properties of asphalt-rubber mixtures, a statistically designed sequentially randomized fixed factorial three factor experiment with two replications per cell was performed. The experimental matrix is shown in Figure 9. Details on experimental design, analysis, and results are contained in Volume I of the report entitled, "Effects of Rubber Type, Concentration, and Asphalt."

7.2 Levels of Independent Variables

7.2.1 Rubber type at six levels:

- TPO44
- TPO27
- 50/50 mix (by weight) of TPO27 and TPO44
- GT274
- USRF
- 50/50 mix (by weight) of GT274 and USRF

7.2.2 Rubber quantity at four levels - 15, 20, 25, and 30 percent by weight of the asphalt-rubber mixture.

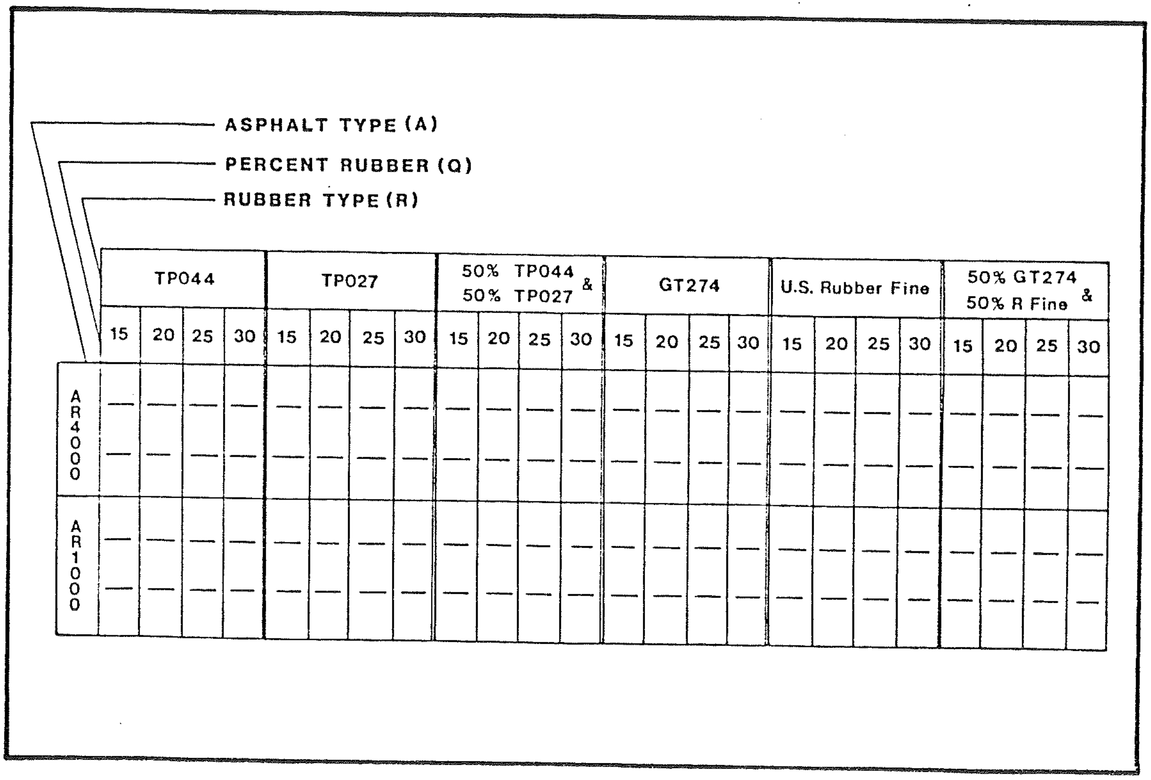
7.2.3 Asphalt at two levels - AR1000, and AR4000 with two percent Califlux GP extender oil.

7.3 Test Procedures Utilized:

- Absolute Viscosity at 140F (60C)
- Schweyer Rheometer at 39.2F (4C)
- Force-Ductility at 39.2F (4C)
- Sliding Plate Microviscometer at 32.0F (0C)
- Viscosity during mixing at 375F (191C) using the Arizona Torque-Fork
- Viscosity during mixing at 375F (191C) using the Haake Rotational Viscometer

7.4 Conclusions

7.4.1 A summary of three-way ANOVA results is tabulated in Table 8. This table indicates independent variables and interactions which were found to significantly affect test parameters studied. From Table 8 it is easily seen that constituent materials, rubber type and concentration, and asphalt, significantly influence many of the test paramters considered in this experiment.



	TP044				TP027				50% TP044 & 50% TP027				GT274				U.S. Rubber Fine				50% GT274 & 50% R Fine			
	15	20	25	30	15	20	25	30	15	20	25	30	15	20	25	30	15	20	25	30	15	20	25	30
AR4000	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —
AR1000	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —	— —

Figure 9 Experimental Test Matrix

TABLE 8 Summary of Three-Way ANOVA Results at the 0.05 Level of Significance

TEST PARAMETER

EFFECT

	R	Q	A	RQ	RA	QA	RQA
ABSOLUTE VISCOSITY (140F)	Y*	Y	Y	Y	Y	-	Y
SCHWEYER RHEOMETER (39.2F)							
Constant(C), G-tube	Y	Y	-	-	-	-	-
Constant(C), F-tube	Y	Y	-	Y	Y	-	Y
App. Viscosity, G-tube	Y	-	Y	-	-	-	-
App. Viscosity, F-tube	Y	Y	Y	Y	Y	-	-
FORCE DUCTILITY (39.2F)							
Load at Failure	Y	Y	Y	Y	Y	Y	Y
Elongation at Failure	Y	Y	Y	Y	-	Y	Y
Eng. Stress at Failure	Y	Y	Y	Y	Y	Y	-
Eng. Strain at Failure	Y	Y	Y	Y	-	Y	Y
True Stress at Failure	Y	Y	Y	Y	-	Y	-
True Strain at Failure	Y	Y	Y	Y	-	Y	Y
Eng. Creep Compliance	Y	Y	Y	Y	Y	Y	Y
True Creep Compliance	Y	Y	Y	Y	Y	Y	Y
Max.True Creep Compliance	Y	Y	Y	Y	Y	Y	Y
Time to Max.T.Creep Compl.	Y	Y	Y	Y	-	Y	-
SLIDING PLATE MICRO-VISCOMETER (32F)							
App. Viscosity	Y	Y	Y	Y	Y	Y	Y
1st Cycle 30 min. Creep	Y	Y	Y	Y	-	Y	-
1st Cycle 30 min.Recovery	Y	Y	Y	Y	-	-	-
2nd Cycle 30 min. Creep	Y	Y	Y	Y	Y	Y	Y
2nd Cycle 30 min.Recovery	Y	Y	Y	Y	-	-	Y
2nd Cycle 20 hr. Recovery	Y	Y	Y	Y	-	-	-
2nd Cyc.20 hr.30 min.Recov.	Y	Y	Y	Y	-	-	-
1st Cycle % Rebound	Y	Y	Y	Y	Y	Y	-
1st Cylcle b	Y	Y	-	Y	Y	-	-
1st Cycle n	-	Y	-	-	Y	-	-
2nd Cycle b	Y	Y	Y	-	-	-	Y

*Note: Y = significant at the 0.05 level
- = not significant at the 0.05 level
R = rubber
Q = rubber concentration
A = asphalt

7.4.2 The following specific conclusions were reached based on test results, statistical analyses, and interpretation performed during this experiment.

7.4.2.1 Absolute viscosity. Results indicate that the absolute viscosity at 140F (60C) of asphalt-rubber mixtures is different for mixtures containing different rubber types and particle sizes, different rubber concentrations, and different asphalt grades. Variability of test results is high (coefficients of variation of 20 percent were common during testing) even with the large capillary tube bore sizes utilized. Data generated tends to indicate that increasing rubber particle size and concentration may increase testing variability. Test results varied from a low of 7,636 poises to a high of 691,256 poises for different formulations.

7.4.2.2 Schweyer Rheometer. Shear susceptibility and apparent viscosity of asphalt-rubber mixtures as measured by the Schweyer rheometer at 39.2F (4C) were found to be different for different rubber types concentrations. Asphalt grade did not influence shear susceptibility, but did influence apparent viscosity.

Testing variability for the parameters obtained from the Schweyer Rheometer was high (many coefficients of variation in excess of 25 percent for shear susceptibility and 50 percent for apparent viscosity). Data indicates that rubber particle size may influence testing variability. Test results for larger rubbers (TPO44) are more variable than with smaller rubbers (USRF). These differences may be due to increased flow interference through capillary bores as rubber particle size increases.

Asphalt-rubber mixtures tested tended to be more pseudoplastic in nature than dilatent.

Viscosity of mixtures tested varied from a low of 17.5 x 10^6 Pa-s to a high of 55,210 x 10^6 Pa-s for different formulations.

7.4.2.3 Force Ductility. Stress, strain, and creep compliance characteristics of asphalt-rubber mixtures, as measured by the force-ductility test at 39.2F (4C) were found to be different for mixtures containing different rubber types and particle sizes, different rubber concentrations, and different asphalt grades. Testing variability for many of the parameters considered was rather low (coefficients of variation generally less than 10 percent) when compared to other test types considered in this experiment. It is suggested that lower testing variability is related to the unconfined tensile nature of the test which would not result in rubber particle interference with testing apparatus as with absolute viscosity, Schweyer Rheometer, or sliding plate microviscometer testing.

True stress at failure for mixtures considered varied from a low of 296 psi to a high of 1279 psi while true strain at failure varied from 1.41 mm/mm to 2.46 mm/mm.

7.2.4.4 Sliding Plate Microviscometer. Apparent viscosity, creep, recovery, and rebound charactertistics of asphalt-rubber mixtures, as measured by the sliding plate microviscometer, were found to be different for different rubber types and particle sizes, different rubber concentrations, and different asphalts. Interactions between mixture components were identified by several of the measurements. Testing variability was rather low (less than 15 percent coefficients of variation) for several of the parameters - second cycle creep and recovery, and first cycle percent rebound and creep, but higher (coefficients of variation between 15 and 20 percent) for other parameters.

7.2.4.5 Vicosity During Mixing. Viscosity during mixing at 375F (191C) as measured to Arizona Torque-Fork or Haake Viscometer of asphalt-rubber varies depending on rubber types, rubber concentration, and asphalt type. The reaction between rubber and asphalt can be monitored during mixing using either device by monitoring mixture viscosity changes. Differences exist in viscosity by the Torque-Fork and Haake at high rubber concentrations and 1 hour of mixing. At 15 minutes of mixing with low rubber concentrations, viscosity results obtained with the two devices are comparable. Significant viscosity increases with increased mixing time were noted with both devices.

8.0 EFFECTS OF ASPHALT

8.1 The effects of asphalt on physical characteristics of asphalt-rubber mixtures was further investigated by performing a statistically designed experiment which utilized rubber types, concentrations, and asphalts comparable to those used by major commercial suppliers of asphalt-rubber mixtures. The experiment was designed as a sequentially randomized, three factor fixed factorial with two replications per cell as shown in Figure 10. Details on experimental design, analysis, and results are contained in Volume II entitled, "Effects of Asphalt."

8.2 Levels of Independent Variables

8.2.1 Rubber type at two levels - TPO44 and GT274.

8.2.2 Rubber quantity at two levels - 20 and 25 percent by weight of the asphalt-rubber mixture.

8.2.3 Asphalt at four levels:

- AR1000
- AR4000 + 2 percent Califlux by weight of asphalt cement
- AR4000 + 6 percent Califlux by weight of asphalt cement
- AR4000 + 15 percent Califlux by weight of asphalt cement.

8.3 Following analysis of variance, significant effects were ranked using the Newman-Keuls multiple range test (16).

8.4 Test procedures utilized in this experiment are:

- Absolute Viscosity at 140F (60C)
- Schweyer Rheometer at 39.2F (4C)
- Force-Ductility at 39.2F (4C)
- Viscosity during mixing at 375F (191C) using the Arizona Torque-Fork
- Viscosity during mixing at 375F (191C) using the Haake Rotational viscometer.

ASPHALT
CONCENTRATION
RUBBER

	GT 274		TP 044	
	20%	25%	20%	25%
AR4000 + 15% Cal.*	— —	— —	— —	— —
AR4000 + 6% Cal.	— —	— —	— —	— —
AR4000 + 2% Cal.	— —	— —	— —	— —
AR1000	— —	— —	— —	— —

*Note: Cal. = Califlux GP

Figure 10 Experimental Test Matrix

8.5 Conclusions

8.5.1 A summary of three-way ANOVA results is tabulated in Table 9. This table indicates independent variables and interactions which were found to significantly affect test parameters studied. From Table 9, it is noted that except for shear susceptibility constants obtained from the Schweyer rheometer, different asphalt cements resulted in asphalt-rubber mixtures with significantly different physical properties.

8.5.2 With many of the parameters investigated only slight or no differences were noted with results obtained from the AR1000 and the AR4000 with 6 percent Califlux.

8.5.3 Apparent viscosity as measured by the Schweyer rheometer is less for asphalt-rubber mixtures containing the AR4000 with 15 percent Califlux than for the other asphalts.

8.5.4 In the force-ductility test, load and stress at failure are highest for the stiffest asphalt (AR4000 with 2 percent Califlux) and lowest for the least viscous asphalt (AR4000 with 15 percent Califlux) for all but one mixture.

8.5.5 In the force-ductility test, elongation or strain at failure for TP044 mixtures is not influenced by asphalt as much as for GT274 mixtures. An asphalt-concentration interaction exists for GT274 mixtures in that at 20 percent rubber, less viscous asphalts result in higher failure strains or elongations, while, at 25 percent rubber, the least viscous asphalt (AR4000 with 15 percent Califlux) resulted in the lowest failure strain and elongation.

8.5.6 Creep compliance as determined in the force-ductility test was highest for the asphalt-rubber mixtures containing the least viscous asphalt (AR4000 with 15 percent Califlux) and lowest for the stiffest (AR4000 with 2 percent Califlux).

8.5.7 Viscosities during mixing at 375F as measured by the Torque-Fork or Haake viscometer are generally lower for the lower viscosity asphalts but several interactions between asphalt, rubber type, and rubber concentration appear to exist.

TABLE 9 Summary of Three-Way ANOVA Results at the 0.05 Level of Significance

TEST PARAMETER EFFECT	R	Q	A	RQ	RA	QA	RQA
ABSOLUTE VISCOSITY (140F)	-*	Y	Y	-	-	-	-
SCHWEYER RHEOMETER (39.2F)							
Constant(C), G-tube	-	-	-	-	-	-	-
Constant(C), F-tube	-	-	-	-	Y	-	-
App. Viscosity, G-tube	-	-	Y	-	-	-	-
App. Viscosity, F-tube	Y	-	Y	-	-	-	-
FORCE DUCTILITY (39.2F)							
Load at Failure	Y	Y	Y	Y	-	Y	-
Elongation at Failure	Y	Y	Y	Y	-	Y	Y
Eng. Stress at Failure	Y	Y	Y	Y	-	Y	-
Eng. Strain at Failure	Y	Y	Y	Y	Y	Y	Y
True Stress at Failure	Y	Y	Y	Y	Y	Y	-
True Strain at Failure	Y	Y	Y	Y	-	Y	Y
Eng. Creep Compliance	Y	Y	Y	Y	Y	Y	Y
True Creep Compliance	-	Y	Y	Y	Y	Y	-
Max.True Creep Compliance	Y	Y	Y	Y	Y	Y	-
Time to Max.T.Creep Compl.	Y	Y	Y	-	Y	-	-

*Note: Y = significant at the 0.05 level
- = not significant at the 0.05 level
R = rubber
Q = rubber concentration
A = asphalt

9.0 EFFECT OF DILUENT

9.1 The effects of additions of diluent and cure time following diluent addition were evaluated by performing a completely randomized two factor fixed factorial experiment with two replications per cell. The experimental matrix is shown in Figure 11. The experiment utilized a single asphalt-rubber formulation, 25 percent TPO44 and 75 percent AR1000 asphalt with four diluent concentrations. Diluents are routinely utilized in commercial production of asphalt-rubber mixtures composed of TPO44 and AR1000. Details on experimental design, analysis, and results are contained in Volume III entitled, "Effects of Diluent."

9.2 Levels of Independent Variables

9.2.1 Diluent concentration at four levels - 0, 2, 4, and 6 percent by weight of asphalt-rubber mixture.

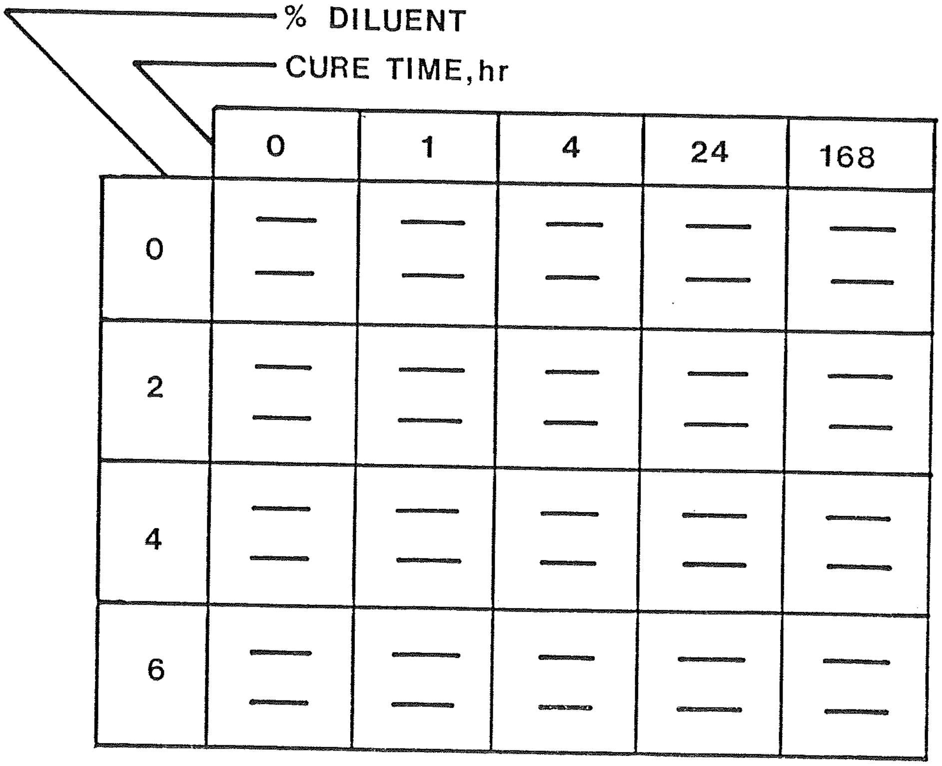
9.2.2 Curing time at 140F (60C) at five levels - 0, 1, 4, 24, and 168 hours.

9.3 Test Procedures Utilized:

- Ring and Ball Softening Point
- Schweyer Rheometer at 39.2F (4C)
- Force-Ductility at 39.2F (4C)
- Viscosity during mixing at 375F (191C) by the Arizona Torque-Fork
- Viscosity during mixing at 375F (191C) by the Haake Rotational viscometer.

9.4 Conclusions

9.4.1 A summary of two-way ANOVA results is tabulated in Table 10. This table indicates that diluent concentration, cure time and the interaction significantly affect the test parameters studied. From Table 10, it is noted that diluent concentration and cure time are significant effects for ring and ball softening point, and force-ductility load and stress at failure as well as for creep compliance results. Elongation and strains at failure from the force-ductility test were not significantly affected by diluent concentration or cure time. Apparent viscosity measured with the Schweyer rheometer varied with percent diluent but not with cure time.



% DILUENT / CURE TIME, hr	0	1	4	24	168
0	___ ___	___ ___	___ ___	___ ___	___ ___
2	___ ___	___ ___	___ ___	___ ___	___ ___
4	___ ___	___ ___	___ ___	___ ___	___ ___
6	___ ___	___ ___	___ ___	___ ___	___ ___

Figure 11 Experimental Test Matrix

TABLE 10 Summary of Significant Effects

TEST PARAMETER

EFFECT

	C	T	CT
Softening Point	Y	Y	Y
SCHWEYER RHEOMETER (39.2F)			
Constant(C), G-tube	Y	-	-
Constant(C), F-tube	-	-	-
App. Viscosity, G-tube	Y	-	-
App. Viscosity, F-tube	Y	-	-
FORCE DUCTILITY (39.2F)			
Load at Failure	Y	Y	Y
Elongation at Failure	-	-	-
Eng. Stress at Failure	Y	Y	Y
Eng. Strain at Failure	-	-	-
True Stress at Failure	Y	Y	-
True Strain at Failure	-	-	-
Eng. Creep Compliance	Y	Y	Y
True Creep Compliance	Y	Y	Y
Max.True Creep Compliance	Y	Y	Y
Time to Max.T.Creep Compl.	Y	Y	Y

*Note: Y = significant at the 0.05 level
- = not significant at the 0.05 level
C = diluent concentration
T = cure time

9.4.2 Ring and ball softening points tend to decrease with increasing diluent concentrations and increased cure time tends to decrease the softening effect of the diluent, possibly due to diluent evaporation.

9.4.3 Increased diluent concentration decreases apparent viscosity at 39.2F as measured using the Schweyer rheometer. Schweyer viscosity is not influenced by cure time.

9.4.4 Increased diluent concentration lowers load and stresses at failure in the force-ductility test. Increased cure time (24 to 168 hours at 140F) increases load and stresses at failure of asphalt-rubber mixtures which contain 2, 4, and 6 percent diluent.

9.4.5 Creep compliance of asphalt-rubber mixtures increase as diluent concentration increases (mixes become softer) and increased cure time tends to decrease the softening effect of the diluent, possibly due to diluent evaporation.

9.4.6 Diluent additions cause slight, but detectable decreases in mixture viscosity at 375F as indicated by the Torque-Fork.

9.4.7 The Haake viscometer did not indicate mixture viscosity differences due to percent diluent.

10.0 EFFECT OF TEMPERATURE

10.1 In order to evaluate the effects of temperature on the physical properties of asphalt-rubber mixtures, a statistically designed experiment which tested eight asphalt-rubber formulations at three temperatures was performed. The experiment was designed as a sequentially randomized four factor fixed factorial model with two replications per cell as shown in Figure 12. Details of the design of the experiment, analyses, and results are contained in Volume V entitled, "Effect of Temperature."

10.2 Levels of Independent Variables

10.2.1 Rubber type at two levels - TPO44 and GT274

10.2.2 Rubber concentration at two levels - 20 and 25 percent by weight of the asphalt-rubber mixture.

10.2.3 Asphalt at two levels - AR1000 and AR4000 with 2 percent Califlux GP extender oil by weight of asphalt.

10.2.4 Testing temperature at three levels: 77F (25C), 39.2F (4C), and -20F (-29C).

10.3 Testing Procedures Utilized:

- Schweyer Rheometer
- Force-Ductility

10.4 Conclusions

10.4.1 A summary of four-way ANOVA results is tabulated in Table 11. This table indicates independent variables and interactions which were found to significantly affect test parameters studied. From Table 11, it is noted that temperature influences all parameters studied and that rubber type and concentration influence nearly all parameters studied. Additionally, several interactions exist including a rubber type-temperature interaction and an asphalt-temperature interaction.

10.4.2 The effect of temperature is to stiffen and strengthen mixtures as temperature is lowered as evidenced by increased loads and stresses at failure, increased viscosity, and decreased elongation, strains, and compliances at failure.

ASPHALT
RUBBER CONCENTRATION
RUBBER TYPE
TEMPERATURE

		77 F		39.2 F		-20 F	
		TP044	GT274	TP044	GT274	TP044	GT274
AR4000	20%	____ ____	____ ____	____ ____	____ ____	____ ____	____ ____
	25%	____ ____	____ ____	____ ____	____ ____	____ ____	____ ____
AR1000	20%	____ ____	____ ____	____ ____	____ ____	____ ____	____ ____
	25%	____ ____	____ ____	____ ____	____ ____	____ ____	____ ____

Figure 12 Experiment Test Matrix

TABLE 11 Summary of Four-Way ANOVA Results at the 0.05 Level of Significance

	R	Q	A	T	RQ	RA	RT	QA	QT	AT	RQA	RAT	QAT	RQT	RQAT
Schweyer Rheometer															
Constant (C), F-tube	Y	-	-	Y	-	-	-	-	-	-	-	-	-	-	-
App.Viscosity, F-tube	-	Y	-	Y	-	-	Y	-	-	-	-	-	-	-	-
Force-Ductility															
Load at Failure	Y	-	-	Y	-	-	Y	-	-	-	-	-	-	-	-
Elongation at Failure	Y	Y	-	Y	-	-	Y	-	Y	-	-	-	-	Y	-
Eng. Stress at Failure	Y	-	-	Y	-	-	Y	-	-	-	-	-	-	-	-
Eng. Strain at Failure	Y	Y	-	Y	-	-	Y	-	-	-	-	-	-	Y	-
True Stress at Failure	Y	-	Y	Y	Y	-	Y	-	-	Y	-	-	-	-	-
True Strain at Failure	Y	Y	Y	Y	-	-	Y	-	-	-	-	-	-	Y	-
Eng. Creep Compliance	Y	Y	Y	Y	-	Y	Y	-	Y	Y	-	Y	-	-	-
True Creep Compliance	Y	Y	Y	Y	-	-	Y	-	-	Y	-	-	-	-	-
Max.True Creep Compliance	Y	-	Y	Y	Y	-	Y	-	Y	Y	-	-	-	-	-

*Note: Y = significant at the 0.05 level
- = not significant at the 0.05 level
R = rubber
Q = rubber concentration
A = asphalt
T = test temperature

10.4.3 The existence of the rubber-temperature and asphalt-temperature interactions for several parameters indicate that asphalt-rubber mixtures containing the different rubber and asphalt types studied react differently at different temperatures, or, more simply stated, have differing temperature susceptibilities.

11.0 PHYSICAL PROPERTIES OF FIELD-MIXED ASPHALT-RUBBERS

11.1 Eight asphalt-rubber samples which were obtained from commercial field-produced asphalt-rubber mixtures used in the Buckeye-Liberty test project in Arizona were tested to determine the physical properties of field produced asphalt-rubber mixtures. Mixtures tested were produced by two of the major commercial asphalt-rubber suppliers designated as Supplier Nos. 1 and 2.

11.2 The field mixed samples contained three different rubber types and three different asphalts. Asphalt and rubber used in the field produced asphalt-rubbers were from the same sources as used in lab-produced mixtures.

Asphalt-rubber mixtures which were tested from Supplier No. 1 were formulated and designated as follows:

	Sample Designation			
Constituent	302	8NC	11A	3A
Asphalt Type	AR4000	AR1000	AR8000	AR1000
Asphalt Source	Edgington	Edgington	Edgington	Edgington
Rubber Type	TPO27	TPO44	TPO27	TPO44
% Rubber (Mix Basis)	20	25	20	25
% Diluent (Mix Basis)	0	0	0	4
Reaction Time, hr.	2.5	1.5	2.2	6.5

Asphalt-rubber mixtures which were tested from Supplier No. 2 were formulated and designated as follows:

	Sample Designation			
Constituent	4A	2A	403	101
Asphalt Type	AR4000	AR4000	AR4000	AR4000
Asphalt Source	Powerene	Powerene	Powerene	Powerene
Rubber Type	GT274	GT274	GT274	GT274
% Rubber (Mix Basis)	20	25	20	20
% Extender Oil (Mix Basis)	1.6	1.6	1.6	1.6
Reaction Time, hr.	4	4	24	166

11.3 Comparisons were made using one-way ANOVA techniques. The analytical matrix is shown in Figure 13. Further details on experimental design, analysis, and results are contained in Volume IV entitled, "Physical Field-Mixed Asphalt-Rubber Mixtures and Comparisons of Lab and Field-Mixed Asphalt-Rubbers."

11.4 Testing procedures utilized were:

- Ring and ball softening point
- Low temperature fracture
- Schweyer rheometer at 39.2F (4C)
- Force-ductility at 39.2F (4C)
- Absolute viscosity at 140F (60C)

11.5 Conclusions

11.5.1 A summary of one-way ANOVA results for field-produced mixtures is tabulated in Table 12. This table indicates significant differences exist between the field-produced mixtures. Significant differences exist in the absolute viscosity, Schweyer apparent viscosity in the G-tube, force-ductility engineering stress, strain, compliance at failure, and true stress, strain and compliance at failure, and ring and ball softening point. Schweyer shear susceptibility constants and apparent viscosity in the F-tube were not significantly different.

11.5.1.1 The addition of diluent to Supplier No. 1 mixtures significantly influenced engineering stress and creep compliance at failure, true stress and creep compliance at failure, and softening point. The addition of diluent softened the mixture as indicated by all tests.

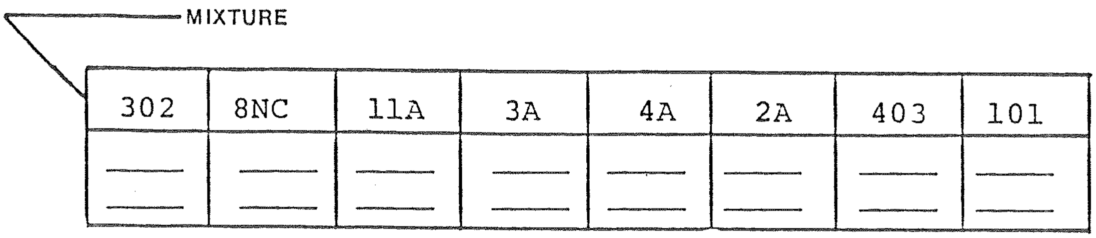
11.5.1.2 Increased reaction (166 hours) of the Supplier No. 2 mixture significantly influenced absolute viscosity, Schweyer apparent viscosity, engineering stress and creep compliance at failure and true stress and creep compliance at failure. Increased reaction time softened the mixtures as indicated by all tests except the Schweyer apparent viscosity which indicates a stiffer mix.

TABLE 12

SUMMARY OF SIGNIFICANCE FOR FIELD-PRODUCED MIXTURES

Absolute Viscosity	Y*
SCHWEYER RHEOMETER (39.2F)	
Constant (C), G-tube	-
Constant (C), F-tube	-
Apparent Viscosity, G-tube	Y
Apparent Viscosity, F-tube	-
FORCE-DUCTILITY (39.2F)	
Engineering Stress at Failure	Y
Engineering Strain at Failure	Y
True Stress at Failure	Y
True Strain at Failure	Y
Engineering Creep Compliance	Y
True Creep Compliance	Y
SOFTENING POINT	Y

*Note: Y = significant at the 0.05 level
- = not significant at the 0.05 level



302	8NC	11A	3A	4A	2A	403	101
______ ______	______ ______	______ ______	______ ______	______ ______	______ ______	______ ______	______ ______

Figure 13 Data Analysis Matrix, Field-Mixtures

11.5.1.3 Supplier No. 1 mixtures tended to have higher absolute viscosities, engineering and true stresses at failure, and softening points than Supplier No. 2 mixtures.

Additionally, Supplier No. 1 mixtures tended to have lower engineering and true strains at failure and lower engineering creep compliance at failure than Supplier No. 2 mixtures.

12.0 COMPARISON OF LAB AND FIELD-MIXED ASPHALT-RUBBERS

12.1 Several of the asphalt-rubber mixtures which were produced in the field were also produced in the laboratory thus enabling comparisons between properties of field and lab mixed asphalt-rubbers. For the comparison between laboratory and field-mixed materials, mixtures which had the same formulations were compared using one-way ANOVA techniques.

12.1.1 For Supplier No. 1, mixtures 3A and 8NC were duplicated in the lab. Comparable laboratory mixtures, however, were tested after curing, 1, 4, 24, and 168 hours of 140F. Therefore, test results for field-produced mixtures 3A (4% diluent) and 8NC (0% diluent) were compared in separate analyses to results of appropriate lab mixtures at the five cure times using one-way ANOVA. The data analysis matrix for these comparisons is presented in Figure 14. This analysis matrix is used for each of the two comparisons (0% diluent, and 4% diluent).

12.2 All mixtures produced by Supplier No. 2 were of the same component formulation but differed only by reaction time. Therefore, field-produced mixtures 4A, 2A, 403, and 101 were compared to the lab produced mixture of the same formulation (20 percent GT274 rubber, AR4000 Powerene asphalt, and 2.0 percent extender oil). Therefore, test results for the lab-produced mixture were compared to results for the four field-produced mixtures with different reaction times using one-way ANOVA. The data analysis matrix for this comparison is presented in Figure 15.

12.3 Further details on materials analytical techniques, and results are contained in Volume IV entitled, "Physical Properties of Field-Mixed Asphalt-Rubber Mixtures and Comparison of Lab and Field-Mixed Asphalt-Rubbers."

12.4 Conclusions

12.4.1 Table 13 is a summary of ANOVA and Newman-Keuls results for the comparison between lab and field mixtures produced by Supplier No. 1.

MIXTURE

FIELD[1]	L-0[2]	L-1	L-4	L-24	L-168

*Notes:

[1] Field mix is either 3A or 8NC

[2] 0, 1, 4, 24, and 168 are hours of curing at 140F

Figure 14 Data Analysis Matrix, Field-Lab Comparison, Supplier No. 1

MIXTURE

LAB	4A	2A	403	101

Figure 15 Data Analysis Matrix, Field-Lab Comparison, Supplier No. 2

TABLE 13

COMPARISON OF LAB-AND-FIELD PRODUCED MIXTURES, SUPPLIER NO. 1

	0% Diluent			4% Diluent		
	Higher	Lower	Same	Higher	Lower	Same
Schweyer Rheometer (39.2F)						
Constant (C), G-tube			X[1]		X	
Constant (C), F-tube			X			X
App.Viscosity,G-tube			X			X
App.Viscosity,F-tube			X		X	
Force-Ductility						
Eng.Stress at Failure		X			X	
Eng.Strain at Failure			X	X		
True Stress at Failure			X		X[2]	
True Strain at Failure			X	X		
Eng. Creep Compliance	X			X		
True Creep Compliance	X[3]					X
Softening Point		X		X		

Notes:

1 X signifies if the field-produced mixture test result is higher, lower, or the same as for the lab produced mixtures.

2 Result is the same as for lab mixture with no cure.

3 Result is the same as for lab mixture with 1 hour cure.

12.4.1.1 For mixtures containing 0 percent diluent, engineering stress at failure is lower for the field-produced mixtures than for lab produced mixtures, engineering and true creep compliances are higher for the field produced mixture, and other tests, results are the same. These results indicate that the field-produced mixture is softer and not as stiff as the lab-produced mixtures.

12.4.1.2 For mixtures containing 4 percent diluent, apparent viscosity, engineering and true stress at failure, and softening point are lower for the field-produced mixture than for lab produced mixtures. Engineering and true strains at failure, and engineering creep compliance at failure are higher for field-produced mixtures than for the lab. For other tests, results are not different. These results indicate that the field-produced mixtures are softer and not as stiff as lab produced mixtures.

12.4.2 Table 14 is a summary of ANOVA and Newman-Keuls results for the comparison between the lab and field mixtures produced by Supplier No. 2.

12.4.2.1 Absolute viscosity, engineering stress at failure and true stress at failure for the lab produced mixture are higher than for field-produced mixtures. Engineering and true strains at failure, and engineering and true creep compliances at failure are lower for the lab mixture than for the field mixtures. These results indicate that field-produced mixtures are softer and not as stiff as the lab produced mixtures. Schweyer Rheometer test results are not different.

TABLE 14

COMPARISON OF LAB-AND-FIELD PRODUCED MIXTURES, SUPPLIER NO. 2

	Higher	Lower	Same
Schweyer Rheometer (39.2F)			
Constant (C), G-tube			X[1]
Constant (C), F-tube			X
App.Viscosity,G-tube			X
App.Viscosity,F-tube			X
Force-Ductility			
Eng.Stress at Failure	X		
Eng.Strain at Failure		X	
True Stress at Failure	X[2]		
True Strain at Failure		X	
Eng. Creep Compliance		X[3]	
True Creep Compliance		X[4]	
Softening Point			

Notes:

1 X signifies if the field-produced mixture test result is higher, lower, or the same as for the lab produced mixtures.

2 Result is the same as for field mixture 4A.

3 Result is the same as for field mixtures 4A and 403.

4 Result is the same as for field mixture 4A.

13.0 CONCLUSIONS

13.1 Based on results of the various laboratory experiments and statistical analyses performed during this investigation, several conclusions regarding physical properties of asphalt-rubber mixtures and testing procedures used were reached.

13.1.1 Physical properties of asphalt-rubber mixtures vary and depend on the type of rubber utilized, rubber concentration, and asphalt grade. Variations in properties are noted at testing temperatures ranging from -20F (-29C) to 375F (191C).

13.1.2 Use of asphalts of lower viscosity result in asphalt-rubber mixtures which have lower viscosities and failure stresses and higher failure strains and creep compliances.

13.1.3 Additions of diluent to asphalt-rubber mixtures tends to soften the mixtures as evidenced by higher failure strains and creep compliances at failure and lower failure stresses and softening points. The effect of cure time is to decrease the effects of diluent additions as cure time increases.

13.1.4 Temperature was found to significantly affect physical properties of asphalt-rubber mixtures. The effect is to stiffen and strengthen mixtures as temperature is lowered. Additionally, it was noted that asphalt-rubber mixtures formulated with the high natural rubber content devulcanized crumb rubber were more temperature susceptible than mixtures formulated with the ambient grind crumb rubber.

13.1.5 Physical properties of the field-produced asphalt-rubber mixtures studied differ widely. Diluent additions and increased mixing times tended to soften the mixtures.

13.1.6 The comparison of physical properties of lab and field produced mixtures showed that the mixing procedure used in the lab produced mixtures which were significantly stiffer than mixtures produced in the field.

13.1.7 The force-ductility test is the most sensitive of all tests utilized in this investigation to changes in asphalt-rubber mixture components and is the least variable of the tests studied.

13.1.8 The force-ductility and sliding plate microviscometer tests yield several parameters with testing variability which is low enough to permit use in specifying asphalt-rubber physical properties.

13.1.9 Use of the Schweyer rheometer and absolute viscosity test procedures with asphalt rubber materials yielded results which had high degrees of variability. Variability, in many cases, was high enough to mask differences in mixture characteristics which were noted with force-ductility and sliding plate microviscometer testing.

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APPENDIX A

MANUFACTURE AND CALIBRATION OF THE ARIZONA TORQUE-FORK FOR MIXING AND MONITORING VISCOSITY OF ASPHALT-RUBBER MATERIALS

INTRODUCTION

The Arizona Torque-Fork is a rotational type instrument which is used to mix asphalt-rubber materials in the laboratory to prepare them for testing. The Torque-Fork consists of a fluid containment vessel and heating mantle, mixing motor and propeller, appropriate mixing motor speed and heating mantle temperature controls, and a data recorder. During mixing at elevated temperatures, the viscous drag between the mixing propeller and the hot asphalt-rubber is monitored by voltage requirements of the stirring motor required to maintain a constant mixing speed. Thus, viscosity changes of asphalt-rubber materials which occur as the asphalt and rubber react can be determined.

COMPONENTS OF THE TORQUE-FORK

The Torque-Fork consists of 5 major components - (1) fluid containment vessel and heating mantle, (2) mixing motor and propeller, (3) motor controls, (4) temperature controls, and (5) a data recorder. A schematic diagram of these components is shown in Figure A-1. A listing of required equipment and ordering information is presented in Table A-1. Discussions of the function and operation of the components follow.

Fluid Vessel and Heating Mantle. Materials being mixed in the Torque-Fork are contained in a covered 2000 ml glass reaction vessel. Temperature is monitored with a temperature sensor immersed in the material being mixed. The temperature sensor is connected to the temperature control unit which varies electrical input at the heating mantle surrounding the glass reaction vessel.

Mixing Motor and Propeller. The motor used in the Torque-Fork is a constant velocity motor which maintains constant speed by drawing a variable voltage as rotational resistance changes. Voltage requirements at different rotational resistances are measured by a voltmeter connected to the motor. Apparent viscosity measurements of the material being mixed are made by correlating the voltage draw of the motor at a specific speed to the viscosity of standard reference oils.

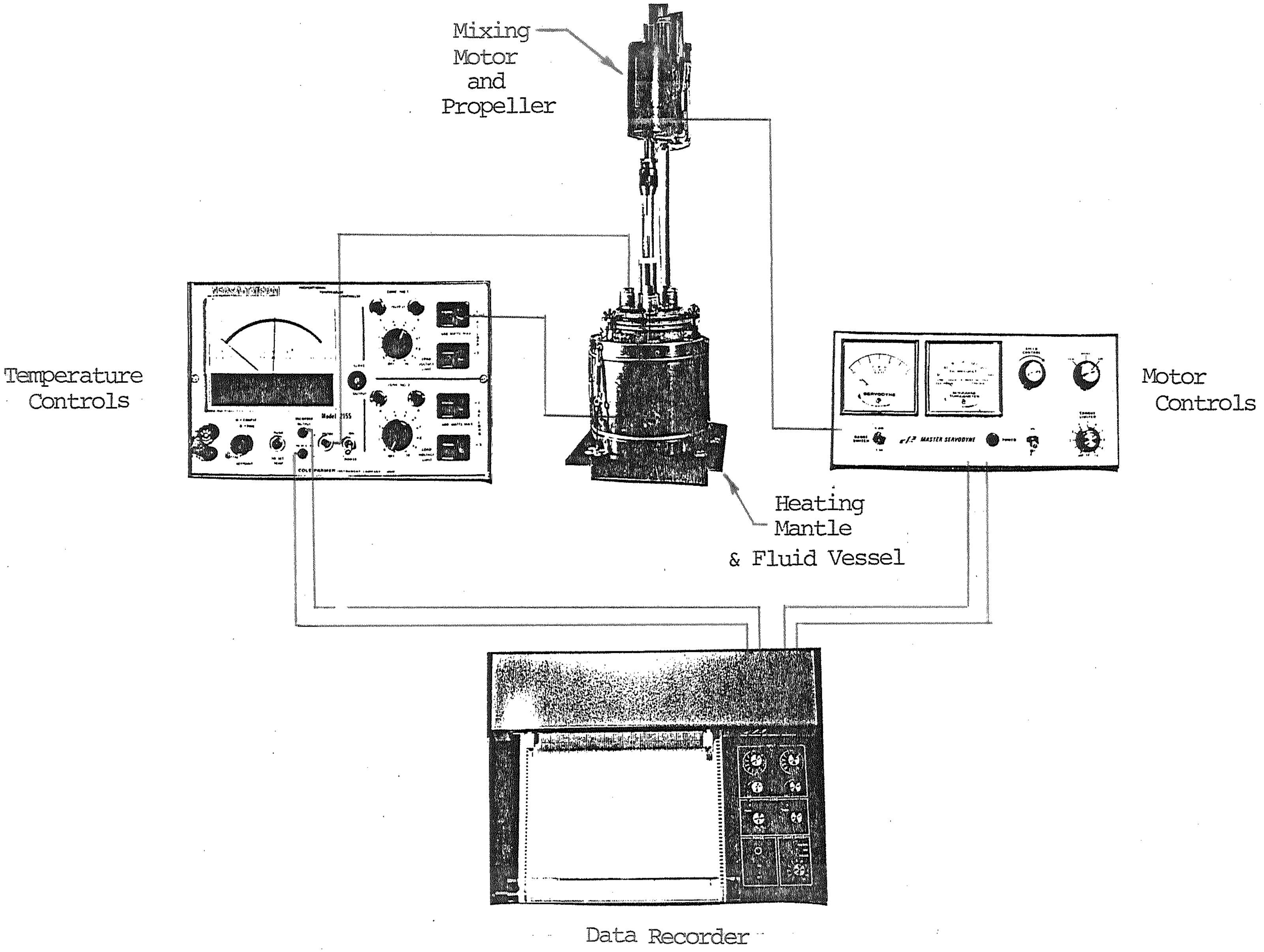

Figure A-1 Schematic Diagram of Arizona Torque-Fork Components

Table A-1

Equipment Required for Manufacture of the Arizona Torque-Fork

QUANTITY	DESCRIPTION	SUPPLIER OR MANUFACTURER ADDRESS	ORDERING INFORMATION
1	2-Channel Chart Recorder (Cole Parmer) State #61062, Serial #31778, Model #385	Cole Parmer Co. 7425 N. Oak Park Ave. Chicago, Ill. 60648	(800) 323-4340
1	Versatherm Proportional Temperature Controller Model 2155	Cole Parmer Co.	(800) 323-4340
1	Master Servodyne Control Unit State #057612, Model #4445-30	Cole Parmer Co.	(800) 323-4340
1	Electrocraft Motomatic Motor Generator, Serial #065000-067	Electrocraft Corp. 1600 Second St. S. Hopkins, Minn.	
1	Glas-Col Apparatus Co. Heating Mantle Serial #44677 Lab Glass #TM576	Glas-Col Apparatus Co. Terre Haute, Ind.	Cal Glass (714) 546-7250 Costa Mesa, Ca.
1	Stainless Steel Temperature Probe for Versatherm Model #2157-60	Cole Parmer Co.	(800) 323-4340
1	Recorder to Temperature Control Cable	Any two conductor cable	
1	Recorder to Servodyne Cable	Any two conductor cable	
2	2,000 ML Resin Reaction Flask Lab Glass #33710	Cal-Glass, Inc. 3012 Enterprise St. Costa Mesa, Ca.	(714) 546-7250
2	Cover for 2,000 ML Resin Reaction Flask LG #33720	Cal Glass	"
1	Set Cover Clamps for Resin Reaction Flask LG #8070	Cal Glass	"
2	10.5" Long Jiffy Mixer Model HS	Jiffy Mixer Co. 17981 Sky Park Crl. Suite G Irvine, Ca.	(714) 557-1272
1	Drill Press Stand, ½" Drills Dayton Model 2Z041	Dayton Equip. Co.	
1	Drill Chuck, Albrecht Model 0-5/16 or 0-8		Adroit Supply Co. 620 W. Adams Phx., Az. (602) 259-1996
1	Mounting Hardware for attachment of drill press to lab bench		

Mixing Motor Control. The rotational velocity of the mixing motor is controlled by and may be adjusted by the Servodyne control unit. The Servodyne controls motor rotational velocity under varying loads by adjusting voltage input to the motor. Voltage output of the Servodyne is monitored by a voltmeter and provision is made for coupling to a data recorder to provide continuous readout. Additionally, the Servodyne has adjustments for limiting torque in both clockwise and counterclockwise directions and for braking.

Temperature Controller. The temperature controller used to maintain appropriate mixing temperature is of the porportional type with controls for two separate output channels and adjustments for temperature set point and output voltage level. Mixing temperature is monitored by an analog output and provision is made for coupling to a data recorder to provide continuous readout.

Data Recorder. Mixing temperature and Servodyne voltage output are simultaneously monitored and recorded during mixing using a dual channel strip chart recorder. The recorder is equipped with multiple voltage ranges for both channels and multiple chart speeds.

CALIBRATION PROCEDURE

Viscosity during mixing measurements are made with the Torque-Fork by comparing the Servodyne voltage output at a constant rotational velocity to voltage output when mixing standard calibration oils of known viscosity. Viscosity-temperature relationships for Cannon Instrument Company S30000 calibration oil determined using vacuum capillary viscometers are tabulated in Table A-2. This calibration oil was then mixed at 250, 500, and 750 RPM in the Torque-Fork at various temperatures and the Servodyne voltage output monitored.

Table A-3 contains Servodyne output data at 500 RPM for S30000 oil. Using regression analysis, the following relationship was developed from the capillary viscosity data:

$$\text{Viscosity, Poise} = 5.55 \times 10^{11} \ (\text{Temp, F})^{-4.7} \qquad \text{(A1)}$$

From the Servodyne output data, the following relationship was developed:

$$\text{Output, Mv} = 3.95 \times 10 \quad (\text{Temp, F})^{-3.13} \qquad \text{(A2)}$$

TABLE A-2

TEMPERATURE-VISCOSITY DATA FOR CANNON S30000 CALIBRATION OIL

Temperature, F	Absolute Viscosity, Poises
66	1138.0
77	710.8
100	239.1
104	200.3
122	94.9
140	48.0
212	5.4

Linear Regression Coefficients:
$r = 1.0$
$a = 5.55 \times 10^{11}$
$b = -4.7$

$$\text{Viscosity, Poise} = 5.55 \times 10^{11} (\text{Temp, F})^{-4.7}$$

Solving equation 2 for temperature yields:

$$\text{Temperature, F} = 523.8\ (\text{Output, Mv})^{-0.30} \qquad \text{(A3)}$$

By substituting equation 3 into 1, the following relationship is developed for the Torque-Fork when mixing at 500 RPM:

$$\text{Viscosity, Poise} = 0.09\ (\text{Output, Mv})^{1.41} \qquad \text{(A4)}$$

Equation 4, and viscosity-output relationships at mixing speeds of 250 and 750 RPM which were determined in a manner similar to that outlined above are plotted in Figure A-2.

TABLE A-3

TORQUE-FORK VOLTAGE OUTPUT AT 500 RPM FOR CANNON S30000 CALIBRATION OIL

Temperature, F	Servodyne Output, Mv			
	Trial 1	Trial 2	Trial 3	Trial 4
110	-	159	-	-
120	-	117	114	-
130	-	81	-	-
140	104	60	102	80
150	79	47	72	-
160	61	39	-	-
170	45	31	43	-
180	31	27	33	33
190	27	23	27	-
200	23	21	-	-
210	21	19	19	20
220	19	18	-	18
230	18	-	-	17
240	16	16	-	16
250	-	15	-	-

Linear Regression Coefficients: $r = 0.95$

$a = 3.95 \times 10^{8}$

$b = -3.13$

$$\text{Output, m.v.} = 3.95 \times 10 \quad (\text{Temp, } {}^{\circ}\text{F})^{-3.13}$$

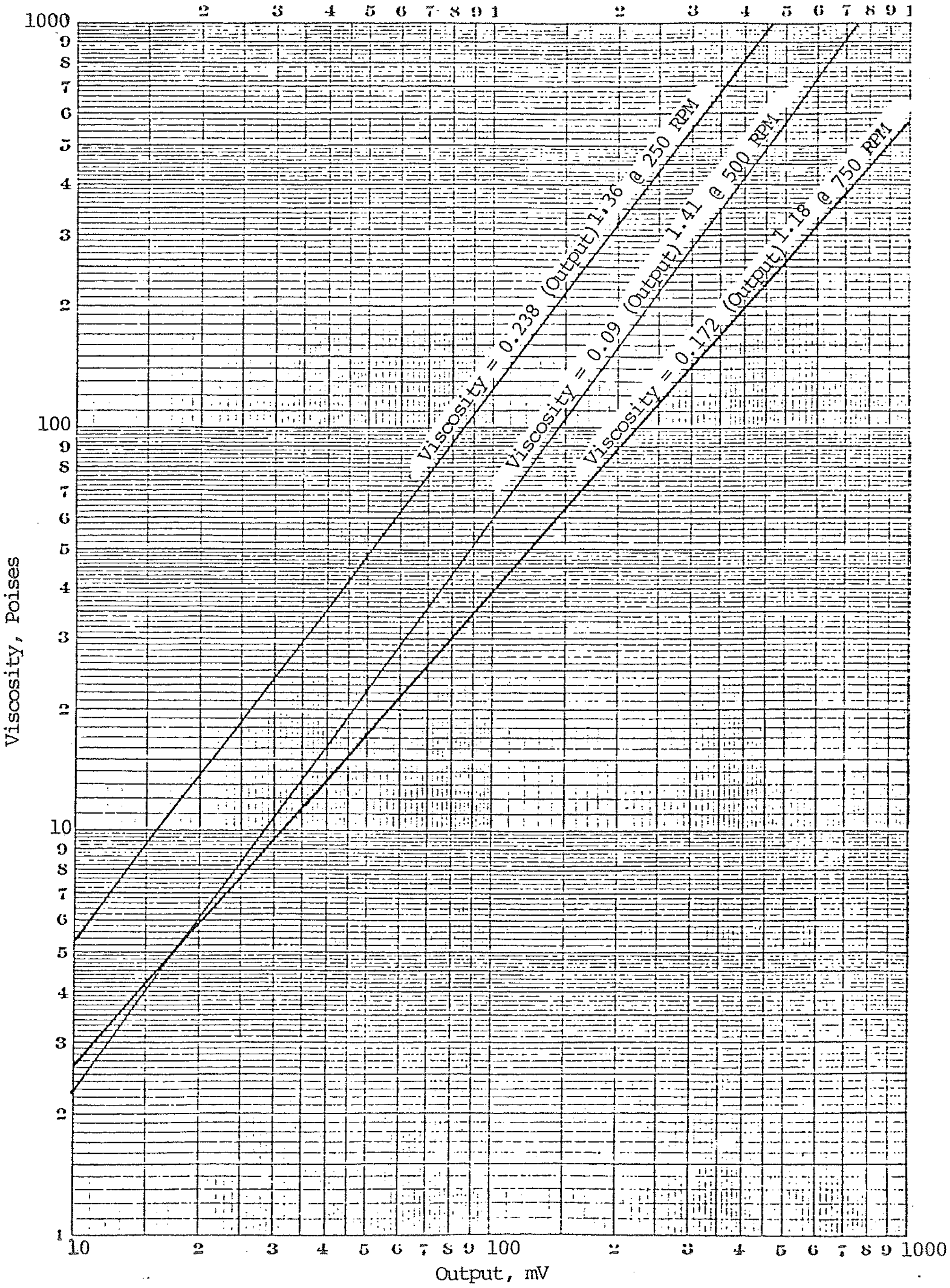

Figure A-2

Viscosity-Output Relationship for
Cannon Instrument Co S30000
Calibration Oil Using the Arizona Torque-Fork

APPENDIX B

Method of Test for

LOW TEMPERATURE FRACTURE OF PAVING AND ROOFING MATERIALS BY CONTROLLED DEFLECTION

Method of Test for

LOW TEMPERATURE FRACTURE OF PAVING AND ROOFING MATERIALS BY CONTROLLED DEFLECTION *

SCOPE

1. This test method describes two procedures for determining a temperature at which plastic and elastomer binder materials, such as asphalt and asphalt-rubber, used for paving and roofing purposes will fracture under specified deflection conditions.

SIGNIFICANCE

2. These methods establish the respective temperatures at which 50 percent of the specimens tested would be expected to fail when subjected to the conditions herein specified. The bending strains under which these materials are subjected in the tests are much more severe than will be experienced in field service. Although the magnitude of the field service deflections are much less, they are subjected to multiple deflection repetitions under much lower strains. The test methods thus are intended to compensate for field service conditions and provide for a short cut procedure for comparative evaluation of plastic and elastomer paving and roofing materials. Resistance to bending fracture at low temperatures is an important factor in the service life of paving and roofing materials.

DEFINITION

3. Low Temperature Fracture: That temperature, estimated statistically, at which 50 percent of the specimens would fail by exhibiting cracking in a specified test.

METHOD A

4. This method is designed to produce fractures at lower temperatures than Method B. It is a deflection test, at a specific temperature, involving a 13-1/2° bend of the specimen over a 12 inch mandrel.

* Note: Procedure developed by Charles McDonald.

APPARATUS

5. (a) Specimen Fabricating Jig: The jig shall be designed to fabricate two or more specimens at the same time. The specimens shall be 16 inches long by 1 inch wide by 1/8 inch thick. Welding rods, 1/8 inch x 18 inch, postitioned over strips of nonabsorbent paper can be used.

(b) Deflection Device: This arrangement shall provide for centering the specimens over a 12 inch cylindrical mandrel and permitting a deflection of 1-1/2 inches at a distance of 6-1/4 inches each side of the mandrel, thus producing a 13-1/2^{o} deflection from a tangent to the mandrel, of two or more specimens simultaneously.

TEST SPECIMENS

6. The specimens shall be fabricated by placing the strips of nonabsorbent paper on the jig and positoning the welding rods over the paper firmly in the jig. The 1 inch space between the rods shall be filled with the specimen materials for a distance of approximately 8 inches away from the center, making a specimen 16 inches long. The material shall then be struck off level with the top of the rods, forming a specimen 1/8 inch thickness.

CONDITIONING

7. Test specimens shall be refrigerated at the starting temperature, and at each succeeding temperature, for not less than one hour prior to test. The starting temperature should be high enough that the specimens are reasonably certain to pass without failure.

PROCEDURE

8. (a) After conditioning, the specimen shall be quickly withdrawn from refrigeration and instantly and vigorously deflected over the mandrel to the stops on either side.

(b) Examine each specimen carefully after each test, and while the specimen is still in the fully deflected position, for cracks. Note and record the kind and extent of any visible crack and the identificaton of the specimen.

(c) Test a minimum of two specimens. In the event that all of the specimens fail or not fail, increase or decrease the temperature of the test by approximately 3°C (approximately 5-1/2°F) and repeat the test. Any one specimen may be used for repeated tests only until it develops a crack visible to the naked eye. This constitutes a specimen failure. However, the degree of failure, such as multiple fractures, should also be noted as an aid in product evaluation. Test all specimens to failure.

REPORT

9. The report shall include the following:

(1) The temperature at which the first visible crack occured in each specimen.

(2) The average of the above individual temperatures for each specimen (obvious anomalyies shall be re- jected from the average). This is the failure temperature for material under test.

(3) Brief description of the type of cracking in each specimen -- for example "2 transverse and 1 longitudinal".

(4) Complete identification of the material including type, source, manufacturer's name and code, and formula if available.

(5) Reference to test method.

(6) Data of test.

(7) Age of material in specimens.

(8) Number of specimens tested.

(9) Name and location of laboratory performing the test.

(10) Any treatment to which the material has been subjected, in addition to the basic formulation, that could affect the physical properties (example: prolonged heating).

METHOD B

10. This method is designed to produce fracture in a higher temperature range than Method A. It is an inverted deflecton test, at a specific temperature, involving a 9° 28' bend tangent to a 1/8 inch diameter mandrel. Slight, but insignificant impact is also present, amounting to only approximately 2.25 feet per second (1-1/2 miles per hour).

APPARATUS

11. (a) Specimen Fabricating Jig: The jig shall be designed similar to that in Method A except that it shall accommodate four or more specimens 4-1/2 inches long by 1 inch wide by 1/8 inch thick, and the base paper for each specimen consists of 3 inch x 5 inch index cards inserted beneath the 1/8 inch welding rods.

(b) Deflection Device: This arrangement shall provide for centering the specimen, index card side up, as a beam bridging a gap 1-1/2 inches wide by 1/8 inch deep. A mandrel, centered over the specimen "bridge", consisting of a striking edge, rounded to a 1/8 inch diameter, shall move relative to the specimen at a linear speed of 2.25 feet per second (1-1/2 miles per hour) at impact and during at least the following 1/8 inch of travel. In order to maintain this speed when testing a single specimen, a mechanically released freely falling mandrel weighing 1200 grams and released at a height 3 cm above the bottom of the gap beneath the specimen "bridge", has proven adequate. If multiple specimen testing or exceptionally rigid materials are involved, the acting forces would have to be increased accordingly to maintain the fracturing speed.

TEST SPECIMENS

12. Except for the shorter length of the specimens, which are only 4-1/2 inches long and centered on the index cards, they are prepared the same as in Method A.

CONDITIONING

13. Same as in Method A.

PROCEDURE

14. (a) This is the same as in Method A except that the specimen is placed index side card up and centered over the gap as a bridge and the mandrel is mechanically or electrically released to deflect the specimen downward at the center.

    (b) This is the same as in Method A.

    (c) This is the same as in Method A except that a minimum of four specimens are tested.

REPORT

15. Same as in Method A.