RAPID TEST METHODS OF BITUMINOUS MATERIALS

FINAL REPORT

BY

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Conducted by
LOUISIANA DEPARTMENT OF HIGHWAYS
Research and Development Section
In cooperation with
U. S. Department of Transportation
FEDERAL HIGHWAY ADMINISTRATION

TABLE OF CONTENTS

LIST OF TABLES*

* The following abbreviations have been made:
TFO; Thin Film Oven, AASHTO T179-74
RTFO; Rolling Thin Film Oven, ASTM 2872-70
RTF-C; Rolling Thin Film Circulating Oven, AASHTO T240-73

LIST OF FIGURES

ABSTRACT

The primary objective of this study was two-fold: First to verify for Louisiana asphalts the close agreement as found by others (Ref. 1-5) between the standard Thin Film Oven Test (AASHTO T 179-74) and the newly adopted Rolling Thin Film Oven Test (AASHTO T240-73); and second, to seek a correlation between the standard Ductility Test (AASHTO T51-74) and a Micro-Ductility Test (Ref. 1 and Appendix).

The oven comparison was made by testing numerous asphalts for such residual properties as viscosity, penetration, and ductility following exposure in both ovens. The data indicate that, except for very high viscosity residual asphalts, the tests are in very good agreement and could be used interchangeably.

A correlation between the standard ductility test and the micro-ductility test is presented, which suggests that a reasonable relationship does exist. However, due to the nature of the resulting correlation, no unique micro-ductility value corresponding to a 100+ ductility value (current Louisiana acceptance specification) can be chosen. Therefore, this test should not be substituted for the well accepted and nationally used ductility test.

INTRODUCTION

The Louisiana Department of Highways bases its acceptance of asphalt cements to be used for highway construction upon compliance to the State Standard Specifications, which in turn are based upon several AASHTO test procedures. Two of the presently specified test methods (T179-74, "Thin Film Oven Test" and T51-74, "Ductility of Bituminous Materials") are time consuming. The TFO test requires 5 hours for completion and the Ductility Test approximately 3 hours.

There exist two rapid test methods that might be substituted for these AASHTO test procedures. These rapid methods are the Rolling Thin Film Oven Test (AASHTO T240-73) and the Micro-Ductility Test (California No. 349). The major advantage of these test methods over those presently specified is the time required to complete the test. The RTFO test requires 85 minutes and the Micro-Ductility Test requires approximately 40 minutes.

PURPOSE OF STUDY

This research was initially undertaken to evaluate, correlate and verify two rapid test methods (ASTM 2872-70 and California No. 349) to the presently specified methods (AASHTO T179-74 and AASHTO T51-74) in an endeavor to shorten the time required to ascertain whether the various asphalt cements submitted to the Louisiana Department of Highways conform to its acceptance specifications.

SCOPE

The study consisted of two phases.

Phase I was the verification of the Thin Film Oven Test (TFO) to the Rolling Thin Film Oven Test (RTFO).* It involved testing four different grades of asphalt cements from each of five different sources for residual properties following both types of oven exposure. The asphalt residuals were tested for viscosity at 140^{O}F (60^{O}C) and penetration, ductility, and micro-ductility at 77^{O}F (25^{O}C).

Phase II involved the development of a correlation between the standard ductility test and the micro-ductility test.

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* The original Rolling Thin Film Oven Test procedure was given as ASTM 2872-70. This procedure has been since modified by converting that oven from a convection oven to a forced draft oven; this modified oven is identified as the Rolling Thin Film Circulating Oven (RTF-C) in AASHTO T240-73. This research contains results from both of these procedures.

METHODOLOGY

For this study, the necessary test equipment (i.e. one rolling thin film oven, one modification kit to convert the original oven (RTFO) to a circulating oven (RTF-C), and one micro-ductility machine) was obtained from Cox and Sons, Colfax, California. The specifications for the oven and the corresponding procedure for its use are as given by AASHTO T240-73; a copy of the test method used for micro-ductility determinations, along with the appropriate equipment as manufactured by Cox and Sons, is given in the Appendix.

In the spring of 1972, the Research and Development Section of the Louisiana Department of Highways made initial preparation for both phases of this study by securing asphalt cements from five different local sources. Each source supply was comprised of three penetration grade asphalts;* in addition, one source also supplied a viscosity graded asphalt. The original properties of these sixteen asphalts are given in Table I. A sufficient quantity of material was taken during the heating of each of these sixteen samples so that the required tests on the residual (viscosity, penetration, ductility, and micro-ductility) from both oven exposures could be performed. The verification of one oven exposure to the other would be based upon the correlation of these residual test results. Table I contains the results of this initial comparison (RTFO versus TFO). In addition to gathering the data sought for oven comparisons, this residual testing also yielded limited data for the ductility and micro-ductility correlation.

It was felt that due to the large number of 150+ cm. ductility values attained after this initial oven comparison that a second oven exposure

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* In 1972, the Louisiana Department of Highways had not officially adopted its present viscosity grading specifications for paving grade asphalt cements.

of the various asphalts would be benefical in serving two purposes: (1) it might reduce the ductility values below 150+, thereby adding data for the ductility and micro-ductility correlation and (2) it would provide more data for the oven comparisons. The results of these reruns are included as Table II.

TABLE I

INITIAL COMPARISON OF TFO AND RTFO

Asphalt	ORIGINAL ASPHALT				T F O RESIDUE				R T F O RESIDUE			
	Pen. @ 77 F. 0.1 mm.	Vis. @ 140 F Poises	Duct. @ 77 F. cm.	Micro-Duct. @ 77 F. mm.	Pen. @ 77 F. 0.1 mm.	Vis. @ 140 F Poises	Duct. @ 77 F. cm.	Micro-Duct. @ 77 F. mm.	Pen. @ 77 F. 0.1 mm.	Vis. @ 140 F. Poises	Duct. @ 77 F. cm.	Micro-Duct. @ 77 F. mm.
A	54	4050	150+	76.8	37	7046	150+	77.1	37	8405	150+	85.2
B	61	3676	150+	45.0	45	7658	86	30.6	44	12409	150+	29.1
C	63	5188	150+	38.2	45	9657	108	20.2	45	14294	50	19.6
D	63	4449	150+	33.6	42	9721	95	22.5	42	13736	85	21.0
E	65	3980	150+	33.8	50	5970	76	29.9	50	7032	131	30.6
F	65	4073	150+	40.1	42	9484	113	24.4	42	12118	121	27.2
G	85	2620	150+	36.2	56	8997	126	23.6	52	11786	98	23.3
H	87	2492	150+	48.9	59	4521	150+	47.2	57	5948	150+	42.5
I	86	1936	150+	38.3	66	2684	150+	40.8	61	3391	150+	40.8
J	98	2026	150+	33.6	58	4099	150+	42.6	57	5348	150+	41.3
K	84	2020	150+	40.1	55	3711	150+	37.4	55	5172	150+	38.4
L	147	762	115	26.9	93	1598	150+	30.3	93	1711	150+	29.2
M	154	831	150+	21.5	89	2141	113	18.7	84	2611	119	18.5
N	156	832	131	28.4	105	1406	150+	37.6	106	1465	150+	40.4
O	155	760	102	22.2	126	956	150+	29.3	119	931	150+	31.3
P	151	721	114	27.6	101	1333	150+	41.0	99	1406	150+	43.2

TABLE II

RERUN OF DUCTILITIES AND MICRO-DUCTILITIES

Asphalts	ORIGINAL		T F O		R T F O	
	Ductility cm.	Micro-Duct. mm.	Ductility cm.	Micro-Duct. mm.	Ductility cm.	Micro-Duct. mm.
A*	150+	76.8	150+	77.1	150+	85.2
A Rerun		70.4	150+	74.5	150+	77.4
B*	150+	45.0	86	30.6	150+	29.1
B Rerun		44.2	105	28.1	65	27.2
C*	150+	38.2	108	20.2	50	19.6
C Rerun		39.7	104	20.6	62	17.4
D*	150+	33.6	95	22.5	85	21.0
D Rerun		38.9	116	19.0	52	20.1
E*	150+	33.8	76	29.9	131	30.6
E Rerun		36.0	---	30.6	---	29.5
F*	150+	40.1	113	24.4	121	27.2
F Rerun		38.3	135	24.7	127	23.8
G*	150+	36.2	126	23.6	98	23.3
G Rerun		34.7	116	23.5	112	21.1
H*	150+	48.9	150+	47.2	150+	42.5
H Rerun		43.4	150+	48.6	150+	46.3
I*	150+	38.3	150+	40.8	150+	40.8
I Rerun		37.0	150+	49.0	150+	40.0
J*	150+	33.6	150+	42.6	150+	41.3
J Rerun		37.9	150+	39.3	150+	40.6
K*	150+	40.1	150+	37.4	150+	38.4
K Rerun		33.2	150+	39.5	150+	31.6

* From Table 1

At this point, it became obvious that extended aging of some of the asphalts would be necessary if data at low ductility values were to be available for use in the correlation. Table III contains the results of this extended aging along with a remark on how the aging was accomplished.

TABLE III

AGED DUCTILITY VERSUS MICRO-DUCTILITY

ASPHALT	DUCTILITY @ 77 F. cm.	MICRO-DUCTILITY @ 77 F. mm.	REMARKS
A	13	7.9	Aged 3-3/4 hrs. in RTFO
D	78	18.4	" 1-1/2 " " "
F	52	13.7	" 1-1/2 " " "
G	40	12.2	" 2 " " "
G	13	6.9	" 3-3/4 " " "

The data collected and presented in the aforementioned Tables were thought to be sufficient to satisfy the scope of the project and, as such, data analysis was initiated. It soon became apparent that a definite bias existed for the viscosities of the RTFO residues; the RTFO was more severe than the TFO, especially with those asphalts having the highest residual viscosities. A specification check was made of both ovens, leading to a suspicion of the air supply for the RTFO; its flow rate and moisture content were both found to be excessive. A retest of the RTFO was thought to be in order, with particular attention being given to a water purge of the air supply prior to testing. The results of this effort are presented in Table IV.

Subsequent to the completion of all testing on the original RTFO, research by others (Ref. 5) lead to the modification of the oven by means of the installation of a squirrel cage blower on the top of the oven, which directs air from the bottom of the oven through a plenum chamber. This modified oven, the Rolling Thin Film Circulating Oven (RTF-C), was adopted by AASHTO in 1973 as test procedure T240. Thus an extension of the scope of this research was in order to evaluate this RTF-C oven. In addition to the modification of our original RTFO, air dryers were installed in the air supply line to eliminate any possible source of error due to wet air. Table V contains the results comparing the RTF-C oven with the standard TFO.

TABLE IV

RERUN OF RTFO RESIDUE

ASPHALT	PENETRATION @ 77 F. 0.1 mm.	VISCOSITY @ 140 F. Poises	DUCTILITY @ 77 F. cm.
A	34	8098	150+
B	43	11363	65
C	46	12689	62
D	40	12900	65
E	47	7251	106
F	42	12341	79
G	45	11666	74
H	59	4786	150+
I	63	3098	145
J	56	4674	150+
K	55	2948	150+
L	92	1853	150+
M	88	2287	98
N	99	1428	150+
O	121	928	150+
P	96	1304	150+

TABLE V

COMPARISON OF TFO AND RTF-C

CODE	TFO RESIDUE			RTF-C RESIDUE		
	Pen @ 77 F. 0.1 mm.	Vis. @ 140 F. Posies	Duct. @ 77 F. cm.	Pen @ 77 F. 0.1 mm.	Vis. @ 140 F. Posies	Duct. @ 77 F. cm.
D	42	14936	53	38	20265	82
E	50	7455	150+	47	8901	88
G	43	11377	36	42	15731	61
H	63	5015	150+	62	4987	150+
I	70	2688	150+	68	2578	150+
J	58	4804	150+	56	5252	150+
L	93	1776	150+	85	2184	150+
M	84	2977	86	83	3148	109
N	104	1621	150+	93	1667	150+
O	125	944	150+	113	1106	150+

DISCUSSION OF TEST RESULTS

Oven Comparisons

Figures 1 and 2 compare the viscosity and penetration of residuals for both oven exposures; RTFO and TFO. Penetrations are shown to be very nearly equal for both, whereas the viscosities show the RTFO to be a more severe test, especially for high viscosity residual asphalts. A comparison of Figures 1a and 2a indicates an improved correlation, with a lessening of bias, when the moisture content and rate of air supply to the RTFO are properly controlled. Figures 3a and 3b indicate a similar acceptable correlation between the RTF-C oven and the standard TFO for residual viscosities and penetrations. It is noted that the RTF-C oven, as was the RTFO, is slightly more severe for higher residual viscosity asphalts.

Ductility and Micro-Ductility Comparisons

Data from Table II, along with results on asphalts L, M, N, O, and P from Table I, are plotted in Figures 4a and 4b. A very good agreement is observed for micro-ductility between both oven exposures. Such is not the case between the standard ductility from both oven exposures. The points labeled A thru E in Figures 4a, 4b, and 5a are explained as follows: Figure 5a shows these points to be outlie points, caused by either an oven error, ductility error, or micro-ductility error. Figure 4b, in conjunction with the good correlation between ovens previously shown in Figures 1, 2, and 3, shows no reason to suspect either the oven test or the micro-ductility test as being the source of the bad data. However, Figure 4a would seem to indicate faulty ductility testing as being the cause of the outlie points. If such explanation is accepted and these outlie points are neglected, Figure 5b would represent a more reasonable correlation between the standard ductility test and the micro-ductility test. It suggests that a reasonable relationship does exist, although the data is certainly too limited at allow more than a range comparison; that is, a micro-ductility value in excess of 18 mm. could suggest a corresponding ductility in excess of 80 cm.

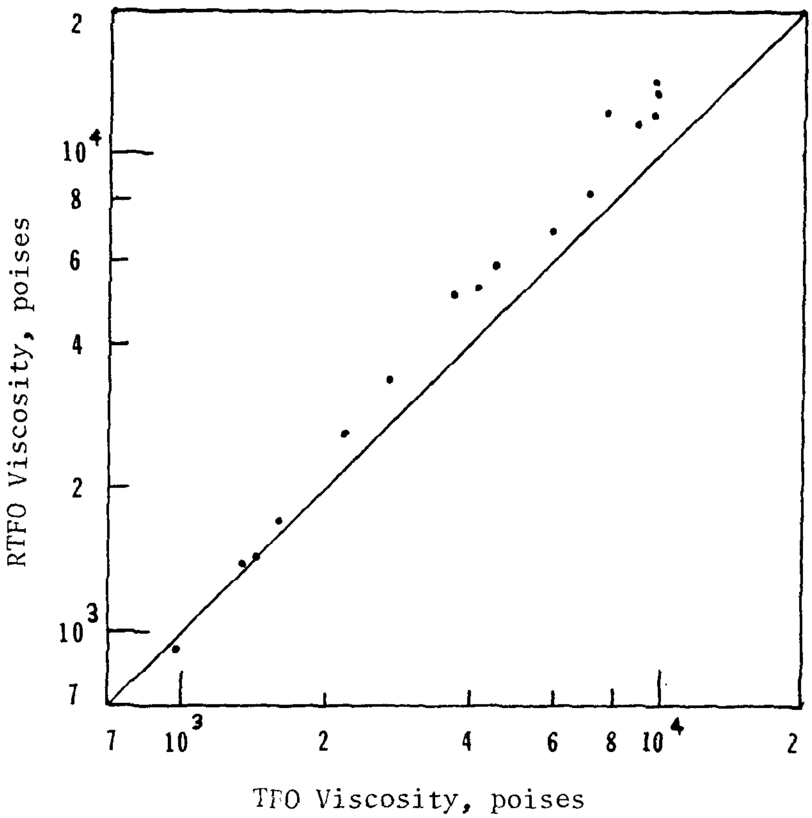

Figure 1a: Initial Comparison of TFO and RTFO, Viscosity at 140°F.(60C)

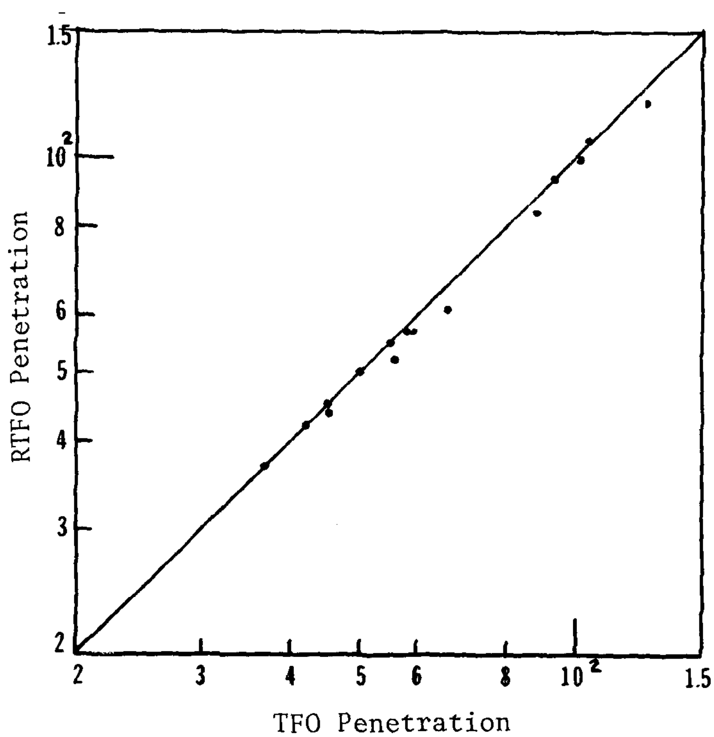

Figure 1b: Initial Comparison of TFO and RTFO, Penetration at 77°F.(25C)

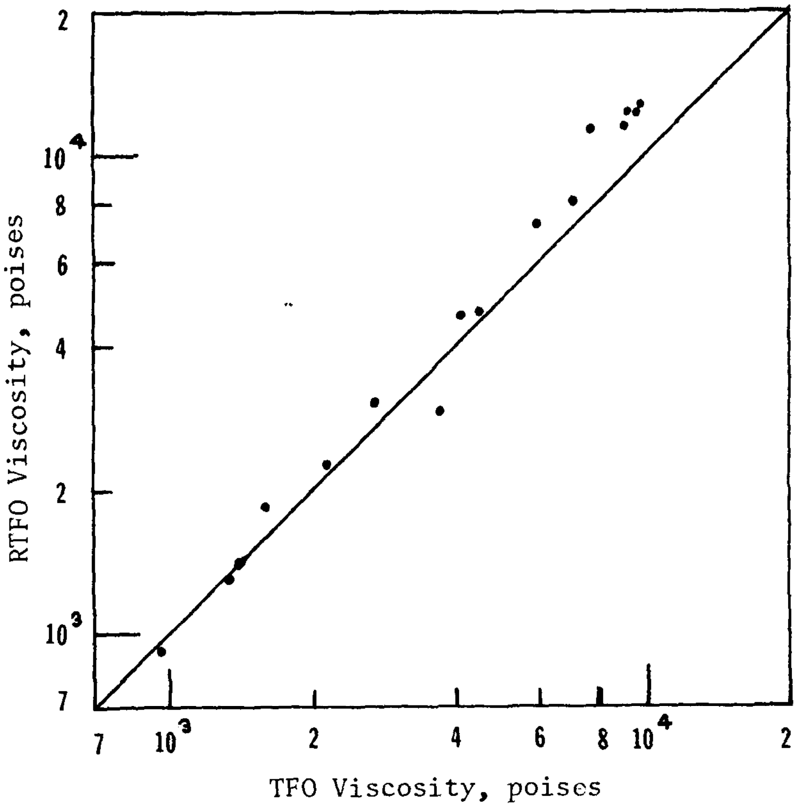

Figure 2a: Rerun Comparison of TFO and RTFO, Viscosity at 140°F.(60C)

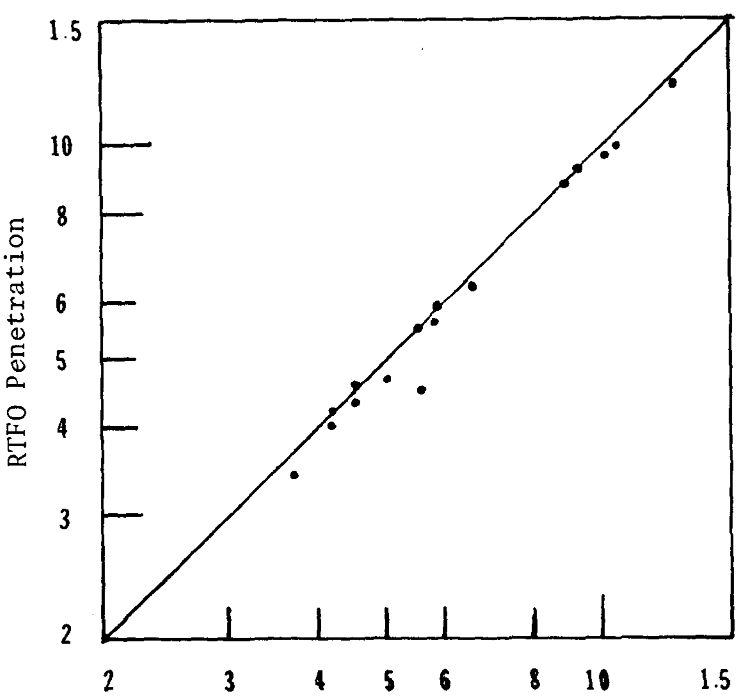

Figure 2b: Rerun Comparison of TFO and RTFO, Penetration at 77°F.(25C)

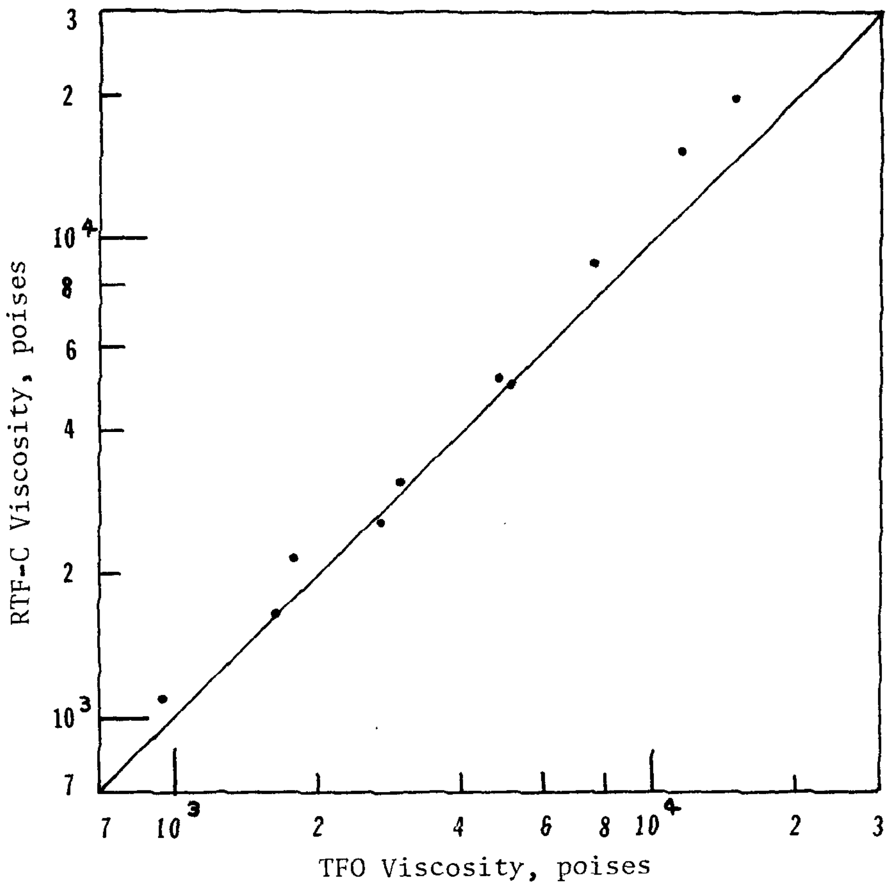

Figure 3a: Comparison of TFO and RTF-C, Viscosity at 140°F. (60C)

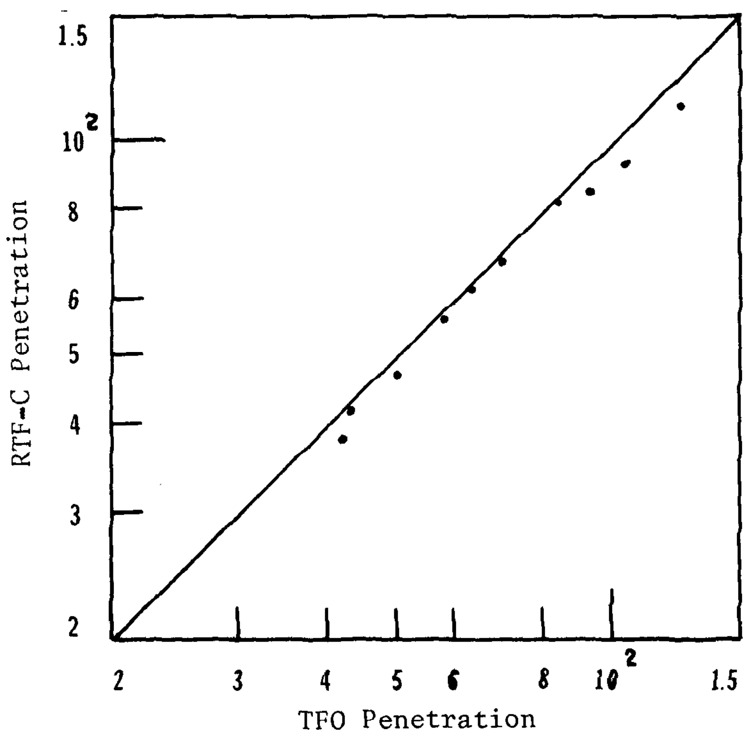

Figure 3b: Comparison of TFO and RTF-C, Penetration at 77°F. (25C)

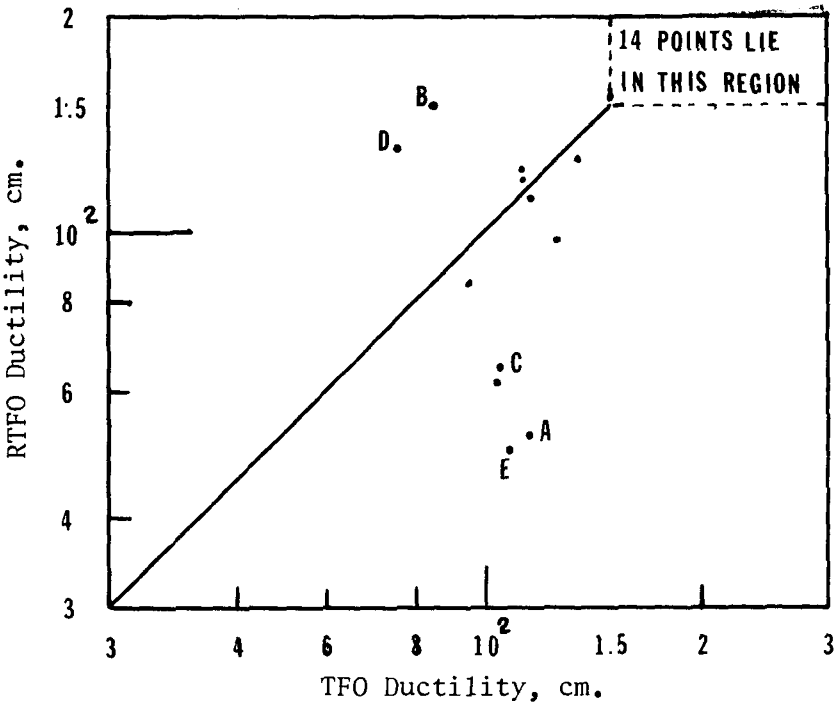

Figure 4a: Comparison of TFO and RTFO, Ductility at 77°F(25C)

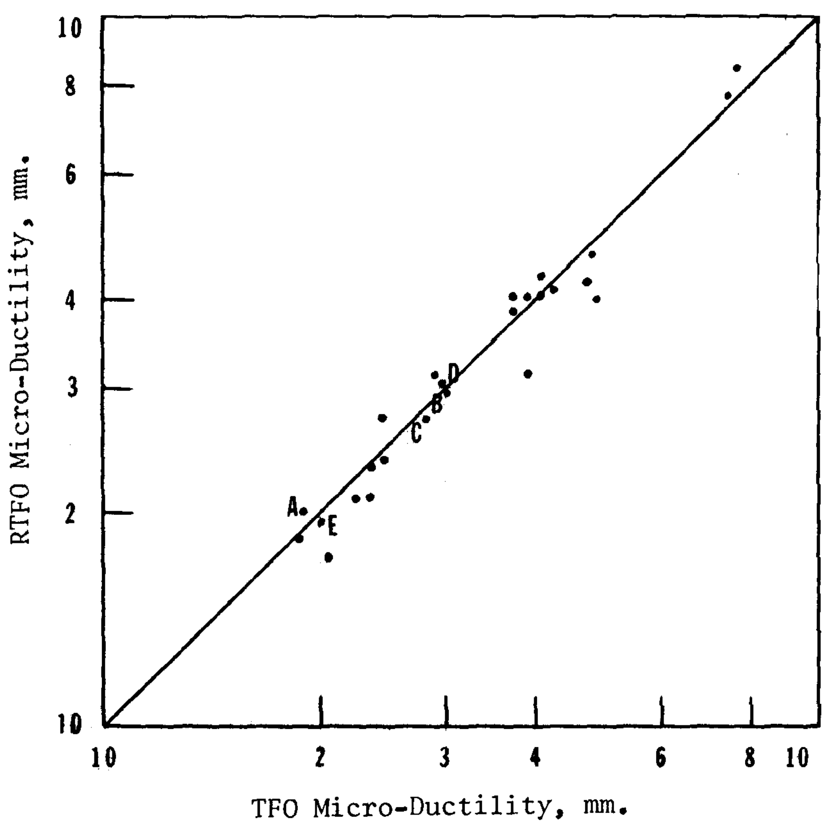

Figure 4b: Comparison of TFO and RTFO, Micro-Ductility at 77°F.(25C)

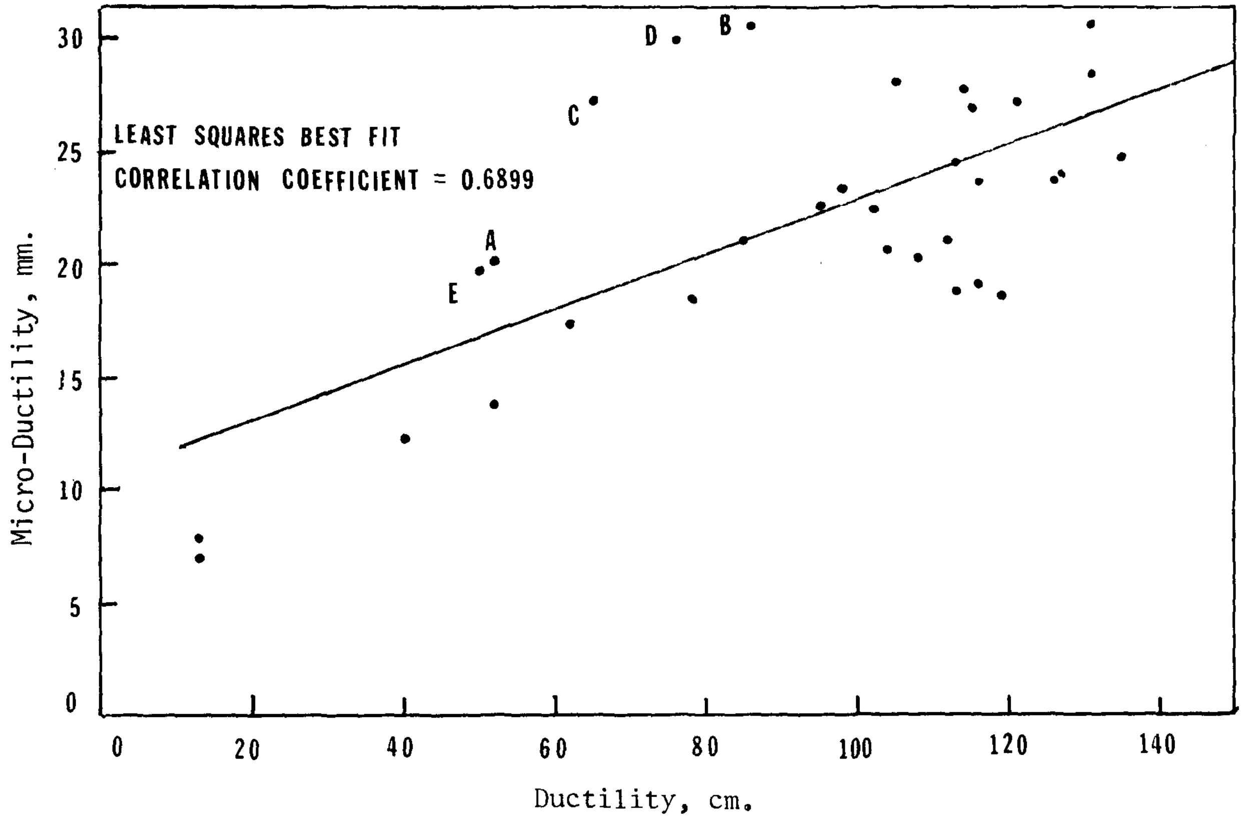

Figure 5a: Comparison of Ductility and Micro-Ductility, All Data

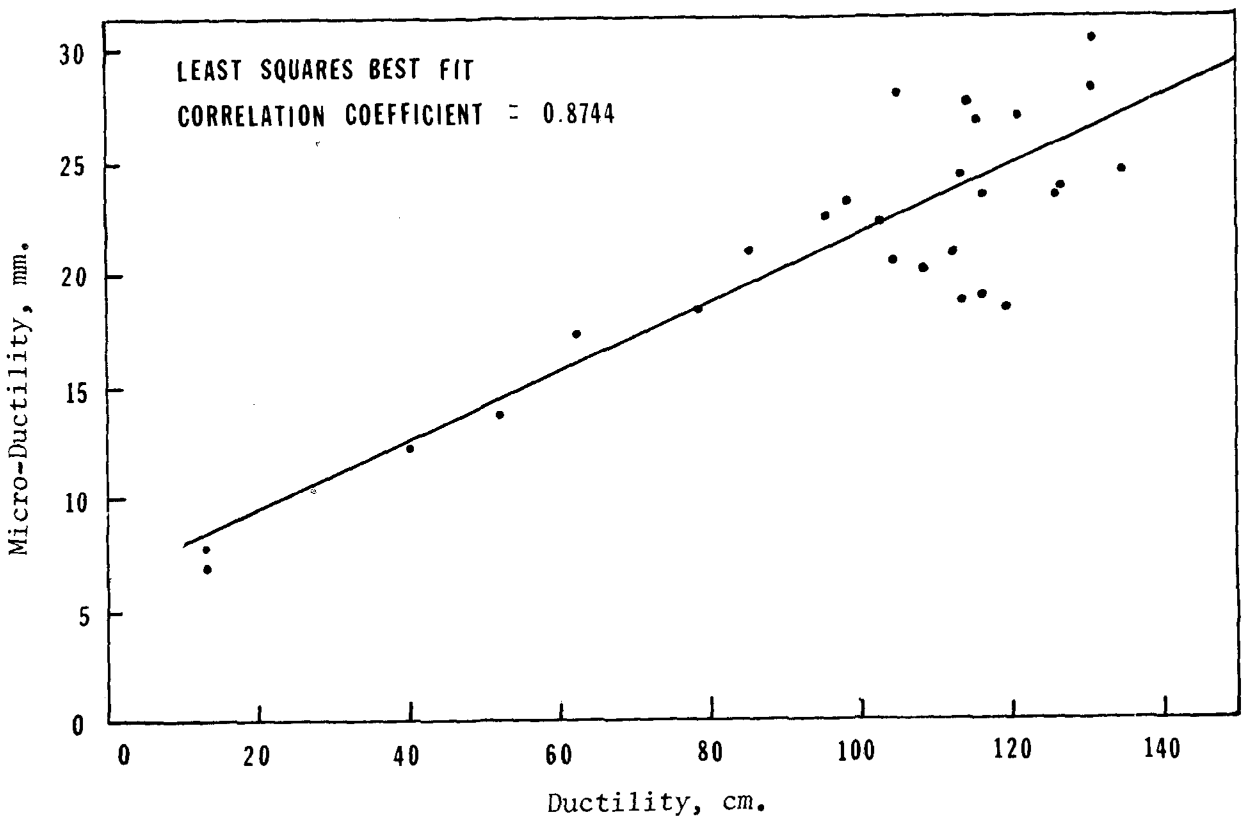

Figure 5b: Correlation of Ductility and Micro-Ductility, Select Data

CONCLUSIONS

The following conclusions are drawn from the data generated in this study and, as such, are confined to Louisiana grade asphalts. In addition, no statistical confirmation is intended.

(1) The Rolling Thin Film Oven (RTFO) and its modification, the Rolling Thin Film Circulating Oven (RTF-C), give exposures very close to the well-accepted TFO for low and medium viscosity residual asphalts. For such asphalts, the RTF-C oven test would be considered an acceptable replacement for the TFO test.

(2) A loss in correlation between the rapid oven exposures and the TFO exposure is apparent for high viscosity residual asphalts; both the RTFO and the RTF-C oven give exposures more severe than the TFO.

30 A reasonable correlation between the standard ductility test (AASHTO T51-74) and the micro-ductility test does exist for Louisiana asphalts. However, more comparison testing would be needed before a unique micro-ductility value could be associated with the department's current requirement of 100+ cm. ductility.

RECOMMENDATIONS

The following recommendations are made in light of the present needs of the Department and the results of this research effort:

(1) The presently specified TFO test procedure (AASHTO T179-74) should continue to be used in conjunction with the department's current acceptance specification for asphalt cements. No replacement of the TFO by the RTF-C oven is warranted due to the definite bias between ovens observed with those asphalts used extensively by Louisiana; namely, the high viscosity residual asphalts.

(2) The Rolling Thin Film Circulating Oven, with its rapid ability to simulate age-hardening of asphalt cements, should be maintained for research and evaluation use in asphalt durability studies.

(3) The presently specified ductility test (AASHTO T51-74) should continue as one of the department's acceptance tests for the sake of national conformance and recognition.

(4) The micro-ductility machine should be maintained for evaluation especially considering its ability to operate with small quantities of asphalt; such a characteristic would be a prime advantage in dealing with recovered asphalt from field cores.

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(3) Hveem, F. N.; Zube, E.; and Skog, J.; ASTM STP No. 277 (1959).

(4) Schmidt, R. J.; Painter, L. J.; Skog, J. B.; and Puzinauskas, V. P.; Proc. AAPT 37, 476 (1968).

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APPENDIX

Page

Test Method No. Calif. 349-A
November, 1962
(6 pages)

State of California
Department of Public Works
Division of Highways

MATERIALS AND RESEARCH DEPARTMENT

TENTATIVE METHOD OF TEST FOR MICRO-DUCTILITY OF BITUMINOUS MATERIALS

Scope

This test method provides a means of testing small quantities of bituminous materials for ductility.

A. Apparatus

1. Ductility machine, capable of pulling specimens at 1/2 cm/min. See Drawing No. D-507.

2. Ductility mold assembly - See Drawing No. B-507.

   a. Molds
   b. Mold Holders

3. Water bath with controls for regulating water temperature at 77±1F.

4. Metric scale with 1 mm divisions.

5. Hot plate.

6. Pick (made by flattening one end of a 4" length of welding rod). See Drawing No. B-507.

7. Magnifying Lamp - (a suitable lamp is the "Luxo Magnifying Lamp," Model LFM-1).

B. Preparation of Material

If the material to be tested is in a container it should be heated and stirred thoroughly before removing a test specimen.

C. Procedure

1. Place assembled molds (less plugs) on a hot plate previously adjusted to about 250 to 300 F surface temperature, a temperature that will liquify the asphalt to be tested. Do not heat plugs.

2. Secure about 0.05 grams of the material to be tested with the pick and transfer to the center of the large end of the plug, see Figure 1. Remove mold from the hot plate with a clamp, being careful that the halves remain tight together. Screw plug with asphalt into mold slowly, noting that the asphalt flows up through the orifice and fills the cone, see Figure 1.

3. Screw second plug into place. Do not tighten either plug excessively, to do so tends to separate the mold.

4. Let mold with clamp set in air at room temperature for 15 minutes minimum.

5. Place mold with clamp in water bath maintained at the desired testing temperature for at least 10 minutes before testing.

6. Transfer mold to ductility machine using clamp.

7. Slide the mold stem into the mold retainer (part No. 6 of Drawing D-507) so that the end of the stem is touching the right end piece, then slide the split nut (part No. 8 of D-507) till it is flush against the other stem end. Clamp the split nut, then slide the mold so that the stem goes into the hole in the split nut. Center the mold so that the knurled thumb screw may be screwed into the slots in the stems.

8. Turn the mold so that the slot will be in a convenient position to view the specimen at the start of the test and tighten the thumb screws.

9. Bring the magnifying lamp into position so that as the mold halves begin to separate, viewing of the asphalt thread is possible.

10. As mold halves start to separate force water with an eye dropper through the molds at the point of separation to force air bubbles off the test specimen. When asphalt thread breaks, shut off motor.

11. Remove apparatus from water bath and measure length of thread to nearest millimeter.

D. Calculation and Report

1. Determine the average value for the three micro-ductility results as specified below:

Group 1 - All three readings on any sample are within 0 through 30 mm. Average all three results for any sample, if the three readings are within a range of 4 mm. If greater than 4 mm, average the two highest results within this range, and discard the third reading. If any two of the three results are not within the 4 mm range, then the test shall be repeated.

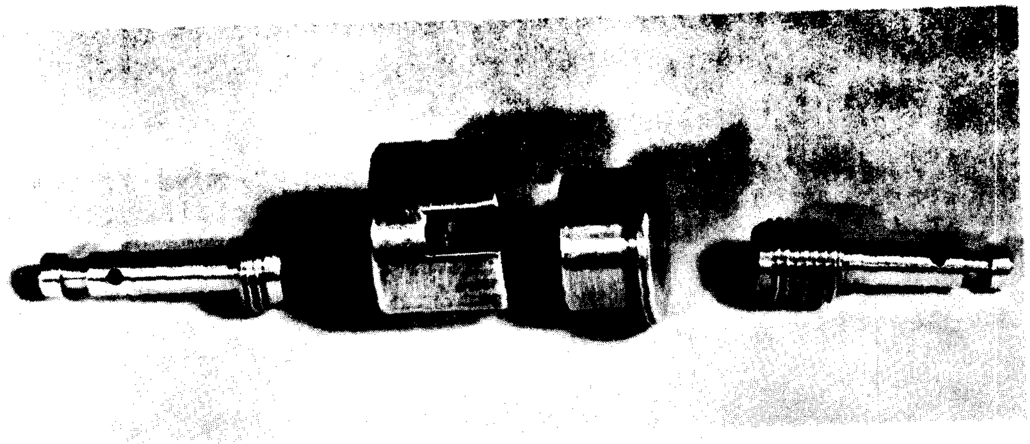
Group 2 - All readings on all samples are above 30+ mm Average the two readings that are nearest to each other and discard the third.

If one reading is above 30 mm and two readings are below, the requirements for Group 1 shall apply. If two readings are above 30 mm, and one below, the requirements for Group 2 shall apply.

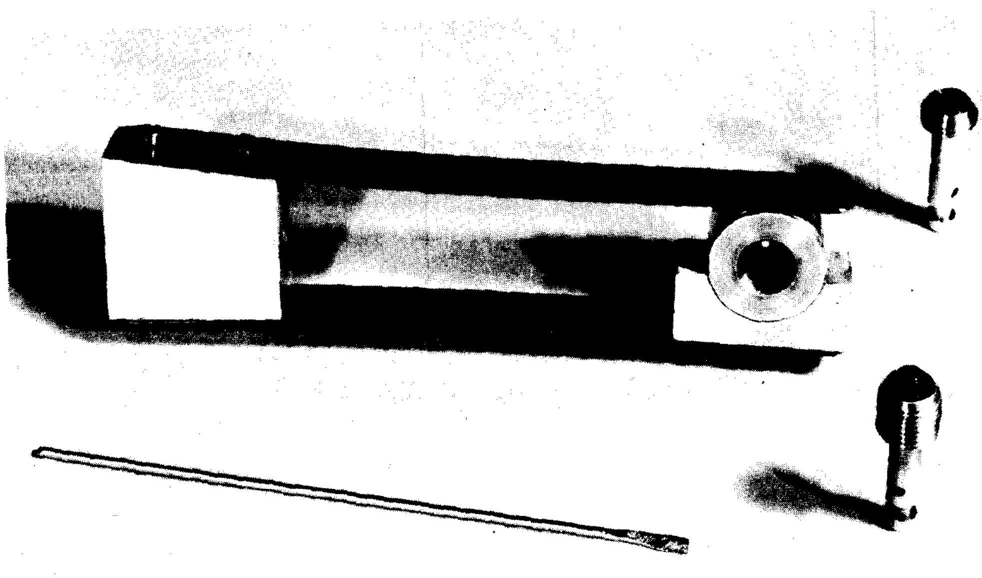
E. Notes

1. For cleaning, plugs may be unscrewed from molds easily by inserting a piece of 1/16" diameter welding rod through hole in stem to give leverage.

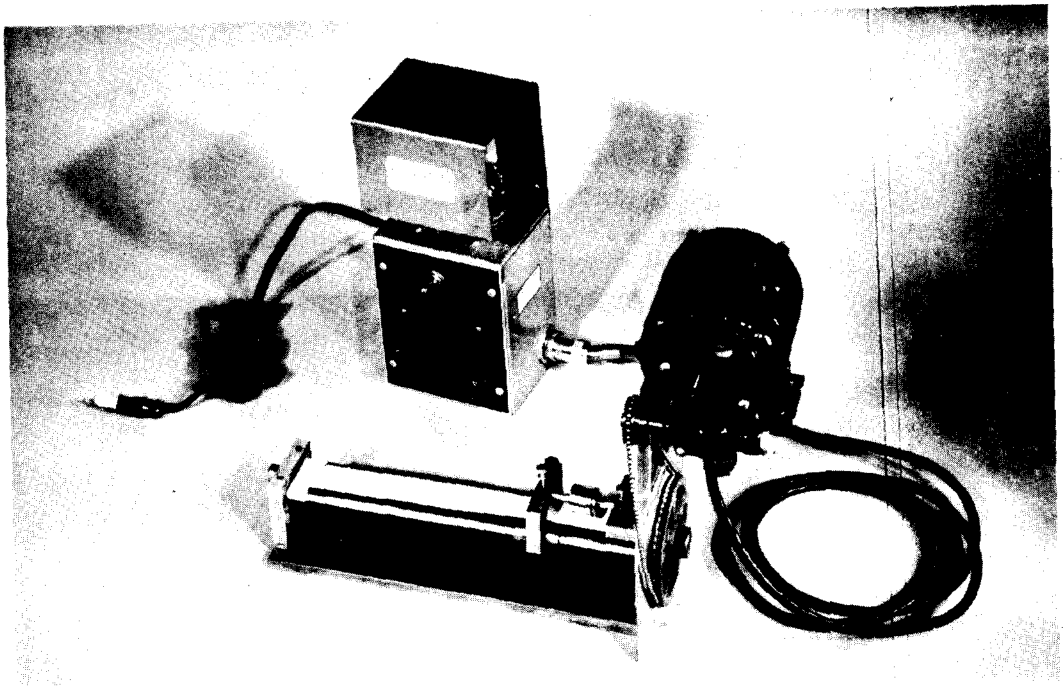
2. In handling molds with clamps be sure that both halves are securely gripped.



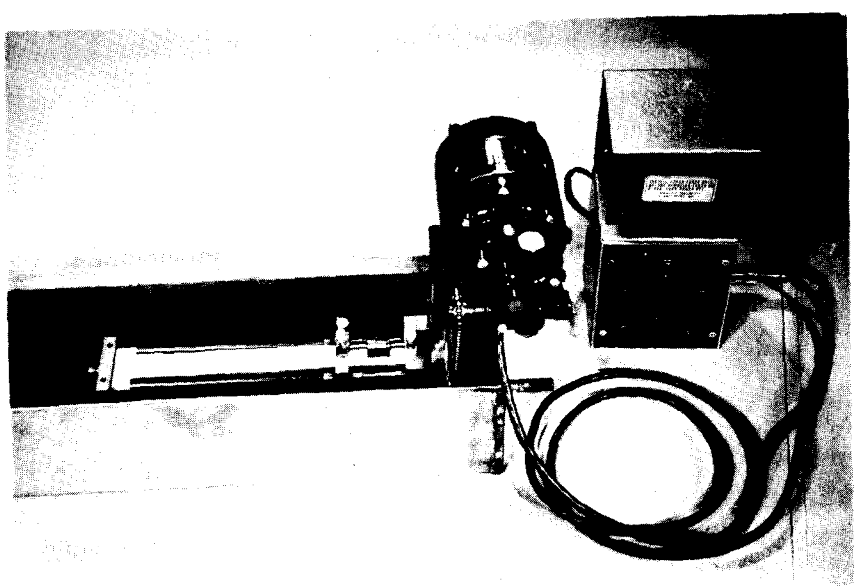
Ductility Mold and Plugs



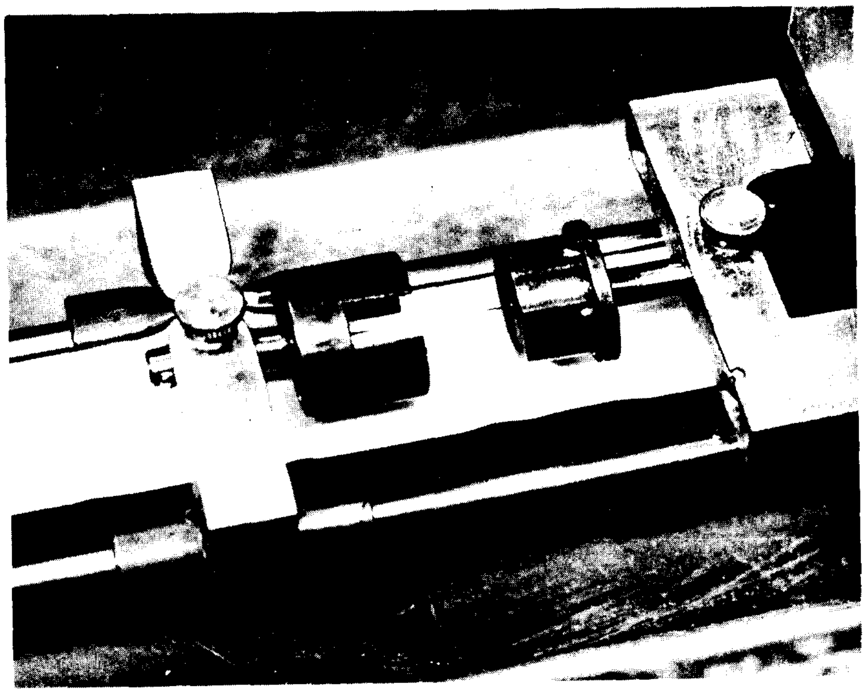
Ductility Mold
Asphalt for Specimen is on Plug in Foreground
Note Pick for Transfer of Asphalt to Plug



Ductility Test Apparatus



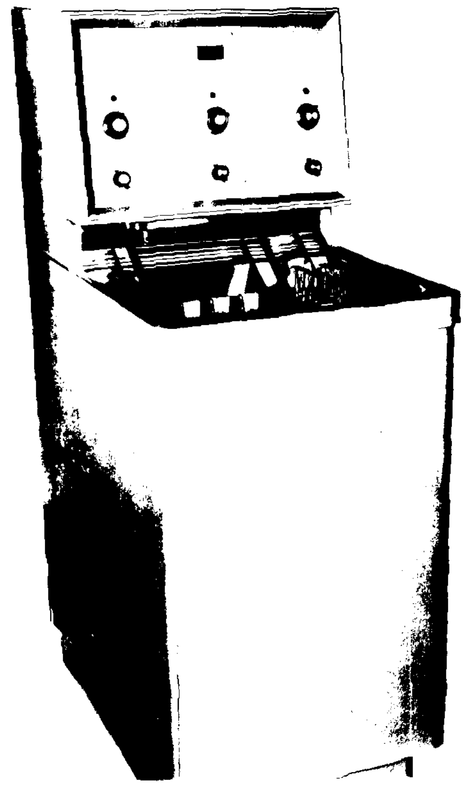
Ductility Test Apparatus
in Water Bath



Ductility Test in Operation
Note Asphalt Thread

Figure III

MICRO DUCTILITY EQUIPMENT AS SUPPLIED BY: JAMES COX AND SONS
P. O. BOX 674
COLFAX, CALIRORNIA 95713



TEST FOR MICRO DUCTILITY OF BITUMINOUS MATERIALS

Apparatus to conform to California Test Method 349

Ductility Instrument
To include:
1. Ductility Instrument with three mold capacity
2. Three channel digital readout
3. Bodine shunt wound DC motor
4. Solid state motor speed control
5. Six (6) mold holders
6. Six (6) mold assemblies

Console Water Bath with Integral Controls
To include:
1. Insulated stainless steel tank
2. Stainless steel isolation tank
3. Temperature Controller - CS 500
4. A 500 watt emersion heater
5. Cooling coil with standard tubing connections
6. Submersible agitator pump
7. Pump motor speed control - solid state
8. All controls to be enclosed and panel mounted
9. Instrument panel
   a) All functions engraved on a laminate phenolic plastic
10. Cabinet
    a) 14 Gage steel
    b) All welded construction
11. Finish
    a) Cabinet - Hammertone grey

Cox & Sons

TESTING EQUIPMENT
Business 925-6434
Residence 487-7992

CUSTOM INSTRUMENTS
2431 PENNLAND DRIVE
SACRAMENTO, CALIFORNIA 95825

DIRECTIONS FOR PLACING MOLDS IN DUCTILITY INSTRUMENT

1. Place sample in mold according to the State of California's instructions

2. While asphalt is still hot, twist mold assembly to align the 1/16 diameter holes located in stem (see Figure 1)

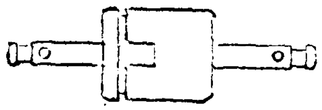


Figure 1

3. Clamp mold assembly in holder

4. Hang holders on rack provided in water bath

5. To warm up test rate control motor
   a) Place rate control switch in test position - allow motor to run until it reaches over travel switch (moveable arm will be in far left position)
   b) Switch to load position (moveable arm will stop when it reaches limit switch)

6. To load sample molds
   a) Insert male stem into moveable arm retaining block - insert stem to depth shown in Figure 2

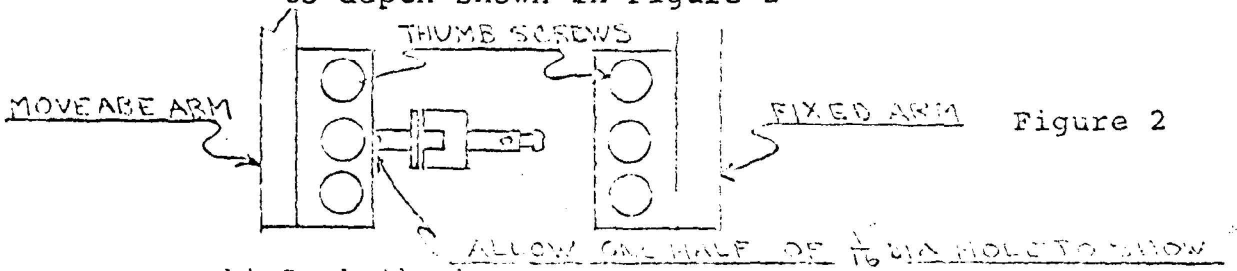


Figure 2

   b) Lock thumb screw
   c) HAZARD - Before placing switch in re-set position, make sure thumb screws located on fixed arm are in retracted position (A good safety proceedure would be to insert mold stem into the fixed arm retaining block before insertion into the moveable arm retaining block and locking of thumb screw)
   d) Switch to re-set position (moveable arm will stop when it reaches limit switch)
   e) Sample mold No. 1
      Switch to test position - watch shortest mold assembly - when half of the 1/16 diameter hole appears, switch to brake and lock thumb screw
   f) Zero counter
      Hazard - Do not zero counter when switch is in test, load, or re-set position

g) Sample mold No. 2
Switch to test position and proceed as in (e)
Note counter reading

H) Sample No. 3
Proceed the same as (g)

i) The above instructions prevent mold seperation by positioning the stem relief directly under the thumb screws and removes any accumulative back lash from feed screw assembly

j) Test in accordance with the State of California instructions

k) Subtract initial counter readings for samples 2 and 3 from their total breaking point readings