

Evaluation of the Laboratory Asphalt Stability Test

PUBLICATION NO. FHWA-HRT-04-111

FEBRUARY 2005



U.S. Department of Transportation
Federal Highway Administration

Research, Development, and Technology
Turner-Fairbank Highway Research Center
6300 Georgetown Pike
McLean, VA 22101-2296

FOREWARD

The Laboratory Asphalt Stability Test (LAST) was proposed by the researchers of the National Cooperative Highway Research Program (NCHRP) 9-10 program as a possible new method to evaluate the storage stability of modified asphalts. Federal Highway Administration (FHWA) was assigned the responsibility for evaluating the LAST device. This involved three tasks:

- Procuring a commercial version of the LAST device.
- Correcting perceived deficiencies in this device.
- Conducting a thorough evaluation of the test method.

The modified asphalts used in NCHRP 90-07 were investigated in this study. These included elastomers, plastomers, elasto-plastomers, and crumb rubber. The eight binders chosen all met the Superpave performance grading of 70-28 (PG70-28). This report details the work at FHWA's Turner-Fairbank Highway Research Center in evaluating the thermal stability of modified binders using the LAST device.

T. Paul Teng, P.E.
Director, Office of Infrastructure
Research and Development

Notice

This document is disseminated under the sponsorship of the U.S. Department of Transportation in the interest of information exchange. The U.S. Government assumes no liability for the use of the information contained in this document. This report does not constitute a standard, specification, or regulation.

The U.S. Government does not endorse products or manufacturers. Trademarks or manufacturers' names appear in this report only because they are considered essential to the objective of the document.

Quality Assurance Statement

The Federal Highway Administration (FHWA) provides high-quality information to serve Government, industry, and the public in a manner that promotes public understanding. Standards and policies are used to ensure and maximize the quality, objectivity, utility, and integrity of its information. FHWA periodically reviews quality issues and adjusts its programs and processes to ensure continuous quality improvement.

Technical Report Documentation Page

1. Report No. FHWA-HRT-04-111	2. Government Accession No.	3. Recipient's Catalog No.	
4. Title and Subtitle Evaluation of the Laboratory Asphalt Stability Test		5. Report Date February 2005	
		6. Performing Organization Code	
7. Author(s): J. Youtcheff, N. Wijayatilleke, and A. Shenoy		8. Performing Organization Report No.	
9. Performing Organization Name and Address Office of Infrastructure Research and Development Federal Highway Administration 6300 Georgetown Pike McLean, VA 22101-2296		10. Work Unit No.	
		11. Contract or Grant No. In House Report	
12. Sponsoring Agency Name and Address Office of Infrastructure Research and Development Federal Highway Administration 6300 Georgetown Pike McLean, VA 22101-2296		13. Type of Report and Period Covered Final—July 2003	
		14. Sponsoring Agency Code	
15. Supplementary Notes FHWA contact: Jack Youtcheff, HRDI-11			
16. Abstract The Laboratory Asphalt Stability Test (LAST) was proposed by the researchers of the National Cooperative Highway Research Program (NCHRP) 9-10 program as a possible new method to evaluate the storage stability of modified asphalts. The test involved exposing the binders with and without mechanical agitation at elevated temperatures for extended lengths of time to assess the thermal stability of the binders. The Federal Highway Administration (FHWA) was assigned the responsibility for evaluating the LAST. The task involved (a) procuring a commercial version of the LAST, (b) correcting perceived deficiencies, and (c) evaluating the test method thoroughly.			
17. Key Words Storage stability, LAST, test method, polymer-modified asphalt binders		18. Distribution Statement No restrictions. This document is available to the public through the National Technical Information Service, Springfield, VA 22161	
19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No of Pages 50	22. Price

SI* (MODERN METRIC) CONVERSION FACTORS

APPROXIMATE CONVERSIONS TO SI UNITS

Symbol	When You Know	Multiply By	To Find	Symbol
LENGTH				
in	inches	25.4	millimeters	mm
ft	feet	0.305	meters	m
yd	yards	0.914	meters	m
mi	miles	1.61	kilometers	km
AREA				
in ²	square inches	645.2	square millimeters	mm ²
ft ²	square feet	0.093	square meters	m ²
yd ²	square yard	0.836	square meters	m ²
ac	acres	0.405	hectares	ha
mi ²	square miles	2.59	square kilometers	km ²
VOLUME				
fl oz	fluid ounces	29.57	milliliters	mL
gal	gallons	3.785	liters	L
ft ³	cubic feet	0.028	cubic meters	m ³
yd ³	cubic yards	0.765	cubic meters	m ³
NOTE: volumes greater than 1000 L shall be shown in m ³				
MASS				
oz	ounces	28.35	grams	g
lb	pounds	0.454	kilograms	kg
T	short tons (2000 lb)	0.907	megagrams (or "metric ton")	Mg (or "t")
TEMPERATURE (exact degrees)				
°F	Fahrenheit	5 (F-32)/9 or (F-32)/1.8	Celsius	°C
ILLUMINATION				
fc	foot-candles	10.76	lux	lx
fl	foot-Lamberts	3.426	candela/m ²	cd/m ²
FORCE and PRESSURE or STRESS				
lbf	poundforce	4.45	newtons	N
lbf/in ²	poundforce per square inch	6.89	kilopascals	kPa

APPROXIMATE CONVERSIONS FROM SI UNITS

Symbol	When You Know	Multiply By	To Find	Symbol
LENGTH				
mm	millimeters	0.039	inches	in
m	meters	3.28	feet	ft
m	meters	1.09	yards	yd
km	kilometers	0.621	miles	mi
AREA				
mm ²	square millimeters	0.0016	square inches	in ²
m ²	square meters	10.764	square feet	ft ²
m ²	square meters	1.195	square yards	yd ²
ha	hectares	2.47	acres	ac
km ²	square kilometers	0.386	square miles	mi ²
VOLUME				
mL	milliliters	0.034	fluid ounces	fl oz
L	liters	0.264	gallons	gal
m ³	cubic meters	35.314	cubic feet	ft ³
m ³	cubic meters	1.307	cubic yards	yd ³
MASS				
g	grams	0.035	ounces	oz
kg	kilograms	2.202	pounds	lb
Mg (or "t")	megagrams (or "metric ton")	1.103	short tons (2000 lb)	T
TEMPERATURE (exact degrees)				
°C	Celsius	1.8C+32	Fahrenheit	°F
ILLUMINATION				
lx	lux	0.0929	foot-candles	fc
cd/m ²	candela/m ²	0.2919	foot-Lamberts	fl
FORCE and PRESSURE or STRESS				
N	newtons	0.225	poundforce	lbf
kPa	kilopascals	0.145	poundforce per square inch	lbf/in ²

*SI is the symbol for the International System of Units. Appropriate rounding should be made to comply with Section 4 of ASTM E380.
(Revised March 2003)

TABLE OF CONTENTS

1. INTRODUCTION.....	1
2. EXPERIMENTAL DETAILS	5
Equipment Used as the LAST Device	5
Equipment Used for Rheological Characterization	5
Materials Used	6
Testing Protocol.....	7
3. RESULTS AND DISCUSSION	9
Shakedown Experiments in the LAST Device	9
<i>Temperature Control of the LAST Device</i>	9
<i>Sampling Methodology</i>	10
<i>Evaluation of the NCHRP 90-07 Binders</i>	11
Experiments Using the CTS Test.....	11
Evaluation of the NCHRP 90-07 Binders.....	11
4. CONCLUDING REMARKS	13
APPENDIX A	15
APPENDIX B	23
APPENDIX C	37
ACKNOWLEDGMENTS	39
REFERENCE	41
ADDITIONAL SOURCES.....	43

LIST OF FIGURES

<u>Figure</u>	<u>Page</u>
1. Schematic diagram of the original prototype LAST device	2
2. The commercial LAST device	6
3. Plot of temperature versus time showing the temperature control efficacy of the heating mantles used in the commercial unit of the LAST device	9
4. Reproducibility of the G* value for six different samples of EVA grafted (B6232) with glass pipette.....	10

LIST OF TABLES

<u>Table</u>	<u>Page</u>
1. Dimensional details of the LAST device	2
2. List of the modified binders and some select Superpave values	8
3. Time to failure in separation ratio R_s and degradation ratio R_d for the modified binders used in the study	11
4. Results of the CTS test for the modified binders	12
5. Results of the LAST device without agitation for the modified binders	12
6. Elvaloy (B6228) separation ratios	15
7. Elvaloy (B6228) degradation ratios	15
8. SBS-lg (B6229) separation ratios	16
9. SBS-lg (B6229) degradation ratios	16
10. SBS-1 (B6230) separation ratios	17
11. SBS-1 (B6230) degradation ratios	17
12. SBS-rg (B6231) separation ratios	18
13. SBS-rg (B6231) degradation ratios	18
14. EVA (B6232) separation ratios	19
15. EVA (B6232) degradation ratios	19
16. EVA-g (B6233) separation ratios	20
17. EVA-g (B6233) degradation ratios	20
18. ESI (B6243) separation ratios	21
19. ESI (B6243) degradation ratios	21
20. CMCRA (B6251) separation ratios	22
21. CMCRA (B6251) degradation ratios	22
22. Elvaloy (B6228) separation ratios on replicates	23
23. Elvaloy (B6228) degradation ratios on replicates	23
24. Elvaloy (B6228) separation ratios on replicate reruns	24
25. Elvaloy (B6228) degradation ratios on replicate reruns	24
26. SBS-lg (B6299) separation ratios on replicates	25
27. SBS-lg (B6229) degradation ratios on replicates	25
28. SBS-lg (B6299) separation ratios on replicate reruns	26
29. SBS-lg (B6229) degradation ratios on replicate reruns	26
30. SBS-1 (B6230) separation ratios on replicates	27
31. SBS-1 (B6230) degradation ratios on replicates	27
32. SBS-rg (B6231) separation ratios on replicates	28
33. SBS-rg (B6231) degradation ratios on replicates	28
34. SBS-rg (B6231) separation ratios on replicate reruns	29
35. SBS-rg (B6231) degradation ratios on replicate reruns	29
36. EVA (B6232) separation ratios on replicates	30
37. EVA (B6232) degradation ratios on replicates	30
38. EVA (B6232) separation ratios on replicate reruns	31
39. EVA (B6232) degradation ratios on replicate reruns	31
40. EVA-g (B6233) separation ratios on replicates	32

41. EVA-g (B6233) degradation ratios on replicates	32
42. EVA-g (B6233) separation ratios on replicate reruns	33
43. EVA-g (B6233) degradation ratios on replicate reruns	33
44. ESI (B6243) separation ratios on replicates	34
45. ESI (B6243) degradation ratios on replicates	34
46. CMCRA (B6251) separation ratios on replicates	35
47. CMCRA (B6251) degradation ratios on replicates.....	35
48. CMCRA (B6251) separation ratios on replicate reruns.....	36
49. CMCRA (B6251) degradation ratios on replicate reruns	36
50. Data of separation rates and degradation rates	37
51. Data of separation rates and degradation rate returns.....	38

1. INTRODUCTION

The National Cooperative Highway Research Program (NCHRP) Project 9-10, “*Protocols for Modified Asphalt Binders*,” was initiated to determine whether the binder and mixture test methods of Superpave[®], an asphalt-aggregate mixture design and analysis system developed under the Strategic Highway Research Program (SHRP), are as effective for modified binders as they are for the unmodified binders for which they were initially developed. One of the tasks (Task 4SS) focused on measuring special characteristics of modified binders and developing specific testing protocols that are not part of the current Superpave testing protocols. Because most modified binders are multiphase systems, one of the subtasks involved assessing the storage stability of the modified asphalts by evaluating the possible phase separation under static and agitated conditions. Consequently, the Laboratory Asphalt Stability Test (LAST) was developed to address one of the deficiencies of the current testing protocols.⁽¹⁾

Based on the review of research done in the past (see “Additional Sources”) and on evaluation of storage conditions in typical storage tanks in actual practice, the NCHRP 9-10 researchers concluded that the new test for storage stability should incorporate an evaluation of the following effects:⁽¹⁾

- Extended storage at high temperatures in the range of 160 °C to 180 °C.
- Mechanical agitation.
- Storage conditions on performance-related properties measured at multiple temperatures and frequencies in the dynamic shear rheometer.

A study of the field practices revealed that in almost all cases, modified asphalts are stored in tanks with facility for continuous mechanical agitation to maintain material homogeneity and temperature uniformity. Existing protocols to determine the separation and degradation of binders, such as the Cigar Tube Separation (CTS) test, do not include any mechanical agitation and may not reflect field conditions. From among the two basic designs of tanks (horizontal and vertical) used in practice, researchers found the vertical tanks with two propellers to be more efficient than the horizontal tanks. The NCHRP 9-10 researchers used a scaled-down version of a typical 757,000 litre vertical storage tank in their study and called it the LAST device.⁽¹⁾ The dimensional details are given in table 1, and the schematic diagram of the LAST device is shown in figure 1.⁽¹⁾

This device features an internal heating element controlled by an electronic temperature-control feedback system to maintain isothermal conditions and a constant speed, double-propeller agitator centered in the middle of the cylindrical container. The dimensions are such that a sample of 400 milliliters (ml) is used, and the sampling is done periodically using a pipette from the top and bottom of the container without interrupting the conditioning process. The thermal stability is determined by measuring the rheological properties of binders using the Dynamic Shear Rheometer (DSR)-selected conditions before and after conditioning in the LAST device.

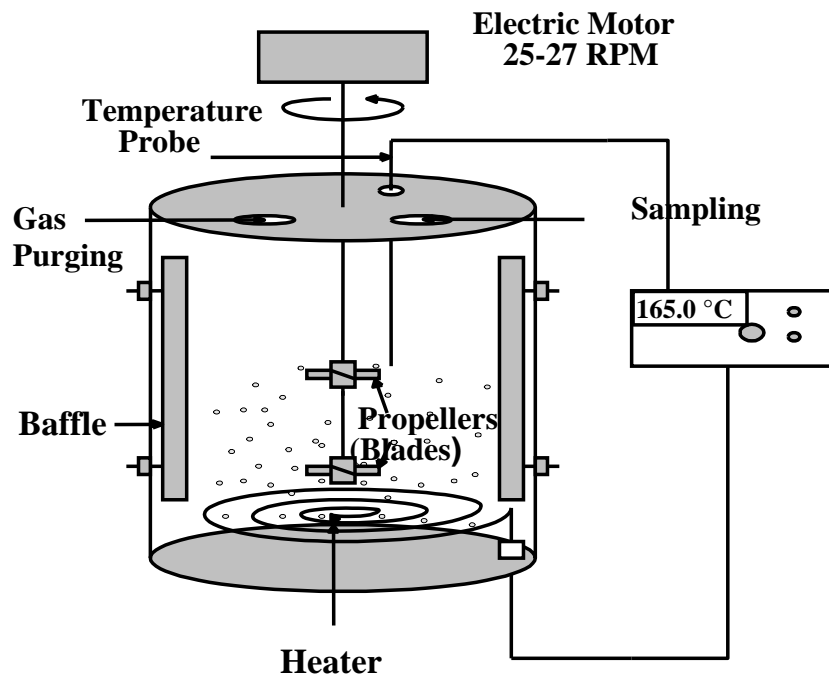
Table 1. Dimensional details of the LAST device

	Original Prototype	Commercial Unit
Internal height of the container	166 mm	203 mm
Internal diameter of the container	62 mm	63.5 mm
Gross volume of the container	501 cm ³	643 cm ³
Diameter of the propellers	22 mm	35 mm
Propeller (1) location on the shaft	85 mm from bottom	19 mm from bottom
Propeller (2) Location on the shaft	20 mm from bottom	Not applicable
Baffle width	4 mm	Not applicable
Amount of binder to be tested	450 g	450 g
Agitation speed	2000 rpm*	365 rpm*
Heating temperature for the test	165 °C	165 °C

*rpm = rotations per minute

Figure 1. Schematic diagram of the original prototype LAST device⁽¹⁾

Changes in properties of samples extracted from the top and bottom of the LAST device as a function of time are used to determine the potential for phase separation or degradation in the field.



The storage stability of the asphalt binders was evaluated using two ratios:

- A separation ratio, R_s , that is calculated by dividing the response (G^* , δ , or a combination of these parameters) of a sample taken from the top portion of the LAST device by the response of another sample taken from the bottom portion of the LAST device under the same conditions.
- A degradation ratio, R_d , that is calculated by dividing the average of the top and bottom responses (G^* , δ , or a combination of these parameters) at a given sampling time by the initial response at time $t=0$ just before the start of the conditioning process in the LAST device.

The parameters investigated were thus,

$$R_s G^* = G^* \text{ separation ratio} = \frac{G^*_{\text{top}}}{G^*_{\text{bottom}}} \quad (1)$$

$$R_s \delta = \delta \text{ separation ratio} = \frac{\delta_{\text{top}}}{\delta_{\text{bottom}}} \quad (2)$$

$$R_d G^* = G^* \text{ degradation ratio} = 0.5 \frac{(G^*_{\text{top}} + G^*_{\text{bottom}})}{G^*_{\text{initial}}} \quad (3)$$

$$R_d \delta = \delta \text{ degradation ratio} = 0.5 \frac{(\delta_{\text{top}} + \delta_{\text{bottom}})}{\delta_{\text{initial}}} \quad (4)$$

The binder is considered to have a potential for separation and for degradation if the ratios are not within 0.8 and 1.2. The other parameters that were determined include K_{si} , which is the separation rate, and K_{di} , which is the degradation rate. These are defined below.

$$K_{si} = \frac{G^* / \text{Sin} \delta (\text{at } T_{cs}) / G^* / \text{Sin} \delta_{\text{initial}}}{T_{cs}} \quad (5)$$

Where T_{cs} is the critical time for separation.

$$K_{di} = \frac{G^* / \text{Sin} \delta (\text{at } T_{cd}) / G^* / \text{Sin} \delta_{\text{initial}}}{T_{cd}} \quad (6)$$

Where T_{cd} is the critical time for degradation.

Federal Highway Administration (FHWA) was assigned the responsibility for evaluating the LAST device. This involved three tasks:

- Procuring a commercial version of the LAST device.
- Correcting perceived deficiencies in this device.
- Conducting a thorough evaluation of the test method.

This report details the work at FHWA's Turner-Fairbank Highway Research Center (TFHRC) in evaluation of the thermal stability of modified binders using the LAST device.

2. EXPERIMENTAL DETAIL

Equipment Used as the LAST Device

The LAST device used for evaluation was a commercial version manufactured using stock materials. This is detailed in table 1 and shown in figure 2. The equipment design equipment was based on available blueprints that maintained the scaled-down dimensions recommended in the original study, but included certain modifications to correct for some perceived deficiencies. The various modifications to the research prototype that were incorporated in the design are noted below:

- Though the device was equipped to provide a nitrogen flow, no nitrogen purge was used. It was believed that the stripping of volatile components due to the purging of nitrogen would be more deleterious than the limited exposure to air during conditioning and sampling.
- No baffle was used, because the shortcomings of this device overshadowed the merits.
- A considerably lower agitation rate of 365 rpm was used. Researchers believed that the excessively high agitation rate of 2000 rpm used in the original study would generate shear rates that could degrade some modifiers.
- Though the propeller shaft was capable of accommodating two impellers a single propeller with a 40° pitch was used, because it was adequate for keeping the modified asphalts stabilized.
- The internal heating that was used in the original study was replaced by an external heat source using side and bottom heating mantles.

Equipment Used for Rheological Characterization

The Rheometrics DSR was used to generate the dynamic data at an intermediate temperature of 16 °C with a set of parallel plates of 8 millimeters (mm) diameter, and a high temperature of 75 °C with a set of parallel plates of 25 mm diameter following the procedure given in the American Association of State Highway and Transportation Officials' (AASHTO) Provisional Standard TP5 (Edition 1A). The samples for the test were prefabricated using a silicone rubber mold. The rheometer and the temperature-controlled unit were operated through a personal computer, and the data acquisition/analysis was done by using specialized software running under Windows® 95. Frequency sweep data from 1 radian/second (s) to 100 radians/s were generated on the binders using a strain low enough to be within the linear viscoelastic response range of the material.



Figure 2. The commercial LAST device

Materials Used

The modified asphalts used in NCHRP 90-07 were investigated in this study. These included elastomers, plastomers, elasto-plastomers, and crumb rubber. The eight binders chosen all met the Superpave performance grading of 70-28 (PG70-28) and included the following polymer-modified systems: Elvaloy[®] (B6228), Styrene-Butadiene-Styrene Linear-Grafted (B6229), Styrene-Butadiene-Styrene Linear (B6230), Styrene-Butadiene-Styrene Radial-Grafted (B6231), Ethylene-Vinyl Acetate (B6232), Ethylene-Vinyl-Acetate Grafted (B6233), Ethylene Styrene Interpolymer (B6243), and Chemically Modified Crumb Rubber (B6251).

The PG number shown is based on the Superpave system description. The polymer-modified

grades were obtained by adding various amounts of different polymers to the asphalt from the same source, namely, Venezuelan crude blend of Boscan and Bachaquero—PG64-28 (base) or the PG52-28 (flux), or mixture of the PG64-28 (base) and the PG52-28 (flux)) in different proportions to achieve the same performance grading. All these asphalts are part of the extensive ongoing polymer research program at the Pavement Testing Facility located at TFHRC. A list of the various binders used in this study and some of their select properties are presented in table 2.

Testing Protocol

Measured quantity of modified binder was heated to 165 °C in a 113.4-gram tin. The tin was removed from the oven and stirred for about 1 minute with an electric mixer to ensure that the asphalt and the modifier were adequately blended. The modified binder then was poured into the vessel, which was preheated to 165 °C. The filled vessel was placed in the heating mantel and connected to the manifold. With the stirrer set to 365 rpm and the temperature of the heating mantles set to 165 °C, the experiment was initiated.

Aliquots of asphalt were withdrawn from the top and bottom of the vessel. These samples were obtained following 0, 6, 24, 31, and 48-hour (hr) conditioning periods. This procedure was repeated twice; once with agitation (365 rpm), and once without agitation (static). Agitation was halted briefly while the bottom aliquot was withdrawn. The withdrawn samples were poured directly into silicone molds of 8 mm and 25 mm in preparation for analyses on the DSR. Frequency sweep data was generated from 1 radian/s to 100 radians/s at two temperatures (16 °C and 75 °C), and the G^* and δ determined at 10 radians/s at these two temperatures was used to calculate the separation ratios, R_s and degradation ratios, R_d .

The protocol discussed above is radically different from the conventional CTS test. In the CTS test, a homogeneous sample is placed in the tube and conditioned in the oven at 165 °C for 2 days. Following this, the material is quench-cooled in a freezer at -20 °C. The frozen specimen is retrieved and cut into three sections. The rheological properties taken from the top and bottom sections are compared, and deviations larger than 10 percent are considered to be representative of phase separation. The CTS test was also used in this study for comparison purposes.

Table 2. List of the modified binders and some select Superpave values

Binder	PG	High Limiting Temperature Neat Binder	Intermediate Limiting Temperature
Elvaloy (B6228)	77-31	75	16
SBS-lg (B6229)	72-33	75	15
SBS-l (B6230)	72-31	70	18
SBS-r (B6231)	72-32	74	16
EVA (B6232)	71-31	70	13
EVA-g (B6233)	74-32	74, 76	15, 13
ESI (B6243)	76-31	76	10
CMCRA (B6251)	76-29	76	17

3. RESULTS AND DISCUSSION

The procedure followed for data analyses in all cases involved (a) generating relevant DSR data, (b) calculating the separation ratio, degradation ratio, separation rate, and degradation rate, and (c) determining whether the criterion for the ratio to be within 0.8 and 1.2 was met to assess the stability of the binder. The data used in the calculations and the values of the R_s , R_d , K_{si} , K_{di} are tabulated in appendices A, B, and C.

Shakedown Experiments in the LAST Device

Temperature Control of the LAST Device

The ability of the commercial unit to maintain a uniform temperature was evaluated. A sample of asphalt was placed in the detachable flask and heated using the side and bottom heating mantles. Figure 3 shows a plot of temperature as a function of time. Within 20 minutes, the set temperature was reached. Although a slight (± 5 °C) temperature fluctuation exists over the next hour, this was found to be of little consequence. To evaluate the effect of agitation, stirring at 365 rpm was initiated at the 3-hour mark. There is an initial drop in temperature, and thermal equilibrium is reestablished within 40 minutes.

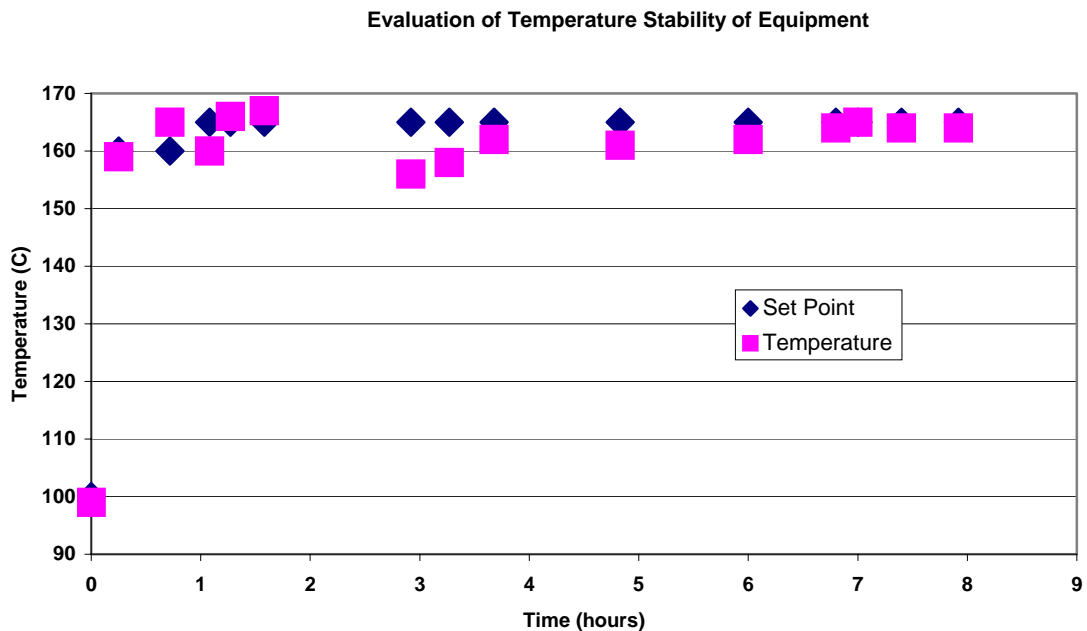


Figure 3. Plot of temperature versus time showing the temperature control efficacy of the heating mantles used in the commercial unit of the LAST device

Sampling Methodology

To assess the effectiveness of the sampling methodology, the EVA-grafted asphalt binder was chosen to represent a heterogeneous material. Based on the CTS test, EVA-grafted asphalt binder exhibited a tendency to phase separate. Twelve aliquots of the EVA-grafted asphalt binder were withdrawn from vessel after 6 hours of heating without stirring; six were taken from the top and six from the bottom. Rheological data on these were determined on the DSR over the course of the following 2 days. Figure 4 shows a plot of six pairs of G^* values obtained at 73 °C. Samples obtained from the top exhibited slightly higher values than those taken from the bottom of the vessel. There is no evidence of steric hardening in these particular samples, because the data is fairly consistent, and no trend of stiffening with time is present. The high variability of sample 1 indicated that replicate samples are advised.

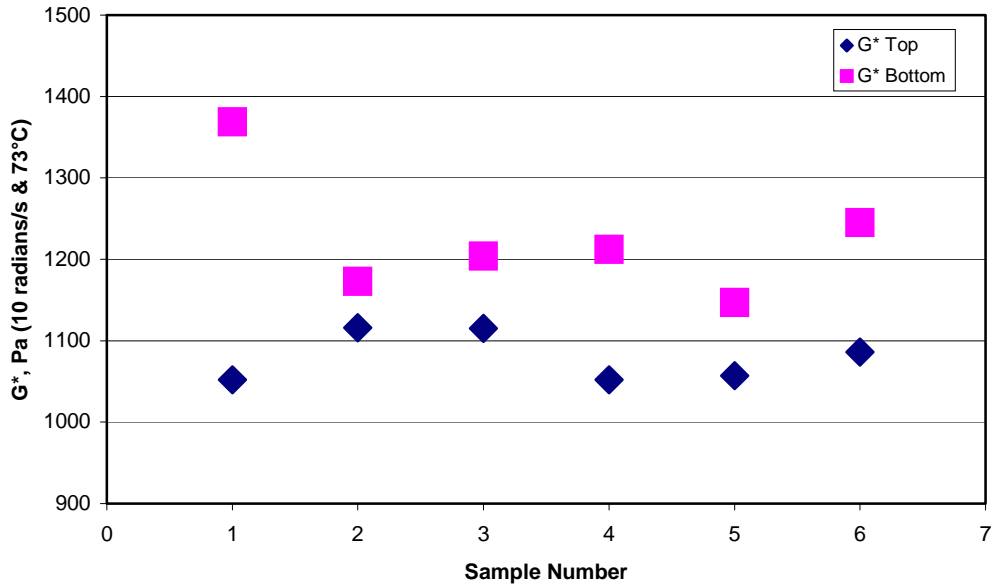


Figure 4. Reproducibility of the G^* value for six different samples of EVA grafted (B6232) with glass pipette

Evaluation of the NCHRP 90-07 Binders

The eight polymer-modified binders were tested for their thermal stability in the LAST device under two conditions: (a) without mechanical agitation and (b) with mechanical agitation. Samples were withdrawn from the top and the bottom at time intervals of 0 hr, 6 hr, 24 hr, 31 hr, and 48 hr. In each case, two replicates were drawn, and in some cases, the entire run was duplicated to verify any observed discrepancies. The rheological properties were determined on the DSR. The separation ratios, degradation ratios, separation rates, and degradation rates then were calculated. The calculated values for the separation ratios and degradation ratios are provided in appendix A for the averaged data and in appendix B for the data on the replicates. Data deviating significantly from the average are highlighted. The calculated values for the separation rates and degradation rates are provided in appendix C.

The results from the runs with mechanical agitation are given in table 3. It can be seen that mechanical agitation prevents separation, although it can degrade certain binders if this agitation continues for a long time. This is evident from the results shown in table 3; four of the eight binders studied failed the degradation test after 48 hrs of agitation.

Table 3. Time to failure in separation ratio R_s and degradation ratio R_d for the modified binders used in the study

Binder	R_s	R_d
Elvaloy (B6228)	–	–
SBS linear-grafted (B6229)	–	48 hr
SBS linear (B6230)	–	–
SBS radial (B6231)	–	48 hr
EVA (B6232)	–	48 hr
EVA grafted (B6233)	–	–
ESI (B6243)	–	–
CMCRA (B6251)	–	48 hr

Experiments Using the CTS Test

Evaluation of the NCHRP 90-07 Binders

The eight polymer-modified binders were tested for their thermal stability using the CTS test. The rheological properties were determined on the DSR for the top and bottom portion after running the CTS test. Major differences were observed in the G^* values between the top and the bottom, while the changes in the δ values between the top and the bottom were marginal, as is seen in table 4.

Table 4. Results of the CTS test for the modified binders*

	TOP G*	TOP δ	BOTTOM G*	BOTTOM δ	Cigar tube <5% Pass Separation	HB Calculation (0.8-1.2 Pass) R_s G*	R_s (δ)
Elvaloy (B6228)	776.46	76.47	811.36	76.629	2.25	0.96	1.00
SBS-lg (B6229)	1751.7	76.255	1780.4	76.79	0.82	0.98	0.99
SBS-l (B6230)	1325.1	78.252	1695.2	78.027	13.96	0.78	1.00
SBS-rg (B6231)	1593.7	76.614	1593	79.075	0.02	1.00	0.97
EVA (B6232)	602.71	84.086	2927.5	82.11	192.86	0.21	1.02
EVA-g (B6233)	859.67	84.085	3920.5	81.599	178.02	0.22	1.03
ESI (B6243)	1209.8	73.935	1210.3	87.583	0.02	1.00	0.84
CMRA (B6251)	2032.4	78.12	1787.2	78.572	6.03	1.14	0.99

*Cigar tube 16 hrs

Because the CTS test was done under static conditions, the separation results from the CTS test were compared with the separation results obtained from the LAST device without any mechanical agitation. These are shown in table 5. Comparing the CTS test results with the separation results from LAST showed an excellent relationship with a correlation coefficient $R^2 \approx 0.9$.

Table 5. Results of the LAST device without agitation for the modified binders*

	TOP G*	TOP δ	BOTTOM G*	BOTTOM δ	Cigar Tube Separation	HB Calculation R_s G*	R_s (δ)
Elvaloy (B6228)	1014	68.4	1018	68.3	0.87	1.00	1.00
SBS-lg (B6229)	991	81.1	971	81.5	1.05	1.02	1.00
SBS-rg (B6231)	1452	77.2	1329	78.1	4.24	1.09	0.99
EVA (B6232)	1238	83.8	2175	82.5	37.82	0.57	1.11
EVA-g (B6233)	861	87.4	1237	84.6	21.81	0.70	1.03
ESI (B6243)	1078	76.0	1095	76	0.76	0.99	1.00
CMCRA (B6251)	906	83.1	950	81.6	1.88	0.95	1.02

*24 hr/ 76 °C/ without mechanical agitation/ frequency=10 radians/s

4. CONCLUDING REMARKS

The NCHRP 9-10 researchers used three sampling times (6 hr, 24 hr, 48 hr) and in the present study, four sampling times (6 hr, 24 hr, 31 hr, 48 hr) were used. While these are acceptable during procedure development, they are not convenient for use during routine testing in practice. Therefore, to shorten the test, it is recommended that sampling be done only at 0 hr and 48 hr. This, however, does not guarantee that the conclusions drawn would always be accurate.

In fact, based on the results obtained for the modified binders analyzed in this study, the LAST cannot be truly termed a reliable test. On a number of occasions, the samples that failed the R_s or R_d criterion in the first instance passed when reruns were performed. This implies that the results based on the LAST cannot always be trusted to be accurate enough to judge the stability of the modified binders.

The test itself is very time-consuming, and too much care must be taken to make sure that the time of drawing of samples matches with the time when the DSR would be available to generate rheological data. If there are delays, then the results cannot be trusted, due to the effect of an extra time variable that has crept into the experimental plan.

To generate rheological data for the LAST, the DSR was locked in for extended periods of time due to the nature of the sampling conditions. However, there might be a way to circumvent this problem. In almost all cases, because the variation in the phase angle δ at different time intervals is marginal, one possibility would be to track the stability of the samples through devices other than the DSR, namely, viscometers that could assess only the viscous property; they should be able to assess the stability with equal efficacy.

APPENDIX A
Averaged Data of Separation Ratios and Degradation Ratios

Table 6. Elvaloy (B6228) separation ratios

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Frequency (rads/s)	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
0 hour	0.95	1.00	1.03	1.00	0.93	1.00	0.93	1.00	1.00	1.00	0.98	1.00	1.76	1.00	1.75	1.00
6 hour	1.00	0.89	1.00	1.00	1.03	1.00	1.03	0.98	0.95	1.00	0.96	1.02	1.08	1.00	1.08	0.99
24 hour	0.99	1.00	0.99	1.00	0.96	1.00	0.96	1.00	0.96	1.00	0.97	1.01	1.06	0.99	1.05	0.99
31 hour	1.02	1.00	1.00	1.00	1.07	1.00	1.07	1.00	1.01	1.00	1.01	1.00	1.00	1.00	1.01	1.00
48 hour	0.97	1.00	0.97	1.01	0.88	1.00	0.89	1.00	0.94	1.00	0.97	1.01	1.01	1.00	1.00	0.99

Table 7. Elvaloy (B6228) degradation ratios

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Frequency (rads/s)	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
0 hour	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
6 hour	0.89	1.03	0.97	1.04	0.98	1.00	0.98	1.00	0.78	1.13	0.90	1.07	0.85	1.00	0.87	1.01
24 hour	0.89	1.05	1.00	1.03	0.97	0.99	0.95	0.99	0.79	1.04	0.93	1.07	0.96	0.99	0.94	0.99
31 hour	0.92	1.05	1.00	1.03	0.99	0.99	0.98	0.99	0.85	1.03	0.98	1.04	0.95	0.99	0.94	0.99
48 hour	0.96	1.04	1.06	1.01	1.01	0.99	0.99	0.98	0.96	1.01	1.05	1.01	0.98	0.99	0.95	0.98

Table 8. SBS-Ig (B6229) separation ratios

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Frequency (rads/s)	HT		HT		IT		IT		HT		HT		IT		IT	
Temperature	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
Time of Sampling	1.00	1.00	0.99	1.00	1.04	1.00	1.04	1.00	0.93	1.02	1.01	1.01	1.03	1.01	1.03	1.01
0 hour	1.00	1.00	0.99	1.00	1.04	1.00	1.04	1.00	0.93	1.02	1.01	1.01	1.03	1.01	1.03	1.01
6 hour	0.97	0.99	0.97	1.01	1.20	0.99	1.06	0.99	0.93	1.00	0.94	1.02	0.92	1.00	0.93	1.00
24 hour	0.99	1.00	1.01	1.00	0.99	1.02	1.30	0.88	0.97	0.99	0.97	1.01	0.92	1.00	0.93	1.00
31 hour	1.02	1.01	1.00	0.99	1.07	1.01	1.08	1.00	0.98	0.99	0.99	1.00	1.09	1.00	1.10	1.00
48 hour	1.69	0.98	1.65	0.90	0.90	1.00	0.90	0.99	1.00	1.01	1.01	0.99	1.00	1.00	0.99	1.00

Table 9. SBS-Ig (B6229) degradation ratios

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Frequency (rads/s)	HT		HT		IT		IT		HT		HT		IT		IT	
Temperature	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
Time of Sampling	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.02	1.00	1.43	1.00	0.95	1.00	0.84
0 hour	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.02	1.00	1.43	1.00	0.95	1.00	0.84
6 hour	1.07	1.02	1.03	0.99	0.95	0.98	0.92	0.99	1.03	1.01	1.03	0.98	1.06	1.00	1.05	1.00
24 hour	1.16	0.99	1.04	0.96	0.90	1.00	0.89	1.00	1.12	0.99	1.04	0.96	0.98	1.00	0.97	1.00
31 hour	1.23	0.96	1.04	0.94	0.97	1.00	0.96	1.00	1.17	0.98	1.03	0.96	1.02	1.01	1.03	1.00
48 hour	4.79	0.90	2.89	0.74	0.89	1.00	0.88	1.00	1.24	0.93	0.99	0.94	0.98	1.01	1.00	1.01

Table 10. SBS-I (B6230) separation ratios

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Frequency (rads/s)	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
0 hour	1.13	0.98	1.10	0.99	0.96	1.00	0.97	1.01	1.07	0.98	1.06	1.00	–	–	–	–
6 hour	1.03	0.99	1.01	0.99	0.97	0.99	0.96	0.99	0.96	1.02	1.00	1.01	1.05	1.01	1.05	1.00
24 hour	1.00	1.00	1.00	1.00	1.01	0.99	0.99	0.99	0.98	1.01	0.99	1.01	0.98	1.00	0.99	1.00
31 hour	0.98	1.00	0.98	1.00	1.04	1.01	1.07	1.01	1.00	1.03	1.00	1.01	1.05	1.00	1.05	1.00
48 hour	0.97	1.01	0.99	1.00	0.93	1.00	0.94	1.00	1.03	0.99	1.01	1.00	1.07	1.00	1.07	1.00

Table 11. SBS-I (B6230) degradation ratios

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Frequency (rads/s)	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
0 hour	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	–	–	–	–
6 hour	1.21	1.03	1.20	0.97	0.95	0.92	0.91	0.98	1.00	1.00	1.05	0.98	1.14	0.99	1.13	1.89
24 hour	1.13	1.01	1.08	0.96	0.90	0.98	0.87	0.99	1.22	0.95	1.06	0.93	1.03	1.00	1.03	1.93
31 hour	1.15	1.02	1.10	0.96	0.91	0.96	0.87	0.97	1.26	0.95	1.10	0.93	1.08	1.00	1.07	0.91
48 hour	1.23	1.01	1.14	0.95	0.97	0.98	0.94	0.98	1.26	0.94	1.03	0.91	1.12	1.00	1.12	0.91

Table 12. SBS-rg (B6231) separation ratios

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Frequency (rads/s)	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
0 hour	0.86	1.01	0.96	1.03	1.04	1.00	1.04	1.00	0.86	1.02	0.91	1.04	0.99	1.01	1.00	1.00
6 hour	0.92	0.99	1.04	0.98	1.00	1.00	1.01	1.00	1.22	1.01	0.93	1.01	0.96	1.11	0.97	1.09
24 hour	1.05	0.98	1.04	0.99	0.97	1.00	0.98	1.00	0.97	0.99	1.00	1.00	1.06	1.00	1.06	1.00
31 hour	1.07	0.99	1.01	0.98	1.10	1.00	1.09	1.00	1.01	0.98	0.97	1.00	1.05	1.01	1.08	1.02
48 hour	1.14	0.99	1.01	0.99	1.06	1.01	1.07	1.00	0.62	1.00	0.99	1.01	0.96	1.00	0.95	1.00

Table 13. SBS-rg (B6231) degradation ratios

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Frequency (rads/s)	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
0 hour	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
6 hour	1.75	0.94	1.13	0.88	0.94	0.96	0.90	0.96	1.12	1.03	1.15	0.99	0.81	0.96	0.80	0.97
24 hour	1.30	0.94	1.03	0.92	0.81	0.98	0.80	0.99	1.15	1.00	1.09	0.98	0.97	1.01	0.99	1.01
31 hour	1.29	0.94	0.98	0.92	0.86	0.97	0.84	0.98	1.12	1.00	1.09	0.98	0.90	1.01	0.91	1.01
48 hour	1.35	0.94	0.98	0.92	0.85	0.98	0.84	0.99	1.73	0.98	1.10	0.97	0.90	1.01	0.92	1.01

Table 14. EVA (B6232) separation ratios

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
0 hour	0.99	1.00	0.99	1.00	1.03	1.01	1.06	1.01	0.93	0.95	0.85	1.02	1.08	1.00	0.99	1.00
6 hour	0.73	1.02	0.76	1.04	0.67	1.02	0.69	1.03	0.94	0.99	0.98	1.00	1.05	1.00	1.04	1.00
24 hour	0.62	1.04	0.67	1.07	0.62	1.04	0.66	1.07	0.97	1.00	0.97	1.01	0.83	0.99	0.83	0.99
31 hour	0.69	1.00	0.70	1.03	0.69	1.05	0.70	1.03	–	–	–	–	–	–	–	–
48 hour	0.40	1.07	0.45	1.11	0.40	1.07	0.45	1.11	0.92	0.99	0.91	1.02	1.11	0.99	1.12	0.99

Table 15. EVA (B6232) degradation ratios

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
0 hour	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
6 hour	0.59	0.66	0.59	0.69	0.87	1.01	0.89	1.03	1.12	1.02	1.13	0.98	0.87	1.02	0.90	1.03
24 hour	1.01	1.01	1.03	1.00	1.02	1.00	1.15	1.01	1.18	1.02	1.19	0.97	0.90	1.01	0.91	1.01
31 hour	1.18	1.00	1.20	0.98	1.02	1.01	1.02	1.01	–	–	–	–	–	–	–	–
48 hour	1.15	0.99	1.13	0.97	0.77	1.01	0.81	1.04	1.24	1.01	1.22	0.96	0.91	1.00	0.92	1.01

Table 16. EVA-g (B6233) separation ratios

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Frequency (rads/s)	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
0 hour	0.99	1.00	0.99	1.00	1.03	1.01	1.06	1.01	0.98	1.00	0.97	1.01	1.07	0.98	1.05	0.98
6 hour	0.90	1.00	0.91	1.02	0.73	1.02	0.76	1.04	0.96	1.00	0.98	1.01	0.97	1.00	0.96	1.00
24 hour	0.75	1.03	0.79	1.08	0.62	1.05	0.67	1.08	0.91	1.02	0.95	1.03	1.09	1.01	1.10	1.01
31 hour	0.75	1.01	0.77	1.07	0.70	1.01	0.71	0.92	1.00	1.00	1.01	1.00	1.04	1.01	1.05	1.01
48 hour	0.72	1.01	0.75	1.08	0.40	1.07	0.45	1.18	1.03	1.00	1.04	1.00	0.89	1.01	0.90	1.01

Table 17. EVA-g (B6233) degradation ratios

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Frequency (rads/s)	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
0 hour	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
6 hour	1.04	1.00	1.03	0.99	0.92	1.02	0.95	1.02	0.94	1.00	0.95	1.02	0.93	1.01	0.94	1.01
24 hour	1.15	0.99	1.10	0.97	1.07	1.00	1.07	1.01	1.04	0.99	1.01	0.99	0.90	1.00	0.90	1.00
31 hour	1.15	0.99	1.11	0.98	0.86	1.02	0.89	1.05	1.04	1.00	1.02	0.99	0.88	1.02	0.91	1.03
48 hour	1.21	0.99	1.17	0.96	1.25	0.99	1.22	1.00	1.08	0.99	1.04	0.98	0.87	1.00	0.88	1.01

Table 18. ESI (B6243) separation ratios

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
0 hour	1.02	1.00	1.02	1.00	1.17	1.00	1.16	0.99	0.96	1.00	0.96	1.01	0.99	0.99	0.99	0.99
6 hour	0.95	1.00	0.95	1.02	0.95	1.01	0.96	1.02	0.94	1.03	0.98	1.01	1.01	1.02	1.00	1.04
24 hour	1.00	1.00	0.99	1.01	0.70	1.03	0.75	1.06	0.98	0.98	0.98	1.00	0.95	0.99	0.96	1.00
31 hour	0.91	1.00	0.92	1.03	0.69	1.05	0.75	1.06	1.01	1.01	1.00	1.00	1.02	0.98	1.01	0.98
48 hour	1.01	1.00	0.98	1.00	0.86	1.01	0.87	1.02	1.04	1.00	1.02	0.99	0.83	1.01	0.86	1.02

Table 19. ESI (B6243) degradation ratios

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
0 hour	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
6 hour	1.01	1.00	1.00	1.00	1.18	0.98	1.14	0.98	1.15	0.98	1.05	0.98	0.92	1.01	0.91	1.00
24 hour	1.04	0.98	1.09	0.66	1.08	0.99	1.05	0.99	1.16	0.98	1.07	0.97	0.81	1.02	0.85	1.02
31 hour	1.03	1.00	1.04	0.99	1.19	0.98	1.14	0.98	1.09	1.00	1.07	0.97	0.85	1.01	0.88	1.01
48 hour	1.18	1.00	1.14	0.95	1.04	0.98	1.01	0.98	1.18	1.00	1.15	0.95	0.97	1.00	0.98	0.99

Table 20. CMCRA (B6251) separation ratios

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Frequency (rads/s)	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
0 hour	0.98	1.00	0.97	1.00	1.12	1.00	1.13	1.00	1.08	1.00	1.10	0.99	1.02	1.00	1.02	0.99
6 hour	0.84	1.01	0.90	1.05	1.07	1.04	1.08	1.00	1.05	0.98	1.04	0.98	0.98	1.01	0.98	1.01
24 hour	–	–	–	–	–	–	–	–	0.98	1.00	1.01	1.00	1.04	1.00	1.05	1.00
31 hour	0.67	1.07	0.83	1.11	1.13	1.00	1.13	1.00	0.88	1.01	0.90	1.04	0.93	1.00	0.93	1.00
48 hour	0.69	1.09	0.89	1.10	1.15	1.00	1.15	0.99	1.00	1.00	1.01	1.00	0.98	1.00	0.98	1.00

Table 21. CMCRA (B6251) degradation ratios

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Frequency (rads/s)	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
0 hour	1.00	1.58	1.00	1.59	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
6 hour	0.69	1.02	0.82	1.11	1.15	1.00	1.14	0.99	1.03	1.01	1.00	0.98	0.97	1.00	0.96	1.00
24 hour	–	–	–	–	–	–	–	–	1.10	1.01	1.05	0.97	0.96	1.00	0.95	1.00
31 hour	1.00	0.97	0.99	1.01	1.14	0.99	1.12	0.99	1.14	1.00	1.06	0.81	0.99	0.99	0.97	0.99
48 hour	1.02	0.96	0.98	1.00	1.14	0.99	1.11	0.98	1.23	1.01	1.15	0.94	0.99	0.99	0.97	0.99

APPENDIX B
Data of Separation Ratios and Degradation Ratios on Replicates
 Data deviating significantly from the average are highlighted.

Table 22. Elvaloy (B6228) separation ratios on replicates

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
0 hour	0.95	1.00	1.03	1.00	0.93	1.00	0.93	1.00	1.00	1.00	0.98	1.00	1.76	1.00	1.75	1.00
6 hour	0.96	1.00	0.98	1.01	1.00	1.00	1.00	1.00	0.95	1.00	0.96	1.01	1.07	1.00	1.08	1.00
	1.04	1.00	1.02	0.99	1.08	1.00	1.11	1.01	0.91	1.00	0.91	1.04	1.15	1.00	1.16	1.00
	1.01	0.66	0.99	1.00	1.01	1.00	1.03	1.00	1.00	1.00	1.00	1.00	1.02	1.00	1.01	0.98
24 hour	0.98	1.00	0.99	1.00	0.97	1.00	0.97	1.00	0.99	1.00	0.97	1.01	1.22	0.97	1.18	0.99
	1.01	1.00	1.00	1.00	1.02	1.00	1.01	1.00	0.96	1.00	0.98	1.01	1.04	1.00	1.05	1.00
	0.97	1.00	0.98	1.01	0.90	1.00	0.90	1.00	0.93	1.01	0.97	1.02	0.93	1.00	0.93	1.00
31 hour	1.02	1.00	1.00	1.00	0.94	1.00	0.93	1.00	1.10	1.01	1.09	0.97	1.11	1.00	1.13	1.00
	1.01	1.00	1.01	0.99	1.22	1.00	1.23	1.00	0.96	1.00	0.97	1.01	0.94	1.00	0.95	1.00
	1.02	1.00	0.98	1.00	1.05	1.00	1.05	1.00	0.97	1.00	0.97	1.00	0.96	1.00	0.96	0.99
48 hour	1.00	0.99	0.97	1.01	0.96	1.00	0.96	1.00	0.93	1.00	0.98	1.02	1.06	1.00	1.04	0.99
	0.92	1.01	0.93	1.03	0.76	1.01	0.78	1.00	0.99	1.00	0.99	1.00	0.96	1.00	0.96	1.00
	1.00	1.00	1.01	1.00	0.91	1.00	0.92	1.00	0.91	1.00	0.93	1.02	1.01	1.00	1.01	1.00

Table 23. Elvaloy (B6228) degradation ratios on replicates

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
0 hour	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
6 hour	0.81	1.04	0.92	1.06	0.91	1.00	0.90	1.00	0.88	1.03	0.96	1.04	0.96	1.00	0.96	1.00
	0.74	1.04	0.87	1.09	0.80	1.00	0.79	0.99	0.89	1.03	0.97	1.04	1.00	1.00	1.00	1.00
	0.79	1.31	0.91	1.07	0.84	1.00	0.84	0.99	0.89	1.03	0.97	1.03	0.98	1.00	0.99	1.01
24 hour	0.80	1.05	0.93	1.07	1.02	0.99	1.01	0.99	0.88	1.05	0.99	1.03	0.96	0.98	0.94	0.99
	0.78	1.04	0.92	1.08	0.98	0.99	0.96	0.99	0.89	1.05	1.00	1.03	0.96	1.00	0.96	0.99
	0.79	1.04	0.94	1.07	0.87	0.99	0.85	0.99	0.91	1.05	1.02	1.03	0.98	0.99	0.96	0.99
31 hour	0.86	1.03	0.98	1.04	0.96	0.99	0.94	0.99	0.92	1.04	1.02	1.03	1.02	0.99	1.01	0.99
	0.85	1.03	0.98	1.04	0.93	0.99	0.91	0.99	0.90	1.05	1.00	1.03	0.93	0.99	0.91	0.99
	0.85	1.03	0.98	1.05	0.98	0.99	0.97	0.99	0.94	1.05	1.05	1.02	1.03	0.99	1.01	0.99
48 hour	0.98	1.01	1.08	1.00	0.96	0.99	0.94	0.98	0.98	1.04	1.08	1.00	1.02	0.99	1.00	0.99
	0.94	1.02	1.03	1.01	1.08	0.98	1.05	0.98	0.98	1.04	1.08	1.00	1.01	0.99	1.00	0.98
	0.95	1.01	1.04	1.01	0.89	0.98	0.86	0.98	0.92	1.04	1.03	1.03	0.99	0.98	0.97	0.98

Table 24. Elvaloy (B6228) separation ratios on replicate reruns

Conditioning Step	External Heat without Agitation							
	5.0		5.0 Rerun		50.0		50.0 Rerun	
Temperature	HT		HT		HT		HT	
Time of Sampling	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
0 hour	0.95	1.00	–	–	1.03	1.00	–	–
6 hour	0.96	1.00	1.04	1.00	0.98	1.01	1.03	1.00
	1.04	1.00	1.00	1.00	1.02	0.99	1.00	1.00
	1.01	0.66	–	–	0.99	1.00	–	–
24 hour	0.98	1.00	0.97	1.01	0.99	1.00	0.98	1.00
	1.01	1.00	1.02	1.00	1.00	1.00	1.01	1.00
	0.97	1.00	–	–	0.98	1.01	–	–
31 hour	1.02	1.00	1.05	0.99	1.00	1.00	1.04	1.00
	1.01	1.00	0.96	1.00	1.01	0.99	0.97	1.00
	1.02	1.00	–	–	0.98	1.00	–	–
48 hour	1.00	0.99	0.97	1.00	0.97	1.01	0.97	1.00
	0.92	1.01	–	–	0.93	1.03	–	–
	1.00	1.00	–	–	1.01	1.00	–	–

Table 25. Elvaloy (B6228) degradation ratios on replicate reruns

Conditioning Step	External Heat without Agitation							
	5.0		5.0 Rerun		50.0		50.0 Rerun	
Temperature	HT		HT		HT		HT	
Time of Sampling	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
0 hour	1.00	1.00	–	–	1.00	1.00	–	–
6 hour	0.81	1.04	0.88	1.03	0.92	1.06	1.00	0.93
	0.74	1.04	0.86	1.03	0.87	1.09	0.90	1.02
	0.79	1.31	–	–	0.91	1.07	–	–
24 hour	0.80	1.05	0.86	1.04	0.93	1.07	0.92	1.03
	0.78	1.04	0.87	1.04	0.92	1.08	0.93	1.03
	0.79	1.04	–	–	0.94	1.07	–	–
31 hour	0.86	1.03	0.90	1.03	0.98	1.04	0.95	1.03
	0.85	1.03	0.86	1.03	0.98	1.04	0.92	1.03
	0.85	1.03	–	–	0.98	1.05	–	–
48 hour	0.98	1.01	0.94	1.02	1.08	1.00	0.98	1.02
	0.94	1.02	–	–	1.03	1.01	–	–
	0.95	1.01	–	–	1.04	1.01	–	–

Table 26. SBS-Ig (B6229) separation ratios on replicates

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Frequency (rads/s)	HT		HT		IT		IT		HT		HT		IT		IT	
Temperature	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
0 hour	1.0	1.00	0.99	1.00	1.04	1.00	1.04	1.00	0.93	1.02	1.01	1.01	1.03	1.01	1.03	1.01
6 hour	1.02	0.99	1.00	1.00	1.3	0.99	1.01	0.99	0.89	1.01	0.92	1.03	0.96	1.01	0.98	1.01
	0.93	0.99	0.95	1.02	1.13	1.00	1.11	1.00	0.97	0.99	0.97	1.01	0.89	1.00	0.89	1.00
24 hour	1.04	0.99	1.02	1.00	1.51	1.03	1.60	1.03	0.97	0.97	0.96	1.01	0.75	1.00	0.76	1.00
	0.97	1.02	1.01	1.00	0.99	1.01	1.00	1.00	0.98	1.01	0.99	1.01	1.10	1.00	1.10	1.00
31 hour	1.03	1.02	1.04	0.99	1.09	1.01	1.11	1.01	1.02	0.97	1.00	1.00	1.13	1.00	1.13	1.00
	1.01	0.99	0.99	1.00	1.05	1.01	1.05	1.00	0.94	1.01	0.99	1.01	1.06	1.00	1.07	1.00
48 hour	0.70	1.01	0.92	1.00	0.79	0.99	0.78	0.99	0.99	1.02	1.01	1.00	1.01	1.00	1.00	1.00
	2.67	0.95	2.37	0.79	1.02	1.00	1.02	1.00	1.00	0.99	1.00	0.97	0.98	1.00	0.98	1.00

Table 27. SBS-Ig (B6229) degradation ratios on replicates

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Frequency (rads/s)	HT		HT		IT		IT		HT		HT		IT		IT	
Temperature	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
0 hour	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
6 hour	1.08	1.01	1.03	0.98	0.93	0.98	0.90	0.99	1.01	1.01	1.01	0.99	1.05	1.00	1.05	1.00
	1.05	1.02	1.02	0.99	0.97	0.98	0.94	0.99	1.04	1.01	1.04	0.97	1.06	1.00	1.05	0.99
24 hour	1.16	1.00	1.04	0.96	0.78	0.99	0.76	0.99	1.11	0.98	1.02	0.96	1.00	1.00	0.99	1.00
	1.16	0.98	1.03	0.96	1.01	1.00	1.01	1.00	1.13	0.99	1.05	0.96	0.95	1.00	0.95	1.00
31 hour	1.26	0.95	1.04	0.94	0.93	1.00	0.92	1.00	1.16	0.97	1.02	0.96	1.03	1.01	1.03	1.00
	1.20	0.97	1.03	0.94	1.01	1.00	1.00	1.00	1.18	0.98	1.04	0.95	1.01	1.01	1.02	1.00
48 hour	6.94	0.88	3.93	0.67	0.84	0.99	0.83	1.00	1.24	0.91	0.98	0.94	1.00	1.01	1.02	1.01
	2.63	0.92	1.84	0.81	0.93	1.00	0.92	1.00	1.23	0.94	1.00	0.94	0.95	1.01	0.97	1.01

Table 28. SBS-Ig (B6229) separation ratios on replicate reruns

Conditioning Step	External Heat without Agitation							
	5.0		5.0 Rerun		50.0		50.0 Rerun	
Temperature	HT		HT		HT		HT	
Time of Sampling	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
0 hour	1.0	1.00	1.04	1.01	0.99	1.00	1.04	1.00
6 hour	1.02	0.99	0.84	1.00	1.00	1.00	0.84	1.01
	0.93	0.99	1.00	0.99	0.95	1.02	1.00	1.00
24 hour	1.04	0.99	0.95	0.99	1.02	1.00	0.94	1.00
	0.97	1.02	0.99	0.99	1.01	1.00	0.98	1.00
31 hour	1.03	1.02	–	–	1.04	0.99	–	–
	1.01	0.99	–	–	0.99	1.00	–	–
48 hour	0.70	1.01	1.03	1.00	0.92	1.00	1.03	1.00
	2.67	0.95	0.98	1.00	2.37	0.79	0.97	1.00

Table 29. SBS-Ig (B6229) degradation ratios on replicate reruns

Conditioning Step	External Heat without Agitation							
	5.0		5.0 Rerun		50.0		50.0 Rerun	
Temperature	HT		HT		HT		HT	
Time of Sampling	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
0 hour	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
6 hour	1.08	1.01	0.89	1.03	1.03	0.98	0.90	1.00
	1.05	1.02	0.94	1.02	1.02	0.99	0.96	1.00
24 hour	1.16	1.00	1.00	1.00	1.04	0.96	0.95	0.97
	1.16	0.98	1.07	0.99	1.03	0.96	1.00	0.97
31 hour	1.26	0.95	–	–	1.04	0.94	–	–
	1.20	0.97	–	–	1.03	0.94	–	–
48 hour	6.94	0.88	1.17	0.94	3.93	0.67	1.01	0.94
	2.63	0.92	1.21	0.92	1.84	0.81	1.03	0.93

Table 30. SBS-I (B6230) separation ratios on replicates

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
0 hour	1.13	0.98	1.10	0.99	0.96	1.00	0.97	1.01	1.07	0.98	1.06	1.00	–	–	–	–
6 hour	1.03	0.98	1.01	0.98	0.89	0.97	0.84	0.97	0.99	1.03	1.04	1.02	1.08	1.01	1.10	1.01
	1.02	0.99	1.01	1.00	1.05	1.01	1.08	1.01	0.93	1.04	1.00	1.02	1.11	1.01	1.12	1.01
	1.04	1.01	1.02	0.99	–	–	–	–	0.97	0.99	0.97	1.00	0.95	1.00	0.94	0.99
24 hour	1.01	1.00	1.02	1.00	0.94	0.98	0.91	0.98	0.99	0.99	0.99	1.01	0.93	1.00	0.94	1.00
	0.94	1.00	0.94	1.00	1.14	0.99	1.13	0.99	1.03	0.99	1.00	1.01	1.04	1.00	1.05	1.00
	1.06	1.00	1.04	0.99	0.94	1.00	0.94	1.00	0.93	1.04	0.98	1.02	0.97	1.01	0.97	1.01
31 hour	0.97	1.01	1.01	1.02	1.03	1.00	1.04	1.00	1.01	1.01	1.03	1.00	1.02	1.01	1.03	1.01
	1.01	0.98	0.98	0.99	1.01	1.01	1.04	1.01	0.98	1.04	0.97	1.01	1.13	1.00	1.13	0.99
	0.96	1.01	0.96	1.00	1.08	1.02	1.12	1.02	–	–	–	–	1.00	1.00	0.99	1.00
48 hour	0.92	1.02	0.96	1.02	0.84	1.01	0.85	1.01	1.06	0.99	1.04	1.01	0.94	1.00	0.96	1.00
	0.96	1.00	0.96	1.01	0.98	1.01	0.98	1.00	1.01	0.98	0.99	1.00	1.16	1.00	1.15	1.00
	1.04	1.00	1.04	1.00	0.99	1.00	0.99	1.00	1.01	1.00	1.00	1.00	1.11	1.00	1.11	1.00

Table 31. SBS-I (B6230) degradation ratios on replicates

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
0 hour	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	–	–	–	–
6 hour	1.22	1.02	1.20	0.96	0.92	0.97	0.88	0.97	1.11	0.99	1.07	0.97	1.11	0.99	1.11	0.90
	1.24	1.02	1.22	0.97	0.98	0.97	0.94	0.98	1.08	1.01	1.05	0.98	1.17	0.99	1.15	0.89
	1.18	1.02	1.18	0.97	–	–	–	–	1.05	1.00	1.03	0.98	1.15	0.99	1.13	0.89
24 hour	1.13	1.01	1.07	0.96	0.89	0.98	0.85	0.98	1.24	0.95	1.07	0.93	1.02	1.00	1.02	0.99
	1.11	1.01	1.07	0.96	0.93	0.98	0.91	0.99	1.20	0.95	1.05	0.94	1.02	0.99	1.02	0.90
	1.14	1.02	1.10	0.97	0.88	0.98	0.85	0.99	1.22	0.94	1.05	0.93	1.04	1.00	1.04	0.91
31 hour	1.17	1.01	1.11	0.96	0.94	0.98	0.9	0.98	1.25	0.96	1.10	0.93	1.11	1.00	1.10	0.90
	1.12	1.03	1.09	0.97	0.93	0.96	0.88	0.97	1.27	0.94	1.10	0.93	1.02	1.00	1.02	0.91
	1.15	1.02	1.11	0.96	0.88	0.96	0.83	0.97	–	–	–	–	1.10	1.00	1.10	0.91
48 hour	1.27	1.00	1.17	0.94	0.94	0.98	0.91	0.98	1.28	0.94	1.04	0.90	1.13	1.00	1.13	0.90
	1.21	1.02	1.13	0.95	1.01	0.98	0.98	0.98	1.23	0.94	1.01	0.91	1.09	1.00	1.09	0.91
	1.20	1.01	1.13	0.95	0.95	0.98	0.93	0.98	1.27	0.94	1.04	0.91	1.13	1.00	1.13	0.91

Table 32. SBS-rg (B6231) separation ratios on replicates

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Frequency (rads/s)	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
0 hour	0.86	1.01	0.96	1.03	1.04	1.00	1.04	1.00	0.86	1.02	0.91	1.04	0.99	1.01	1.00	1.00
6 hour	1.23	1.01	1.11	0.98	1.10	1.00	1.11	1.00	0.92	1.01	0.89	1.00	3.89	1.22	6.41	1.17
	0.62	0.98	0.98	0.98	0.90	1.00	0.90	1.01	1.53	1.01	0.94	1.01	0.96	1.00	0.97	1.00
24 hour	1.06	0.99	1.11	0.99	1.03	0.99	1.02	0.99	0.94	0.98	1.02	1.00	0.98	1.00	0.98	1.00
	1.03	0.96	0.97	0.99	0.91	1.01	0.93	1.01	0.99	1.00	0.97	1.00	1.14	1.00	1.14	0.99
31 hour	1.05	0.98	0.97	0.99	1.08	1.00	1.08	1.00	0.92	0.98	0.91	1.02	1.10	1.02	1.15	1.03
	1.09	0.99	1.05	0.97	1.11	0.99	1.10	1.00	1.10	0.98	1.02	0.98	0.99	1.00	1.00	1.00
48 hour	1.00	0.98	0.98	1.00	0.96	1.00	0.96	1.00	0.97	1.00	0.95	1.02	0.99	1.00	0.98	1.00
	1.28	0.99	1.04	0.98	1.15	1.01	1.17	1.00	0.26	0.99	1.02	0.99	0.92	1.00	0.92	1.00

Table 33. SBS-rg (B6231) degradation ratios on replicates

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Frequency (rads/s)	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
0 hour	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
6 hour	1.50	0.95	1.1	0.9	0.99	0.96	0.94	0.96	1.05	1.03	1.14	0.99	0.58	0.91	0.54	0.93
	2.04	0.94	1.16	0.87	0.9	0.96	0.86	0.97	1.38	1.03	1.15	0.99	1.04	1.01	1.05	1.01
24 hour	1.31	0.94	1.05	0.92	0.79	0.97	0.77	0.98	1.19	1.00	1.10	0.98	1.01	1.01	1.03	1.01
	1.31	0.94	1.00	0.91	0.83	0.98	0.82	0.99	1.11	1.00	1.08	0.98	0.92	1.01	0.94	1.01
31 hour	1.31	0.94	0.99	0.91	0.86	0.97	0.84	0.98	1.10	0.99	1.07	0.98	0.79	1.00	0.80	1.00
	1.28	0.94	0.96	0.92	0.85	0.97	0.83	0.98	1.14	1.00	1.11	0.98	1.00	1.01	1.02	1.01
48 hour	1.31	0.94	0.97	0.92	0.84	0.98	0.83	0.99	1.17	0.98	1.10	0.97	0.95	1.01	0.97	1.01
	1.40	0.94	0.99	0.91	0.85	0.98	0.85	0.99	2.29	0.97	1.10	0.96	0.85	1.01	0.86	1.01

Table 34. SBS-rg (B6231) separation ratios on replicate reruns

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		5.0 Rerun		50.0		50.0 Rerun		5.0		5.0 Rerun		50.0		50.0 Rerun	
Temperature	HT		HT		HT		HT		HT		HT		HT		HT	
Time of Sampling	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
0 hour	0.86	1.01	1.19	0.99	0.96	1.03	1.19	0.99	0.86	1.02	1.03	0.99	0.91	1.04	1.02	1.00
6 hour	1.23	1.01	1.12	0.97	1.11	0.98	1.06	0.98	0.92	1.01	1.03	0.99	0.89	1.00	0.99	0.98
	0.62	0.98	1.11	0.98	0.98	0.98	1.05	0.97	1.53	1.01	1.12	0.99	0.94	1.01	1.06	0.97
24 hour	1.06	0.99	1.00	0.98	1.11	0.99	0.96	0.99	0.94	0.98	1.01	1.00	1.02	1.00	1.01	1.00
	1.03	0.96	1.11	0.97	0.97	0.99	1.07	0.99	0.99	1.00	0.97	1.00	0.97	1.00	0.97	1.00
31 hour	1.05	0.98	–	–	0.97	0.99	–	–	0.92	0.98	–	–	0.91	1.02	–	–
	1.09	0.99	–	–	1.05	0.97	–	–	1.10	0.98	–	–	1.02	0.98	–	–
48 hour	1.00	0.98	1.09	0.99	0.98	1.00	1.07	1.00	0.97	1.00	1.03	0.99	0.95	1.02	1.01	1.00
	1.28	0.99	0.98	1.00	1.04	0.98	0.97	1.00	0.26	0.99	0.96	1.00	1.02	0.99	0.96	1.00

Table 35. SBS-rg (B6231) degradation ratios on replicate reruns

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		5.0 Rerun		50.0		50.0 Rerun		5.0		5.0 Rerun		50.0		50.0 Rerun	
Temperature	HT		HT		HT		HT		HT		HT		HT		HT	
Time of Sampling	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
0 hour	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
6 hour	1.50	0.95	1.00	0.99	1.10	0.90	0.83	0.97	1.05	1.03	1.00	1.02	0.58	0.91	0.98	0.98
	2.04	0.94	1.05	0.98	1.16	0.87	0.86	0.97	1.38	1.03	1.06	1.02	1.04	1.01	1.04	0.97
24 hour	1.31	0.94	1.01	0.97	1.05	0.92	0.82	0.97	1.19	1.00	1.13	0.98	1.01	1.01	1.02	0.94
	1.31	0.94	1.04	0.98	1.00	0.91	0.86	0.98	1.11	1.00	1.08	1.00	0.92	1.01	1.01	0.96
31 hour	1.31	0.94	–	–	0.99	0.91	–	–	1.10	0.99	–	–	0.79	1.00	–	–
	1.28	0.94	–	–	0.96	0.92	–	–	1.14	1.00	–	–	1.00	1.01	–	–
48 hour	1.31	0.94	1.06	0.96	0.97	0.92	0.85	0.97	1.17	0.98	1.18	0.95	0.95	1.01	1.01	0.93
	1.40	0.94	1.05	0.96	0.99	0.91	0.85	0.97	2.29	0.97	1.14	0.94	0.85	1.01	0.98	0.93

Table 36. EVA (B6232) separation ratios on replicates

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
0 hour	0.97	0.99	0.93	1.01	1.02	0.99	1.01	1.00	0.93	0.95	0.85	1.02	1.08	1.00	1.09	1.00
6 hour	0.78	1.00	0.78	1.06	0.60	1.04	0.65	1.08	0.98	0.99	0.96	1.01	1.13	1.01	1.12	1.01
	0.58	1.01	0.58	1.11	1.13	0.99	1.11	0.97	0.90	1.00	1.01	1.00	0.97	0.99	0.96	1.00
	0.61	1.01	0.60	1.11	0.56	1.05	0.61	1.08	–	–	–	–	–	–	–	–
24 hour	0.79	1.01	0.81	1.05	0.81	1.01	0.82	1.01	0.92	1.00	0.91	1.03	0.74	1.00	0.74	1.00
	0.69	1.01	0.70	1.07	0.37	1.05	0.41	1.12	1.03	1.00	1.03	1.00	0.93	0.99	0.92	0.99
31 hour	0.50	0.97	0.47	1.13	0.97	0.98	0.95	0.99	–	–	–	–	–	–	–	–
	0.62	1.01	0.63	1.09	0.75	0.99	0.75	1.00	–	–	–	–	–	–	–	–
	0.55	1.01	0.55	1.11	0.33	1.06	0.37	1.13	–	–	–	–	–	–	–	–
48 hour	0.51	1.03	0.50	1.14	0.87	0.98	0.83	0.97	0.88	0.98	0.87	1.03	1.11	1.01	1.15	1.01
	0.63	1.00	0.63	1.09	0.81	1.04	0.85	1.03	0.96	1.27	0.95	1.26	1.12	0.98	1.09	0.97
	0.72	0.97	0.69	1.07	0.85	0.99	0.84	1.00	–	–	–	–	–	–	–	–

Table 37. EVA (B6232) degradation ratios on replicates

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
0 hour	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
6 hour	0.98	1.00	0.98	1.00	0.72	1.03	0.77	1.06	1.09	1.02	1.10	0.98	0.81	1.01	0.85	1.02
	0.41	0.49	0.40	0.52	0.64	1.02	0.68	1.06	1.14	1.02	1.16	0.97	0.92	1.02	0.95	1.03
	–	–	–	–	1.26	0.99	1.23	0.98	–	–	–	–	–	–	–	–
24 hour	0.39	0.50	0.40	0.52	0.90	1.01	0.93	1.02	1.16	1.02	1.18	0.97	0.88	1.00	0.88	1.00
	0.93	1.01	0.95	1.01	1.14	0.99	1.37	0.99	1.20	1.02	1.20	0.96	0.91	1.02	0.93	1.02
	1.09	1.01	1.11	0.98	–	–	–	–	–	–	–	–	–	–	–	–
31 hour	1.24	0.99	1.25	0.98	0.76	1.02	0.80	1.04	–	–	–	–	–	–	–	–
	1.12	1.00	1.13	0.98	0.74	1.02	0.78	1.03	–	–	–	–	–	–	–	–
	1.19	1.01	1.21	0.98	1.56	0.98	1.48	0.97	–	–	–	–	–	–	–	–
48 hour	1.25	0.99	1.26	0.97	0.84	1.00	0.86	1.02	1.20	1.00	1.18	0.96	0.88	1.00	0.89	1.01
	1.19	0.99	1.14	0.94	0.74	1.02	0.78	1.05	1.27	1.01	1.26	0.95	0.94	1.00	0.94	1.00
	1.02	0.99	1.00	1.00	0.74	1.02	0.79	1.05	–	–	–	–	–	–	–	–

Table 38. EVA (B6232) separation ratios on replicate reruns

Conditioning Step	External Heat without Agitation							
	5.0		5.0 Rerun		50.0		50.0 Rerun	
Temperature	HT		HT		HT		HT	
Time of Sampling	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
0 hour	0.97	0.99	–	–	0.93	1.01	–	–
6 hour	0.78	1.00	0.74	1.01	0.78	1.06	0.74	1.00
	0.58	1.01	–	–	0.58	1.11	–	–
	0.61	1.01	–	–	0.60	1.11	–	–
24 hour	0.79	1.01	0.57	1.01	0.81	1.05	0.57	0.98
	0.69	1.01	0.52	1.01	0.70	1.07	0.51	0.98
31 hour	0.50	0.97	–	–	0.47	1.13	–	–
	0.62	1.01	–	–	0.63	1.09	–	–
	0.55	1.01	–	–	0.55	1.11	–	–
48 hour	0.51	1.03	–	–	0.50	1.14	–	–
	0.63	1.00	0.52	1.01	0.63	1.09	0.51	0.99
	0.72	0.97	0.56	1.00	0.69	1.07	0.55	0.97

Table 39. EVA (B6232) degradation ratios on replicate reruns

Conditioning Step	External Heat without Agitation							
	5.0		5.0 Rerun		50.0		50.0 Rerun	
Temperature	HT		HT		HT		HT	
Time of Sampling	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
0 hour	1.00	1.00	–	–	1.00	1.00	–	–
6 hour	0.98	1.00	0.89	0.99	0.98	1.00	0.89	0.98
	0.41	0.49	–	–	0.40	0.52	–	–
	–	–	–	–	–	–	–	–
24 hour	0.39	0.50	1.05	0.99	0.40	0.52	1.05	1.00
	0.93	1.01	1.07	0.99	0.95	1.01	1.07	0.99
	1.09	1.01	–	–	1.11	0.98	–	–
31 hour	1.24	0.99	–	–	1.25	0.98	–	–
	1.12	1.00	–	–	1.13	0.98	–	–
	1.19	1.01	1.04	0.99	1.21	0.98	1.03	0.99
41 hour	1.25	0.99	1.06	0.99	1.26	0.97	1.05	0.99
	1.19	0.99	–	–	1.14	0.94	–	–
	1.02	0.99	–	–	1.00	1.00	–	–

Table 40. EVA-g (B6233) separation ratios on replicates

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Frequency (rads/s)	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
0 hour	0.99	1.00	0.99	1.00	1.03	1.01	1.06	1.01	0.98	1.00	0.97	1.01	1.07	0.98	1.05	0.98
6 hour	0.87	1.01	0.88	1.03	0.79	1.03	0.83	1.04	0.90	1.00	0.94	1.03	0.99	1.00	0.99	1.00
	0.93	1.00	0.94	1.02	0.67	1.02	0.70	1.04	1.01	1.00	1.02	0.99	0.94	0.99	0.93	1.00
24 hour	0.69	1.03	0.75	1.10	0.76	1.04	0.81	1.06	0.87	1.03	0.93	1.04	1.14	1.01	1.16	1.01
	0.81	1.02	0.83	1.05	0.48	1.05	0.52	1.09	0.95	1.00	0.97	1.01	1.04	1.00	1.04	1.00
31 hour	0.78	1.01	0.80	1.06	0.81	0.99	0.80	0.77	0.97	1.00	0.99	1.00	0.94	1.01	0.95	1.01
	0.71	1.01	0.73	1.08	0.58	1.02	0.61	1.06	1.02	1.00	1.02	0.99	1.14	1.00	1.14	1.00
48 hour	0.78	1.01	0.80	1.06	0.45	1.03	0.48	1.06	1.02	1.00	1.02	1.00	0.66	1.00	0.66	0.99
	0.66	1.01	0.70	1.09	0.35	1.11	0.42	1.29	1.03	1.00	1.05	0.99	1.12	1.01	1.13	1.02

Table 41. EVA-g (B6233) degradation ratios on replicates

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Frequency (rads/s)	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
0 hour	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
6 hour	1.04	1.00	1.04	0.99	0.84	1.03	0.88	1.03	0.91	1.00	0.93	1.03	0.94	1.00	0.94	1.00
	1.03	1.00	1.02	0.99	1.00	1.00	1.01	1.01	0.97	1.00	0.97	1.01	0.91	1.01	0.93	1.01
24 hour	1.14	0.99	1.09	0.97	0.88	1.02	0.90	1.03	1.10	0.98	1.05	0.97	0.83	1.00	0.83	1.00
	1.15	0.99	1.11	0.97	1.26	1.00	1.23	0.99	0.98	1.00	0.97	1.01	0.97	0.99	0.97	1.00
31 hour	1.12	0.99	1.07	0.98	0.74	1.01	0.77	1.05	1.05	0.99	1.03	0.98	0.89	1.00	0.90	1.01
	1.17	0.99	1.14	0.97	0.97	1.02	1.01	1.04	1.03	1.00	1.01	0.99	0.87	1.03	0.92	1.04
48 hour	1.14	0.99	1.11	0.96	1.16	0.99	1.14	1.00	1.09	0.99	1.04	0.98	0.84	0.99	0.83	1.00
	1.27	0.99	1.22	0.95	1.34	0.99	1.29	1.00	1.07	0.99	1.04	0.98	0.90	1.00	0.92	1.01

Table 42. EVA-g (B6233) separation ratios on replicate reruns

Conditioning Step	External Heat without Agitation							
	5.0		5.0 Rerun		50.0		50.0 Rerun	
Temperature	HT		HT		HT		HT	
Time of Sampling	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
0 hour	1.03	1.01	1.06	1.00	1.06	1.01	1.06	1.00
6 hour	0.79	1.03	0.89	1.02	0.83	1.04	0.90	1.02
	0.67	1.02	0.80	1.02	0.70	1.04	0.83	1.02
24 hour	0.76	1.04	0.82	1.04	0.81	1.06	0.84	1.02
	0.48	1.05	0.59	1.04	0.52	1.09	0.63	1.08
31 hour	0.81	0.99	1.09	0.98	0.80	0.77	1.72	0.96
	0.58	1.02	0.34	1.09	0.61	1.06	0.39	1.15
48 hour	0.45	1.03	0.53	1.04	0.48	1.06	0.58	1.08
	0.35	1.11	0.26	1.08	0.42	1.29	0.31	1.18

Table 43. EVA-g (B6233) degradation ratios on replicate reruns

Conditioning Step	External Heat without Agitation							
	5.0		5.0 Rerun		50.0		50.0 Rerun	
Temperature	HT		HT		HT		HT	
Time of Sampling	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
0 hour	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
6 hour	0.84	1.03	0.84	1.02	0.88	1.03	0.88	1.04
	1.00	1.00	1.02	1.02	1.01	1.01	1.05	1.02
24 hour	0.88	1.02	0.65	1.03	0.90	1.03	0.70	1.07
	1.26	1.00	0.85	1.08	1.23	0.99	0.90	1.05
31 hour	0.74	1.01	0.71	1.02	0.77	1.05	0.61	1.06
	0.97	1.02	1.29	1.00	1.01	1.04	1.27	1.01
48 hour	1.16	0.99	0.87	1.01	1.14	1.00	0.89	1.03
	1.34	0.99	1.33	1.00	1.29	1.00	1.29	1.01

Table 44. ESI (B6243) separation ratios on replicates

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
0 hour	1.02	1.00	1.02	1.00	1.17	1.00	1.16	0.99	0.96	1.00	0.96	1.01	0.99	1.00	0.99	0.99
6 hour	0.95	1.00	0.95	1.02	0.69	1.05	0.76	1.09	0.93	1.02	0.99	1.01	1.07	1.04	1.17	1.07
	–	–	–	–	1.22	0.98	1.17	0.96	0.86	1.08	0.95	1.03	0.98	1.01	0.99	1.00
	–	–	–	–	–	–	–	–	1.03	0.99	1.02	0.99	0.79	1.02	0.84	1.05
24 hour	1.01	1.00	1.00	1.01	0.70	1.03	0.75	1.06	1.02	0.98	0.98	1.00	0.84	0.99	0.85	1.01
	0.99	1.00	0.99	1.00	–	–	–	–	0.95	1.01	0.98	1.01	0.97	0.99	0.97	1.00
	–	–	–	–	–	–	–	–	0.99	0.97	0.98	1.00	1.05	1.01	1.06	1.00
31 hour	0.92	1.01	0.94	1.03	0.73	1.05	0.80	1.07	1.00	1.01	1.00	1.00	1.09	0.97	1.04	0.96
	0.91	1.00	0.90	1.03	0.65	1.05	0.70	1.06	1.01	1.00	1.00	1.00	1.11	0.99	1.14	0.98
	–	–	–	–	–	–	–	–	1.03	1.00	1.01	1.00	0.87	1.00	0.87	1.01
48 hour	0.99	1.00	0.99	1.00	0.76	1.03	0.79	1.05	1.02	1.00	1.03	0.99	0.78	1.01	0.80	1.02
	1.03	0.99	0.97	1.01	0.97	0.99	0.95	0.99	1.05	0.99	1.01	0.99	0.76	1.01	0.80	1.02
	–	–	–	–	–	–	–	–	1.05	1.00	1.03	0.99	0.96	1.02	0.99	1.02

Table 45. ESI (B6243) degradation ratios on replicates

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
0 hour	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
6 hour	1.01	1.00	1.00	1.00	1.16	0.98	1.13	0.98	1.05	0.99	1.03	0.99	0.88	1.00	0.89	0.98
	–	–	–	–	1.20	0.98	1.15	0.97	1.14	0.96	1.05	0.97	0.95	1.01	0.98	1.00
	–	–	–	–	–	–	–	–	1.25	0.98	1.07	0.97	0.84	1.01	0.87	1.01
24 hour	1.05	0.98	1.10	0.66	1.08	0.99	1.05	0.99	1.21	0.96	1.07	0.96	0.81	1.01	0.85	1.02
	1.03	0.98	1.08	0.66	–	–	–	–	1.10	1.00	1.08	0.97	0.70	1.03	0.76	1.05
	–	–	–	–	–	–	–	–	1.16	0.98	1.07	0.97	0.93	1.01	0.95	0.99
31 hour	1.01	1.00	1.01	0.99	1.16	0.98	1.12	0.98	1.07	1.00	1.05	0.98	0.85	1.01	0.88	1.01
	1.05	1.00	1.06	0.98	1.22	0.98	1.16	0.97	1.09	1.00	1.07	0.97	0.84	1.01	0.87	1.01
	–	–	–	–	–	–	–	–	1.12	1.00	1.10	0.96	0.86	1.01	0.88	1.01
48 hour	1.15	1.00	1.11	0.95	1.05	0.98	1.02	0.98	1.15	1.00	1.12	0.96	1.01	0.99	1.02	0.99
	1.21	1.00	1.16	0.94	1.03	0.97	0.99	0.98	1.24	1.00	1.18	0.94	0.85	1.00	0.87	1.00
	–	–	–	–	–	–	–	–	1.15	1.01	1.14	0.95	1.05	1.00	1.06	0.99

Table 46. CMCRA (B6251) separation ratios on replicates

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Frequency (rads/s)	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
0 hour	0.98	1.00	0.97	1.00	1.12	1.00	1.13	1.00	1.08	1.00	1.10	0.99	1.02	1.00	1.02	0.99
6 hour	0.84	1.02	0.90	1.05	1.12	1.02	1.14	1.01	1.08	0.99	1.07	0.98	0.98	1.01	0.99	1.01
	0.84	1.01	0.90	1.04	1.05	1.01	1.05	1.00	1.01	1.00	1.00	1.00	1.03	1.00	1.03	1.03
24 hour	–	–	–	–	1.04	1.00	1.05	1.00	1.08	0.97	1.07	0.98	0.93	1.00	0.94	1.00
	–	–	–	–	–	–	–	–	0.99	1.00	0.98	1.00	0.94	1.00	0.94	1.00
	–	–	–	–	–	–	–	–	0.98	1.00	1.03	1.00	1.18	1.00	1.19	1.00
31 hour	–	–	–	–	–	–	–	–	0.99	1.00	1.02	1.00	1.02	1.00	1.03	1.00
	0.61	1.09	0.78	1.14	1.26	1.00	1.26	0.99	0.88	1.01	0.90	1.04	0.85	1.00	0.85	1.00
	0.72	1.07	0.89	1.10	1.21	1.01	1.21	1.01	0.99	1.00	0.99	1.00	0.97	1.01	0.97	1.01
48 hour	0.68	1.07	0.84	1.11	1.14	1.01	1.14	1.00	–	–	–	–	0.99	1.00	0.98	1.00
	0.72	1.08	0.89	1.09	1.23	1.00	1.23	0.99	0.98	1.00	0.97	1.01	0.96	1.00	0.96	1.00
	0.64	1.11	0.92	1.12	1.12	1.00	1.12	0.99	1.05	1.00	1.05	0.99	0.93	1.00	0.92	1.00
	0.72	1.08	0.87	1.10	1.12	1.00	1.11	1.00	0.99	1.00	1.02	1.00	1.07	1.00	1.07	1.00

Table 47. CMCRA (B6251) degradation ratios on replicates

Conditioning Step	External Heat without Agitation								External Heat with Mechanical Agitation							
	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Frequency (rads/s)	5.0		50.0		5.0		50.0		5.0		50.0		5.0		50.0	
Temperature	HT		HT		IT		IT		HT		HT		IT		IT	
Time of Sampling	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
0 hour	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
6 hour	0.69	1.02	0.82	1.11	1.13	1.01	1.12	0.99	1.02	1.01	1.00	0.98	0.96	1.00	0.95	0.99
	0.90	0.98	0.93	1.03	1.14	1.00	1.13	0.99	1.02	1.01	1.01	0.99	0.98	1.00	0.97	1.00
24 hour	–	–	–	–	1.18	1.00	1.17	0.99	1.04	1.00	1.00	0.98	0.96	1.00	0.95	1.00
	–	–	–	–	–	–	–	–	1.07	1.00	1.00	0.97	0.93	1.00	0.91	1.00
	–	–	–	–	–	–	–	–	1.14	1.00	1.08	0.96	1.01	1.00	1.00	1.00
31 hour	–	–	–	–	–	–	–	–	1.09	1.02	1.07	0.97	0.94	1.00	0.93	0.99
	1.03	0.96	0.99	1.00	1.06	0.99	1.04	0.99	1.27	1.01	1.20	0.93	0.97	0.99	0.96	0.99
	1.01	0.97	1.03	1.00	1.17	1.00	1.14	0.99	1.00	0.99	0.91	0.69	1.00	0.99	0.98	0.99
48 hour	0.95	0.99	0.96	1.02	1.20	0.99	1.17	0.98	–	–	–	–	1.01	0.99	0.99	0.99
	1.02	0.97	0.99	1.00	1.07	0.99	1.04	0.98	1.24	1.01	1.14	0.94	1.01	0.99	0.98	0.99
	1.05	0.96	0.99	1.00	1.13	0.99	1.10	0.98	1.21	1.01	1.15	0.94	0.95	0.99	0.93	0.99
	1.00	0.96	0.95	1.00	1.23	0.99	1.20	0.98	1.25	1.01	1.16	0.93	0.94	0.99	0.92	0.99

Table 48. CMCRA (B6251) separation ratios on replicate reruns

Conditioning Step	External Heat without Agitation							
	5.0		5.0 Rerun		50.0		50.0 Rerun	
Temperature	HT		HT		HT		HT	
Time of Sampling	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ	RS G*	RS δ
0 hour	0.98	1.00	1.04	1.00	0.97	1.00	1.04	1.00
6 hour	0.84	1.02	0.90	1.03	0.90	1.05	0.96	1.03
	0.84	1.01	0.91	1.01	0.90	1.04	0.94	1.02
24 hour	–	–	–	–	–	–	–	–
	–	–	–	–	–	–	–	–
	–	–	–	–	–	–	–	–
31 hour	0.61	1.09	–	–	0.78	1.14	–	–
	0.72	1.07	–	–	0.89	1.10	–	–
	0.68	1.07	–	–	0.84	1.11	–	–
48 hour	0.72	1.08	1.03	1.01	0.89	1.09	1.06	1.01
	0.64	1.11	0.85	1.03	0.92	1.12	0.90	1.03
	0.72	1.08	–	–	0.87	1.10	–	–

Table 49. CMCRA (B6251) degradation ratios on replicate reruns

Conditioning Step	External Heat without Agitation							
	5.0		5.0 Rerun		50.0		50.0 Rerun	
Temperature	HT		HT		HT		HT	
Time of Sampling	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ	Rd G*	Rd δ
0 hour	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
6 hour	0.69	1.02	0.99	0.98	0.82	1.11	0.97	0.99
	0.90	0.98	0.92	0.99	0.93	1.03	0.91	1.00
24 hour	–	–	–	–	–	–	–	–
	–	–	0.89	1.00	–	–	0.92	1.02
	–	–	0.93	0.99	–	–	0.93	1.01
31 hour	1.03	0.96	–	–	0.99	1.00	–	–
	1.01	0.97	–	–	1.03	1.00	–	–
	0.95	0.99	–	–	0.96	1.02	–	–
48 hour	1.02	0.97	–	–	0.99	1.00	–	–
	1.05	0.96	–	–	0.99	1.00	–	–
	1.00	0.96	–	–	0.95	1.00	–	–

APPENDIX C

Data of Separation Rates and Degradation Rates and Rate Reruns

Table 50. Data of separation rates and degradation rates

	Without Mechanical Agitation				With Mechanical Agitation					
	T _{cs}	K _{si}		T _{ed}	K _{di}	T _{cs}	K _{si}	T _{ed}	K _{di}	
Elvaloy (B6228)	N/A	N/A		N/A	N/A	N/A	N/A	N/A	N/A	
SBS-lg (B6229)	24 hour	Top = 0.0381		31 hour	Top = 0.0416		N/A	N/A	48 hour	
		Bottom = 0.0252			Bottom = 0.0409				Top = 0.262	
SBS-l (B6230)	N/A	N/A		48 hour	Top = 0.0238		N/A	N/A	24 hour	
		N/A			Bottom = 0.0295				Top = 0.0507	
SBS-rg (B6231)	N/A	N/A		6 hour	Top = 0.305		N/A	N/A	6 hour	
		N/A			Bottom = 0.214				Top = 0.170	
EVA (B6232)	6 hour	Top = 0.09		6 hour	Top = 0.15		N/A	N/A	48 hour	
		Bottom = 0.15			Bottom = 0.18				Top (5) = 0.02	
EVA-g (B6233)	6 hour	Top (5) = 0.12		48 hour	Top = 0.015		N/A	N/A	N/A	
		Top (50) = 0.042			Bottom = 0.034				Bottom (5) = 0.03	
ESI (B6243)	6 hour	Top (5) = 0.15		24 hour	Top = 0.03		N/A	N/A	N/A	
		Top (50) = 0.089			Bottom = 0.04				Bottom (5) = 0.10	
CMCRA (B6251)	31 hour	Top = 0.025		6 hour	Top = 0.138		N/A	N/A	48 hour	
		Bottom = 0.041			Bottom = 0.161				Top = 0.025	

Table 51. Data of separation rates and degradation rate reruns

	Without Mechanical Agitation				With Mechanical Agitation				
	T_{cs}	K_{si}		T_{cd}	K_{di}	T_{cs}	K_{si}	T_{cd}	K_{di}
Elvaloy (B6228)	N/A	N/A		N/A	N/A	N/A	N/A	N/A	N/A
SBS-lg (B6229)	N/A	N/A		N/A	N/A	N/A	N/A	N/A	N/A
SBS-l (B6230)	N/A	N/A		6 hour	Top = 0.0238 Bottom = 0.0295	N/A	N/A	24 hour	Top = 0.0507 Bottom = 0.0547
SBS-rg (B6231)	N/A	N/A		N/A	N/A	N/A	N/A	N/A	N/A
EVA (B6232)	6 hour	Top = 0.09 Bottom = 0.15		6 hour	Top = 0.15 Bottom = 0.18	N/A	N/A	N/A	N/A
EVA-g (B6233)	6 hour	Top (5) = 0.12 Bottom (5) = 0.16	Top (50) = 0.042 Bottom (50) = 0.042	48 hour	Top = 0.015 Bottom = 0.034	N/A	N/A	N/A	N/A
ESI (B6243)	24 hour	Top (5) = 0.15 Bottom (5) = 0.10	Top (50) = 0.089 Bottom (50) = 0.010	24 hour	Top = 0.03 Bottom = 0.04	N/A	N/A	N/A	N/A
CMCRA (B6251)	N/A	N/A		N/A	N/A	N/A	N/A	N/A	N/A

ACKNOWLEDGMENTS

The authors would like to thank the AASHTO Task Force on the SHRP Implementation for the financial support rendered for this NCHRP Project No. 90-04.

REFERENCE

1. Bahia, H. U., Hanson, D. I., Zeng, M., Zhai, H., Khatri, M. A. and R. M. Anderson, "Characterization of Modified Asphalt Binders in Superpave Mix Design," *NCHRP Report 459*, Transportation Research Board, National Research Council, National Academy Press, Washington DC (2001).

ADDITIONAL SOURCES

Beigen, J. R. and A. W. Czanderna, "Analysis of Thermal Processes: The Exponential Integral," *Journal of Thermal Analysis*, Vol. 4 (1972), p. 39.

Billington, N. C. and P. D. Calvert, "The Physical Chemistry of Oxidation and Stabilization of Polyolefins," Chapter 5 in *Developments in Polymer Stabilization-3*, G. Scott (ed.), Applied Science Publishers, London (1980) pp. 139-190.

Brule, B., Ramond, G. and C. Such, "Relationship Between Composition, Structure and Properties of Road Asphalts: State of Research at the French Public Works Central Laboratory," *Transportation Research Record 1096*, Transportation Research Board, National Research Council, National Academy Press, Washington, DC (1986), pp. 22-34.

Collins, J. M. and M. G. Bouldin, "Long and Short Term Stability of Straight and Polymer Modified Asphalts," *Rubber World* (August 1992).

Davies, A. and D. Gordon, "Rapid Assessment of Weathering Stability from Exposure of Polymers," *Journal of Applied Polymer Science*, Vol. 18 (1974), p. 1159.

Gilroy, H. M., "Long-Term Photo- and Thermal Oxidation of Polyethylene," *American Chemical Society Symposium Series*, Vol. 95 (1979), p. 63.

Hesp, S. A. M. and R. T. Woodhams, "Stabilization Mechanisms in Polyolefin-Asphalt Emulsions," *Polymer Modified Asphalt Binders*, K. R. Wardlaw and S. Shuler (eds.), American Society for Testing and Materials, Philadelphia, PA (1992), pp. 1-19.

Hesp, S. A. M. and R. T. Woodhams, "Asphalt-Polyolefin Emulsion Breakdown," *Colloid and Polymer Science*, Vol. 269 (8), 1991 pp. 825-834.

Khudyakova, T. S., Rozenhal, D. A., Mal'kova, S., and T. I. Volkov, "Effect of Heat Treatment on the Structure of Polyethylene-Bitumen Compositions," *Stroit Mater*, Vol. 11 (1981), p. 30.

Melik, D. H. and H. S. Folger, "Fundamentals of Colloidal Stability in Quiescent Media," *Encyclopedia of Emulsion Technology*, Vol. 3, Chapter 1, P. Becher (ed.), Marcel Dekker, New York, NY (1988).

Phromsorn, C. J. and T. W. Kennedy, "Evaluation of Laboratory Methods Simulating Aging Effects of Asphalt Binder," *Transportation Research Record 1488*, Transportation Research Board, National Research Council, Washington, DC (1995), pp. 13-20.

Ranby, B. and J. F. Rabek, *Photodegradation, Photo-oxidation and Photostabilization of Polymers*, John Wiley and Sons, London, U.K. (1975).

Schnabel, W., *Polymer Degradation*, Hanser Publishers, Munich, Germany (1982).

Shelton, J. R., "Stabilization Against Thermal Oxidation," in *Polymer Stabilization*, W. L. Hawkins (ed.), Wiley-Interscience, New York, NY (1972).

Yoon, H. H., Tarrer, A. R. and V. P. Wagh, "Thermal Degradation of Antistripping Agents and Enhanced Performance by Curing," *Journal of Materials in Civil Engineering*, Vol. 5 (1) (1993), pp. 1-18.

