NATIONAL BUREAU OF STANDARDS REPORT

7420

AIR OXIDATION OF ASPHALTS

by

Sidney H. Greenfeld

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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and

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U. S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

AIR OXIDATION OF ASPHALTS

1. INTRODUCTION

Prior to 1961, all asphalts used in the ARIB project at the National Bureau of Standards were supplied from outside sources. The descriptions and identifications were frequently incomplete and not very well authenticated. The precise processing conditions were never made known. It became quite obvious that when completely identified coating-grade asphalts were required, they would have to be produced in the laboratory.

In addition to the above considerations, it was found [1][1/] that the durability of asphalts could be appreciably changed through the use of various additives during the blowing process. It was felt that the durability of coating-grade asphalts might vary with some of the blowing variables, as well as with the use of additives. A program was designed to encompass a study of the blowing process and the relations that exist between the variables in this process and the durability of the asphalts produced.

This report covers the first phase of this work, in which the effects of blowing temperature, air rate and degree of agitation on the blowing time and asphalt properties were investigated.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

Two 2-liter stainless steel oxidation (blowing) stills were constructed. The assembled stills are shown in Figure 1. An expanded view of still No. 1, the one of the left, is shown in Figure 2. This still was the prototype and was used for a number of runs before the second still was constructed. As seen in Figure 2, the body of the still was made from a piece of 4-inch O.D. stainless steel pipe, 21 inches long. An impeller containing three triple-bladed turbo-wheels (open on the bottom side) was used to agitate the hot asphalt and disperse the air, which entered through the long "J" shaped 3/8-in. O.D. stainless steel tube to the right of the impeller assembly. The impellers could be run at 900, 1400, 2200 and 3250 r.p.m.

At the base of the still, a drain valve is on the left and a thermometer well on the right. Approximately 15 inches up on the right is a fitting for the introduction of steam. The exhaust opening for the still consists of a 45°, 1/2-inch i.p.s. stainless steel elbow on the top of the impeller assembly.

[1/] Numbers in brackets refer to literature references at the end of this report.

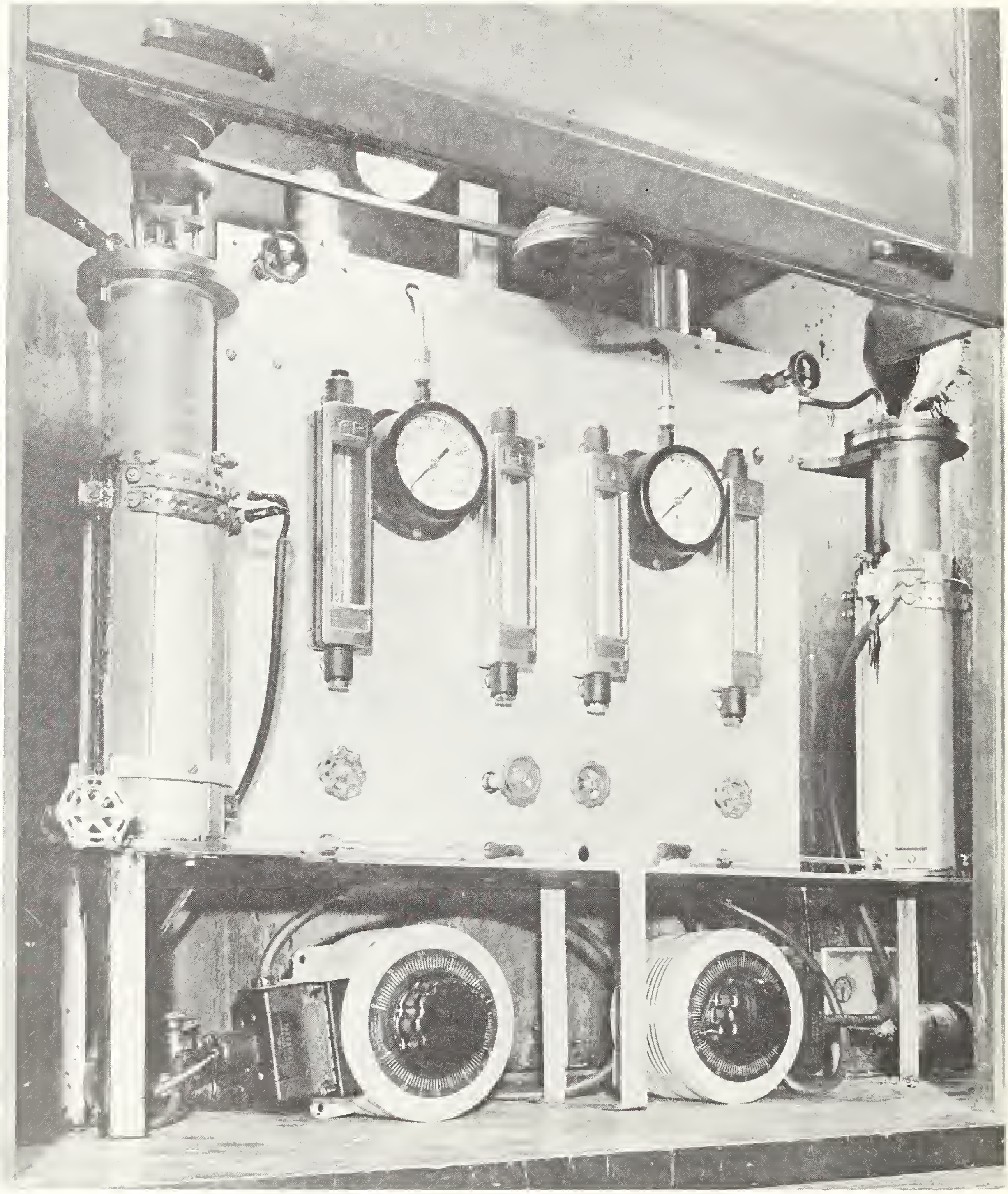


FIGURE 1. BLOWING STILLS

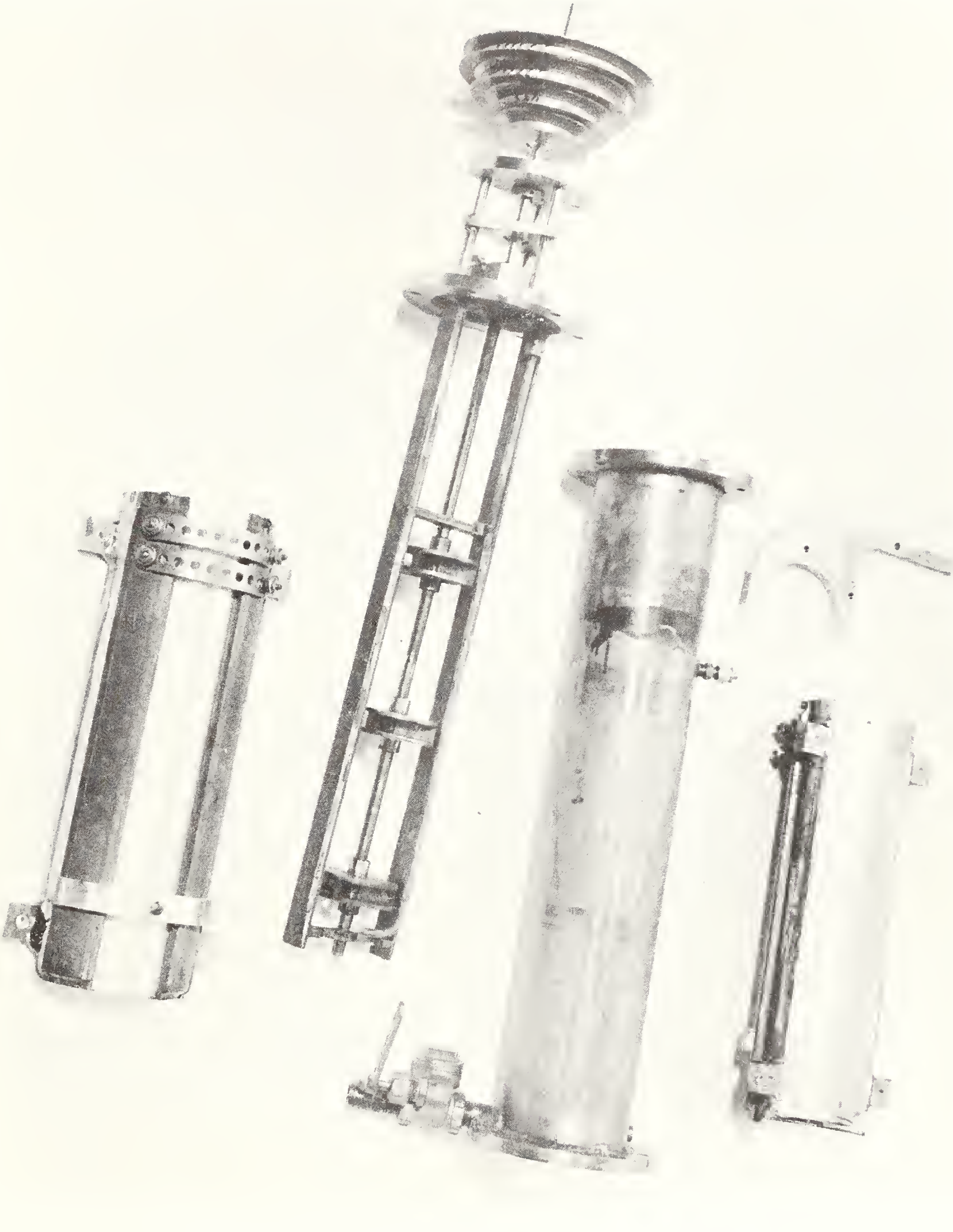


FIGURE 2. A DISASSEMBLED VIEW OF STILL NO. 1.

The still is heated by four 500-watt strip heaters fastened to an aluminum jacket, which in turn is clamped on the stainless steel still body.

The still on the right is essentially a mirror image of the one on the left. However, it has a second head (shown in Figure 1) to replace the impeller assembly. This head contains no impellers, but an "X" shaped air distributor containing 12 small uniformly spaced holes on the lower side of the "X" member. The holes have an aggregate area 1.2 times the internal area of the 3/8-inch air-feed line. The two stills can be operated simultaneously.

As seen in Figure 1, there are two rotameters to each still. The rotameter closest to each still meters filtered, room-temperature air. The second rotameter on each still is for metering other gases to be blended with the air. The pressure gages read "zero" most of the time; however, when carbonization occurs, a positive pressure appears and the stills are cleaned. (Cleaning is accomplished by heating the still components in a muffle furnace at 1000°F. and burning off the residue.)

The voltage to the heating elements is manually regulated by the variable transformers under the still base.

2.2 Materials

The following fluxes are being investigated:

<u>Talco - American Petrofina Co. of Texas</u>

Two Grades -	180/200	85/100
S.P., °F	104	120
Pen. at 32, dmm	44	25
Pen. at 77, dmm	190	92
Pen. at 115, dmm	TS	TS
Sp. Gr. at 60/60°F	1.025	1.032
Ductility at 77°F, 5 cm/min., cm	100+	100+
Flash Point (COC), °F	555	560

<u>Tia-Juana Medium - Cities Service Oil Co.</u>

Two Grades -	C-612-TS	C-611-TS
S.P., °F	104	111
Pen. at 32, dmm	44	25
Pen. at 77, dmm	152	91
Pen. at 115, dmm	222	232
Flash Point (COC), °F	575	620

California Coastal - American Asphalt & Bitumuls Co.

One Grade -	
S.P., °F	122
Pen. at 32°F, dmm	20
Pen. at 77°F, dmm	64
Pen. at 115°F, dmm	TS
Flash Point (COC), °F	450

Kansas - Skelly Oil Co.

Two Grades -	L22	L27
S.P., °F	101	120
Pen. at 32°F, dmm	44	18
Pen. at 77°F, dmm	237	80
Pen. at 115°F, dmm	TS	TS
Ductility, 5 cm/min. at 77, cm.	93	135+
Sp. Gr. at 77°F	0.9949	1.0041
Flash Point (COC), °F	635	645

All except the California flux were supplied in two grades, the softer of which could be blown to coating-grade specifications and the harder of which required modification to be blown into grade, but was the softest material that could be supplied complying with the 450°F minimum flash point requirement.

3. PROCEDURE

Two thousand grams of flux were heated in a closed gallon can in an air oven to about 300°F. while the still was being preheated to about 400°F. The flux was poured into the still through a funnel. The quantity put into the still was determined by difference, i.e., the funnel and gallon can were weighed before and after the flux was poured into the still. The temperature of the still was raised to the blowing temperature desired and the air was turned on.

Periodically, samples were taken and softening points determined. The air was turned off while samples were taken and immediately turned on again afterwards. When the expected softening point was over about 200°F, the air was kept off during the softening-point determination to avoid over-blowing. The still was drained into two penetration cans and five pint cans (partially full) when the run was complete.

The asphalt in all of the cans was weighed (the intermediate samples before softening point rings were poured) and the losses determined by difference.

Penetrations and asphaltene contents were determined on the final product.

4. RESULTS

The blowing conditions and characteristics of the coating-grade asphalts produced are summarized in Tables 1, 2, and 3. Table 1 contains the data on the effect of blowing temperature in the range of 435 to 530°F. Table 2 contains the data on the effect of air rate in the range of 2400 to 9500 ml/min., corresponding to 38 to 152 ft^3/ton min. Table 3 contains the data on the effect of agitation in the range of 0 to 2200 r.p.m. At speeds greater than 2200 r.p.m., the asphalt was blown out through the vent.

Figures 3 to 6 contain the blowing curves, showing the increase of softening point with time as a function of temperature of blowing for California, Talco, Tia Juana and Kansas asphalts, respectively.

Figures 7 and 8 contain the blowing curves as functions of air rate.

Figure 9 contains the blowing curves as functions of degree of agitation for all four asphalts.

It is customary to present the change in penetration with softening point as blowing progresses, but since coating-grade asphalts are the principle concern of this project, the penetrations on the intermediate points were not determined (to conserve time).

Figures 10-12 contain the curves showing the variation of blowing time with blowing temperature, air rate, and degree of agitation, respectively.

5. DISCUSSION OF RESULTS

The four asphalts investigated behaved differently during the blowing process. Each will be discussed separately, where necessary, under each of the three variables studied and then the four asphalts will be compared and contrasted. Accelerated weathering data are not complete on any of the asphalts and will not be discussed.

TABLE 1. ASPHALT CHARACTERISTICS

EFFECT OF TEMPERATURE

Run No.	Flux Identification	Blowing Conditions Time, min.	Temp., °F	Air Rate ml/min.	Agitation, r.p.m.	S.P. °F	Penetrations at 32°F	at 77°F	at 115°F	Asphaltenes, %
B38	Calif. Coastal	84	439	4750	1400	221	7	15	28	43.1
B37	" "	69	483	4750	1400	221	7	12	26	42.7
B39	" "	64	531	4750	1400	223	6	10	22	44.0
B56	" "	390	437	4750	0	219	5	10	22	42.8
B55	" "	300	482	4750	0	223	4	9	17	43.7
B52	" "	225	518	4750	0	225	3	6	12	44.3
B15	Talco 175/200	87	432	4750	1400	217	9	21	34	39.2
B16	" "	65	478	4750	1400	217	13	20	34	39.7
B17	" "	60	530	4750	1400	226	10	16	28	40.7
B108	" "	919	436	4750	0	225	9	16	28	41.1
B105	" "	600	472	4750	0	219	11	17	33	40.0
B111	" "	453	526	4750	0	223	10	16	26	41.6
B79	Tia Juana 612	76	439	4750	1400	217	14	22	38	34.6
B78	" "	64	475	4750	1400	217	13	21	37	35.2
B75	" "	52	529	4750	1400	219	12	20	37	35.2
B80	" "	670	435	4750	0	217	12	20	37	35.2
B76	" "	390	470	4750	0	218	13	21	38	34.3
B77	" "	270	521	4750	0	217	12	19	34	35.2
B93	Kansas L22	110	437	4750	1400	221	12	19	35	33.2
B85	" "	81	473	4750	1400	219	12	21	36	34.5
B94	" "	46	526	4750	1400	217	13	22	38	32.6
B98	" "	690	437	4750	0	220	12	20	34	34.1
B86	" "	562	473	4750	0	217	12	21	34	33.8
B97	" "	295	526	4750	0	219	12	21	32	33.8

TABLE 2. ASPHALT CHARACTERISTICS

EFFECT OF AIR RATE

Run No.	Flux Identification	Time, min.	Blowing Conditions: Air Rate ml/min.	Blowing Conditions: Temp., °F	Blowing Conditions: Agitation, r.p.m.	S.P. °F	Penetrations: at 32°F	Penetrations: at 77°F	Penetrations: at 115°F	Asphaltenes, %
B43	Calif. Coastal	117	2400	480	1400	223	6	14	25	42.5
B37	" "	69	4750	483	1400	221	7	12	26	42.7
B42	" "	61	9500	479	1400	223	6	16	25	42.7
B116	" "	810	2400	472	0	223	4	8	18	44.7
B55	" "	300	4750	482	0	223	4	9	17	43.7
B60	" "	240	9500	468	0	217	4	8	18	44.1
B20	Talco 175/200	107	2400	471	1400	217	14	23	33	40.1
B16	" "	65	4750	478	1400	217	13	20	34	39.7
B19	" "	55	9500	473	1400	219	12	20	32	40.4
B102	" "	1200	2400	473	0	217	11	18	33	40.9
B105	" "	600	4750	472	0	219	11	17	33	40.0
B100	" "	317	9500	471	0	219	12	19	36	40.0
B96	Tia Juana 612	96	2400	472	1400	217	12	22	35	35.4
B78	" "	64	4750	475	1400	217	13	21	37	35.2
B95	" "	56	9500	472	1400	217	13	22	37	35.1
B114	" "	915	2400	472	0	221	12	19	33	36.8
B76	" "	390	4750	470	0	218	13	21	38	34.3
B84	" "	206	9500	472	0	217	14	22	39	35.6
B91	Kansas L22	119	2400	472	1400	222	13	20	34	32.8
B85	" "	81	4750	473	1400	219	12	21	36	34.5
B90	" "	68	9500	472	1400	221	12	20	34	33.4
B92	" "	944	2400	472	0	217	13	20	35	33.3
B86	" "	562	4750	473	0	217	12	21	34	33.8
B89	" "	330	9500	475	0	221	11	19	29	34.6

TABLE 3. ASPHALT CHARACTERISTICS.

<u>EFFECT OF AGITATION</u>

Run No.	Flux Identification	Blowing Conditions Time, min.	Agitation, r.p.m.	Temp., °F	Air Rate ml/min.	S.P. °F	Penetrations at 32°F	at 77°F	at 115°F	Asphaltenes, %
B55	Calif. Coastal	300	0	482	4750	223	4	9	17	43.7
B40	" "	104	900	484	4750	221	7	12	24	42.9
B37	" "	69	1400	483	4750	221	7	12	26	42.7
B41	" "	42	2200	479	4750	217	7	14	27	42.0
B165	Talco 175/200	600	0	473	4750	219	11	17	33	40.0
B18	" "	125	900	472	4750	219	10	20	29	40.4
B16	" "	65	1400	478	4750	217	13	20	34	39.7
B99	" "	56	2200	472	4750	217	14	21	36	39.6
B76	Tia Juana 612	390	0	470	4750	218	13	21	38	34.3
B82	" "	124	900	470	4750	221	12	19	34	35.4
B78	" "	64	1400	475	4750	217	13	21	37	35.2
B83	" "	52	2200	472	4750	219	12	20	36	35.6
B86	Kansas L22	562	0	473	4750	217	12	20	34	33.8
B88	" "	124	900	473	4750	219	12	22	36	32.4
B85	" "	81	1400	473	4750	219	12	21	36	34.5
B87	" "	55	2200	473	4750	220	12	22	36	32.9

TABLE 3. ASPHALT CHARACTERISTICS.

EFFECT OF AGITATION

Run No.	Flux Identification	Blowing Conditions Time, min.	Agitation, r.p.m.	Temp., °F	Air Rate ml/min.	S.P. °F	Penetrations at 32°F	at 77°F	at 115°F	Asphaltenes, %
B55	Calif. Coastal	300	0	482	4750	223	4	9	17	43.7
B40	" "	104	900	484	4750	221	7	12	24	42.9
B37	" "	69	1400	483	4750	221	7	12	26	42.7
B41	" "	42	2200	479	4750	217	7	14	27	42.0
B165	Talco 175/200	600	0	473	4750	219	11	17	33	40.0
B18	" "	125	900	472	4750	219	10	20	29	40.4
B16	" "	65	1400	478	4750	217	13	20	34	39.7
B99	" "	56	2200	472	4750	217	14	21	36	39.6
B76	Tia Juana 612	390	0	470	4750	218	13	21	38	34.3
B82	" "	124	900	470	4750	221	12	19	34	35.4
B78	" "	64	1400	475	4750	217	13	21	37	35.2
B83	" "	52	2200	472	4750	219	12	20	36	35.6
B86	Kansas L22	562	0	473	4750	217	12	20	34	33.8
B88	" "	124	900	473	4750	219	12	22	36	32.4
B85	" "	81	1400	473	4750	219	12	21	36	34.5
B87	" "	55	2200	473	4750	220	12	22	36	32.9

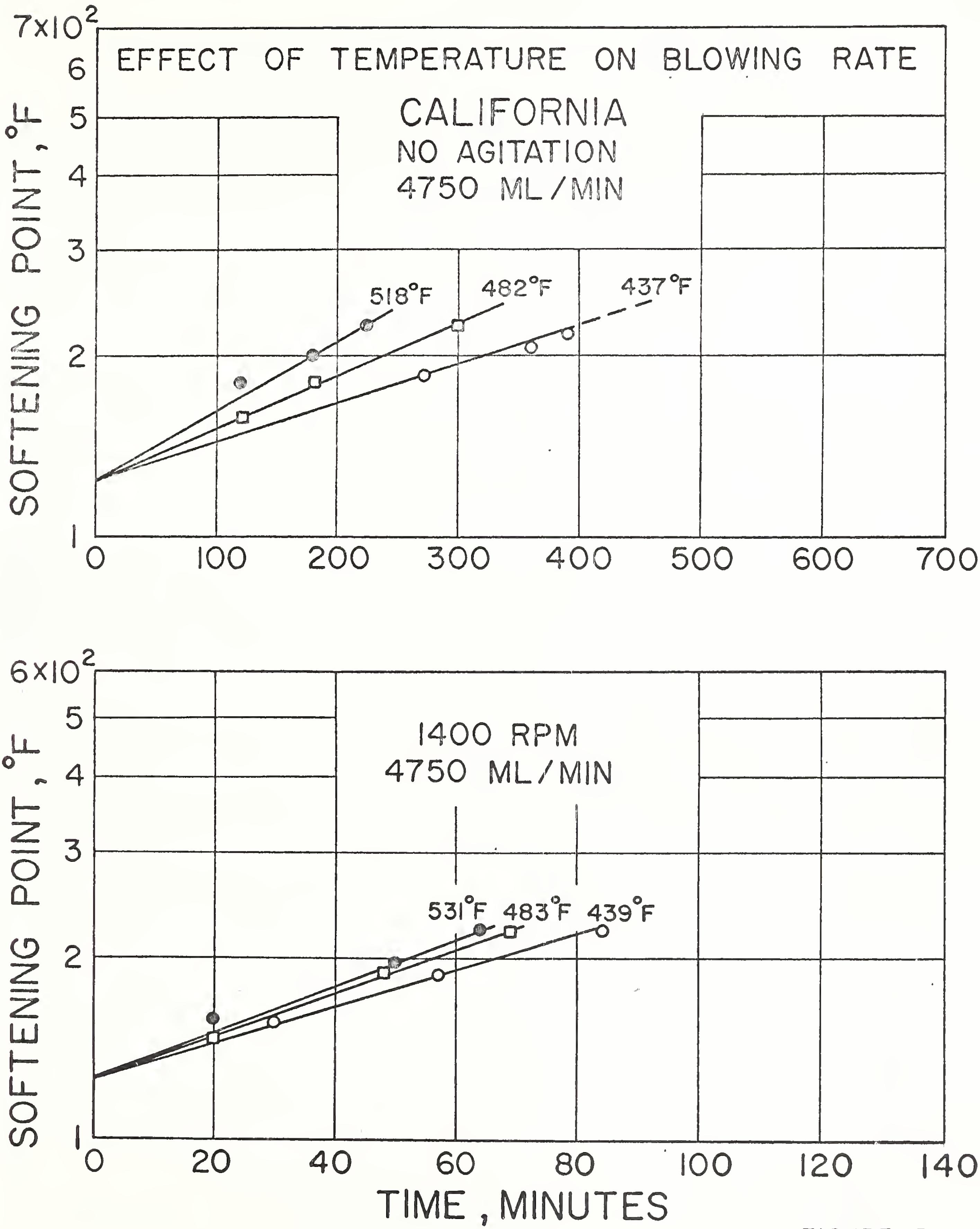

EFFECT OF TEMPERATURE ON BLOWING RATE
CALIFORNIA
NO AGITATION
4750 ML/MIN
518°F
482°F
437°F
SOFTENING POINT, °F
7x10²
6
5
4
3
2
1
0
100
200
300
400
500
600
700
1400 RPM
4750 ML/MIN
531°F
483°F
439°F
SOFTENING POINT, °F
6x10²
5
4
3
2
1
0
20
40
60
80
100
120
140
TIME, MINUTES

FIGURE 3

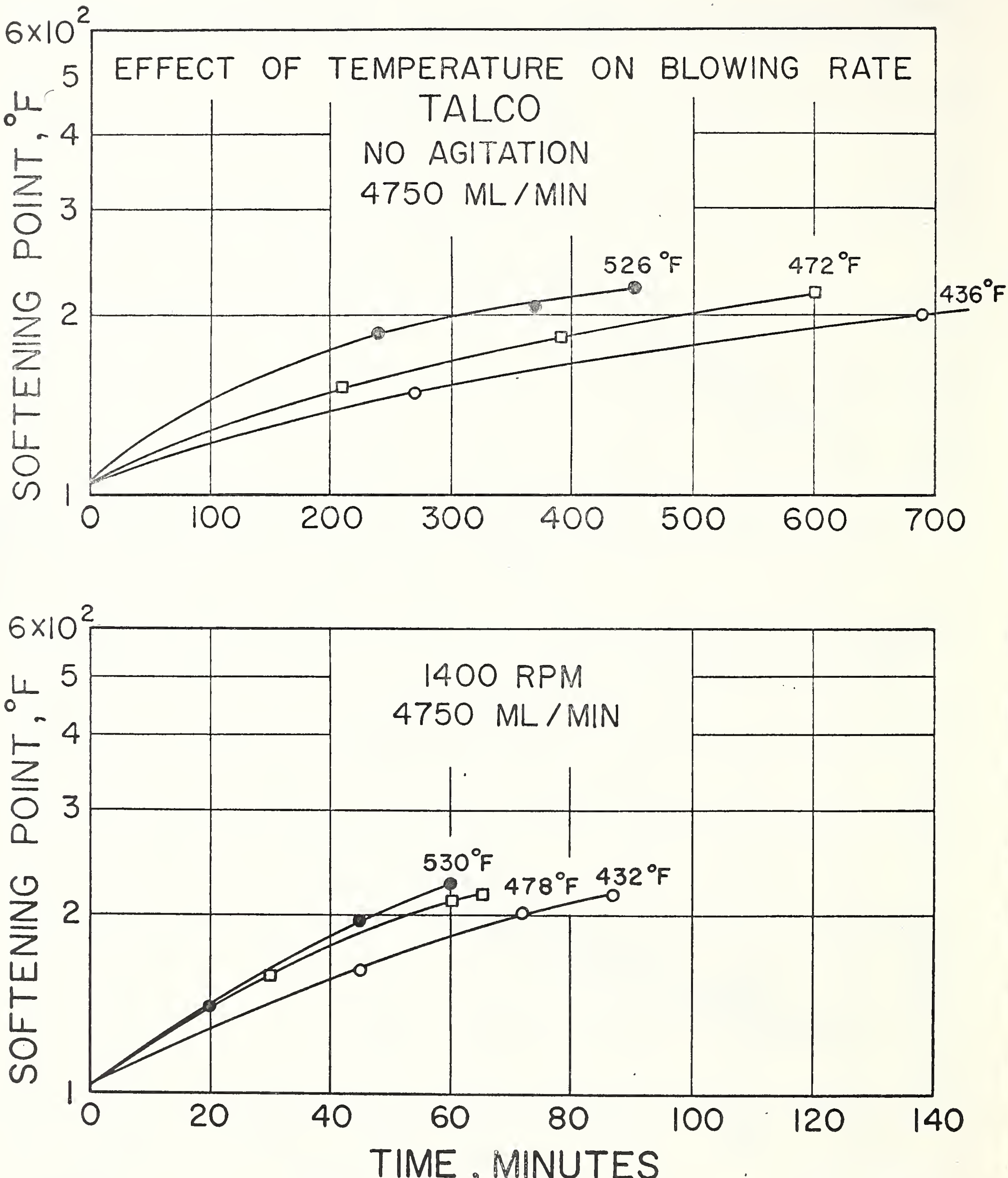
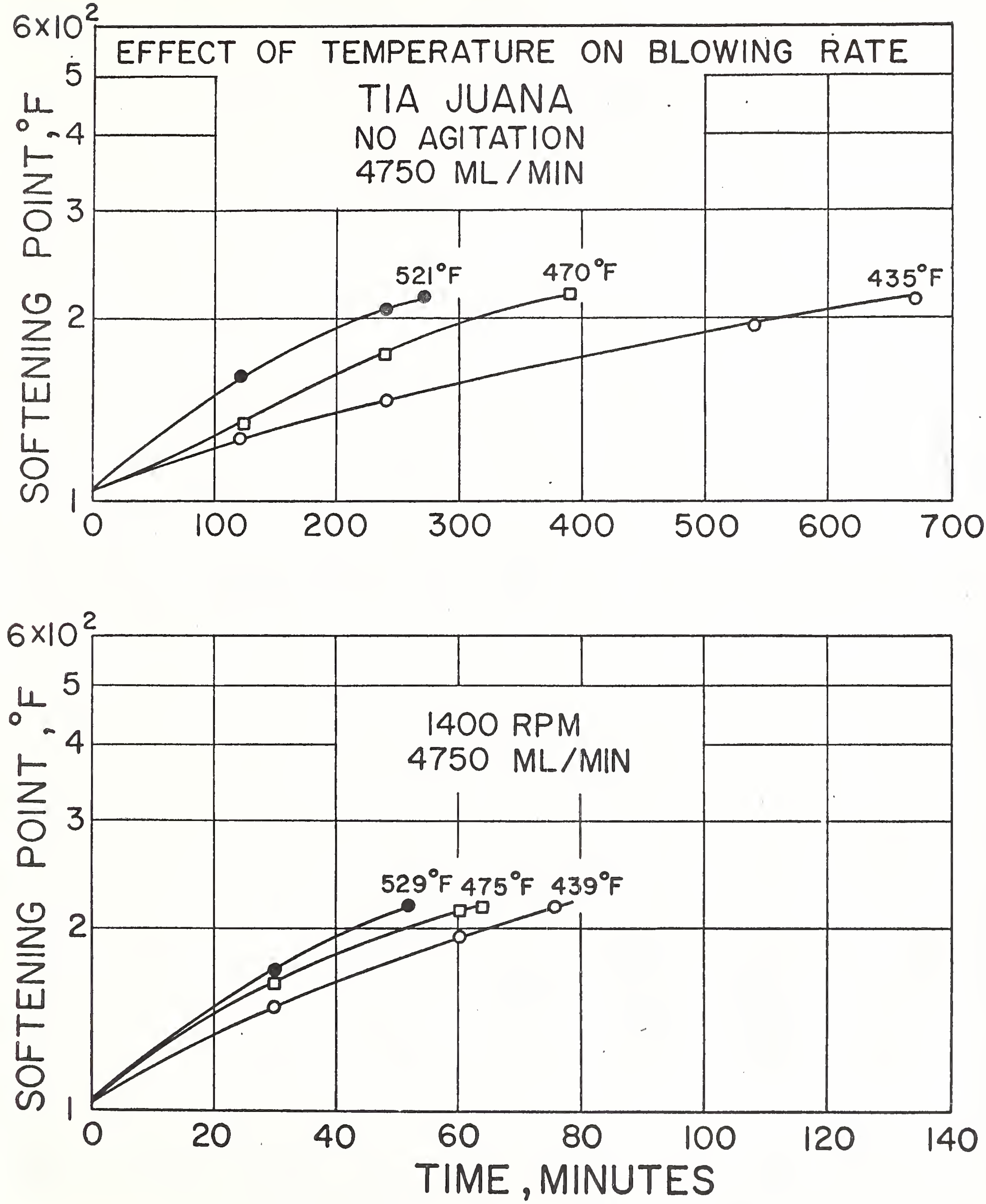
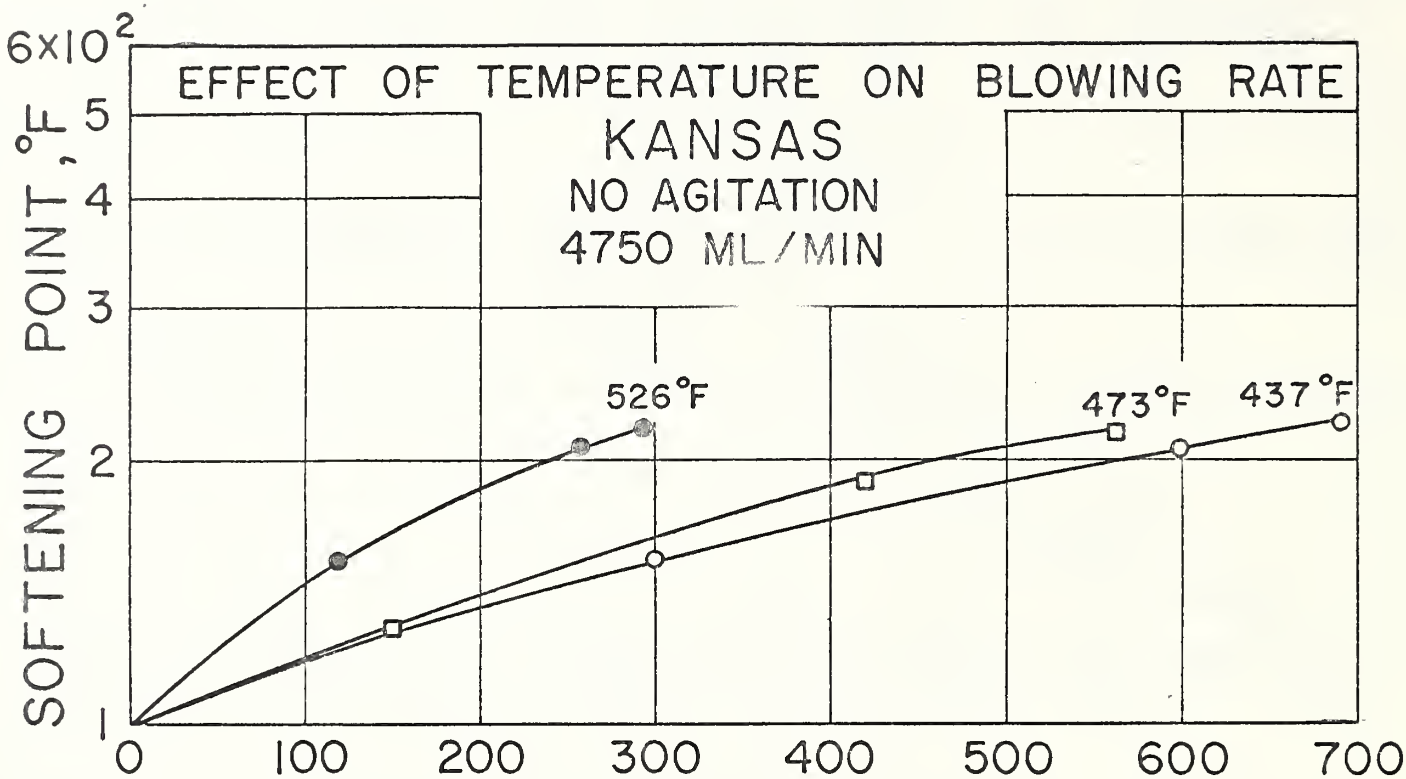

EFFECT OF TEMPERATURE ON BLOWING RATE
TALCO
NO AGITATION
4750 ML / MIN
526 °F
472 °F
436 °F
SOFTENING POINT, °F
6x10^2
5
4
3
2
1
0
100
200
300
400
500
600
700
1400 RPM
4750 ML / MIN
530 °F
478 °F
432 °F
SOFTENING POINT, °F
6x10^2
5
4
3
2
1
0
20
40
60
80
100
120
140
TIME, MINUTES

FIGURE 4



6x10^2
5
4
3
2
1
EFFECT OF TEMPERATURE ON BLOWING RATE
TIA JUANA
NO AGITATION
4750 ML/MIN
521°F
470°F
435°F
SOFTENING POINT, °F
0
100
200
300
400
500
600
700
1400 RPM
4750 ML/MIN
529°F
475°F
439°F
20
40
60
80
100
120
140
TIME, MINUTES

FIGURE 5


EFFECT OF TEMPERATURE ON BLOWING RATE
KANSAS
NO AGITATION
4750 ML/MIN
526°F
473°F
437°F
SOFTENING POINT, °F
6x10²
5
4
3
2
1
0
100
200
300
400
500
600
700

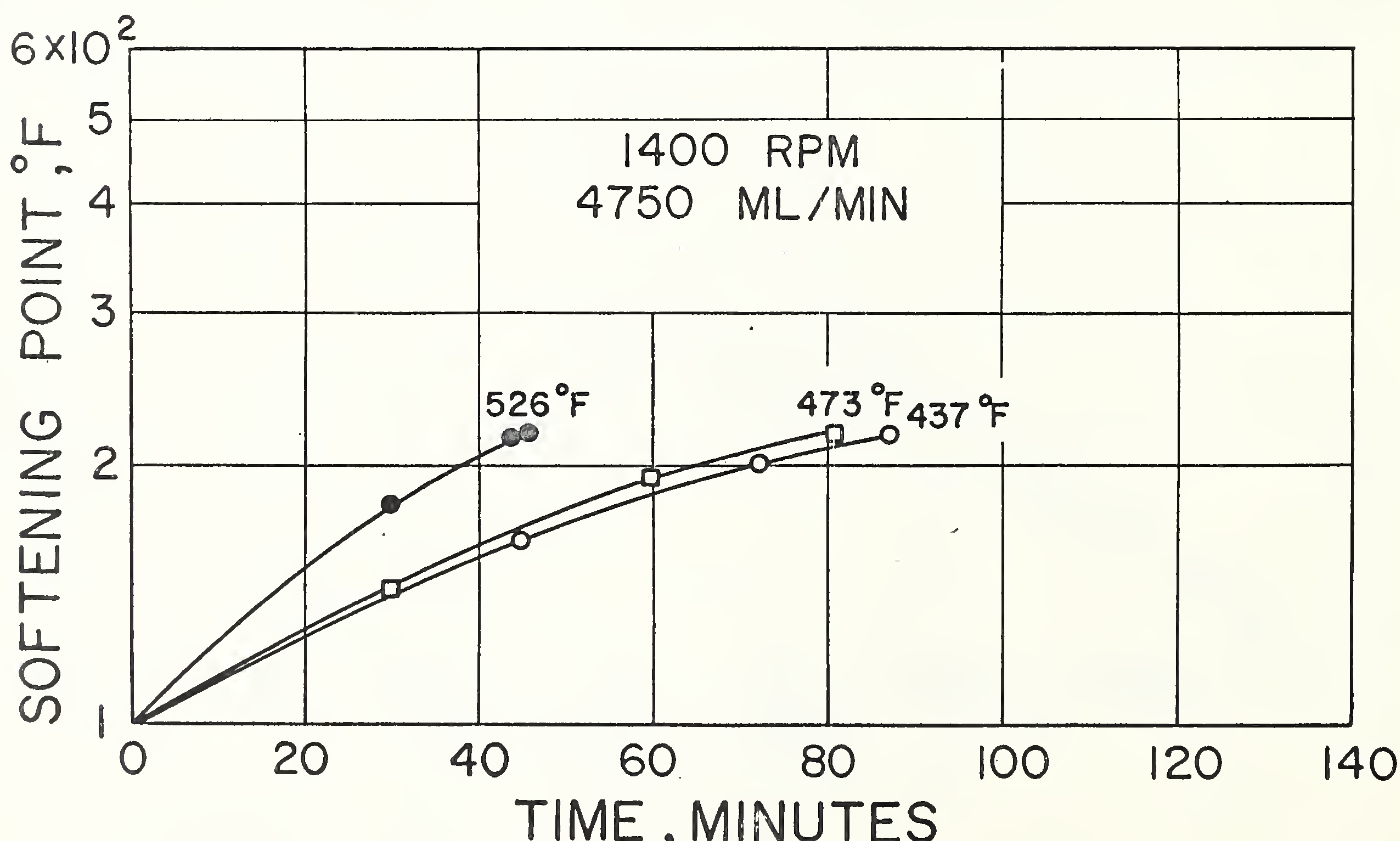
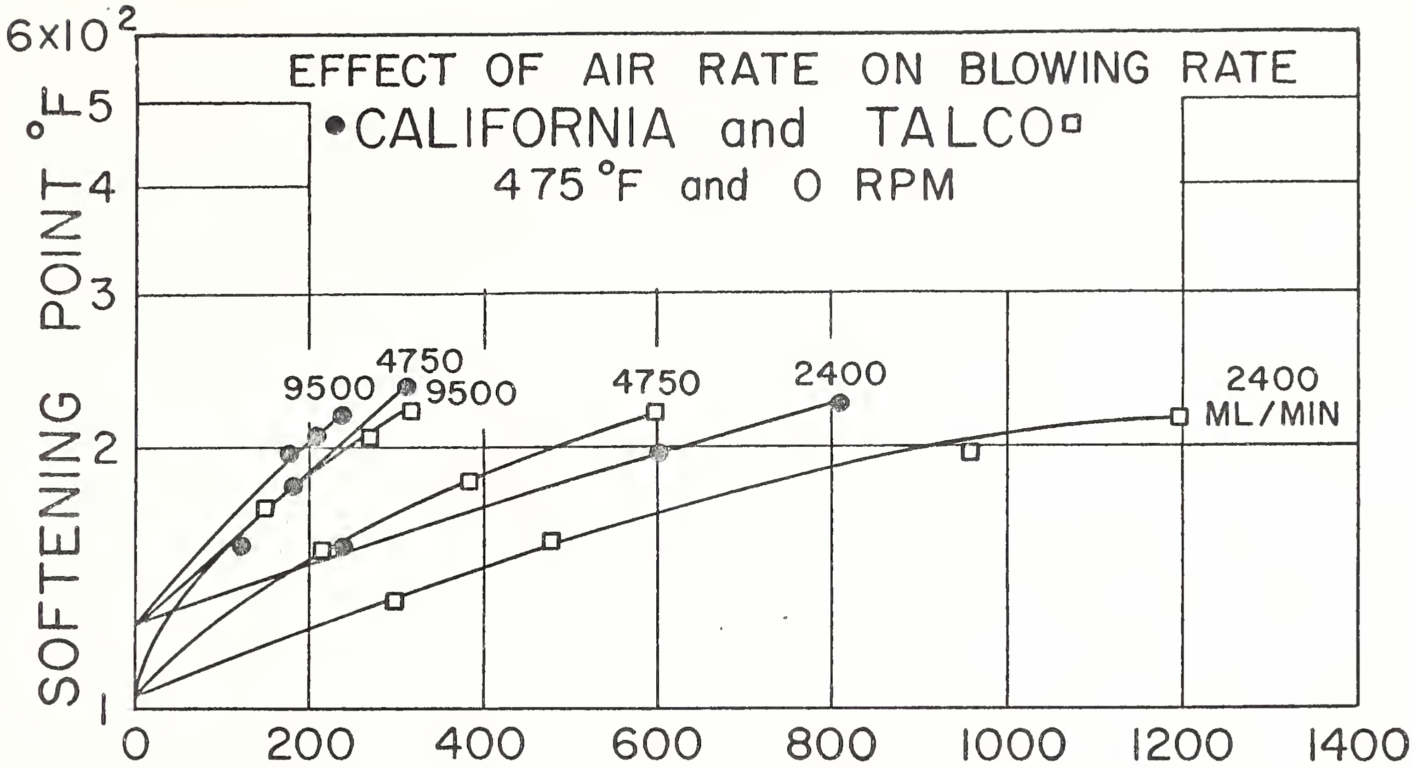

1400 RPM
4750 ML/MIN
526°F
473°F
437°F
SOFTENING POINT, °F
6x10²
5
4
3
2
1
0
20
40
60
80
100
120
140
TIME, MINUTES

FIGURE 6


EFFECT OF AIR RATE ON BLOWING RATE
•CALIFORNIA and TALCO□
475 °F and 0 RPM
SOFTENING POINT °F
6x10²
5
4
3
2
1
9500
4750
9500
4750
2400
2400
ML/MIN
0
200
400
600
800
1000
1200
1400

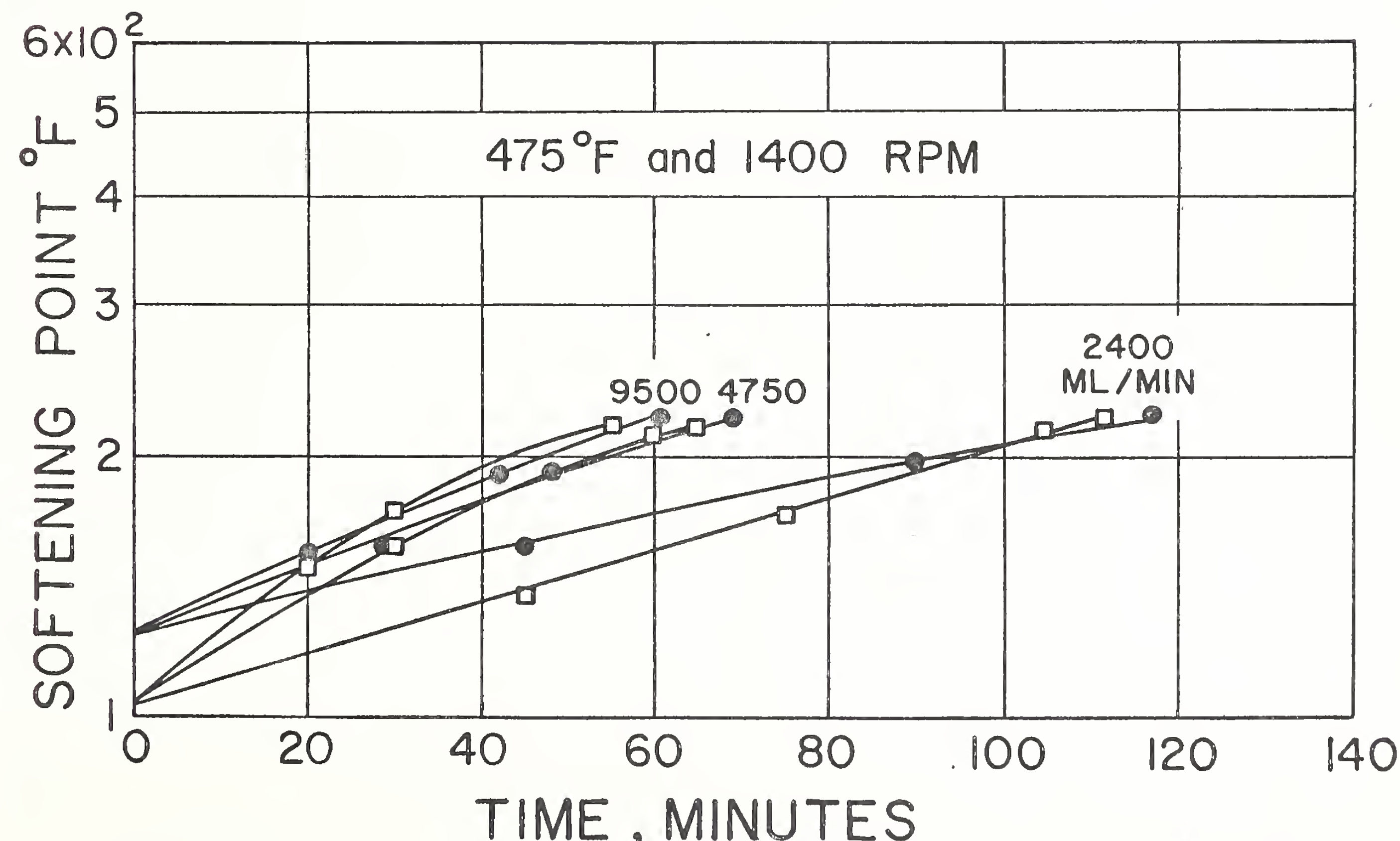
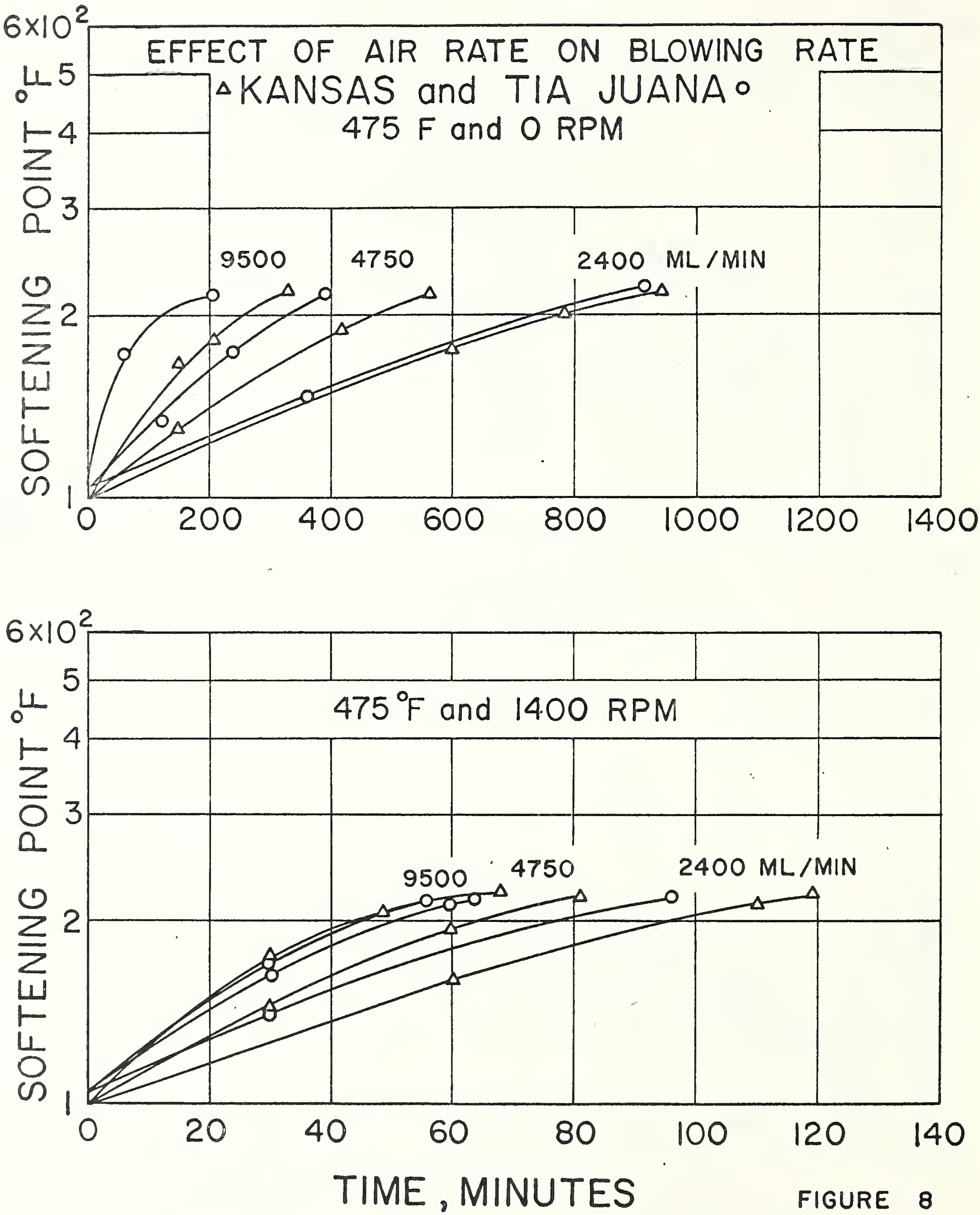
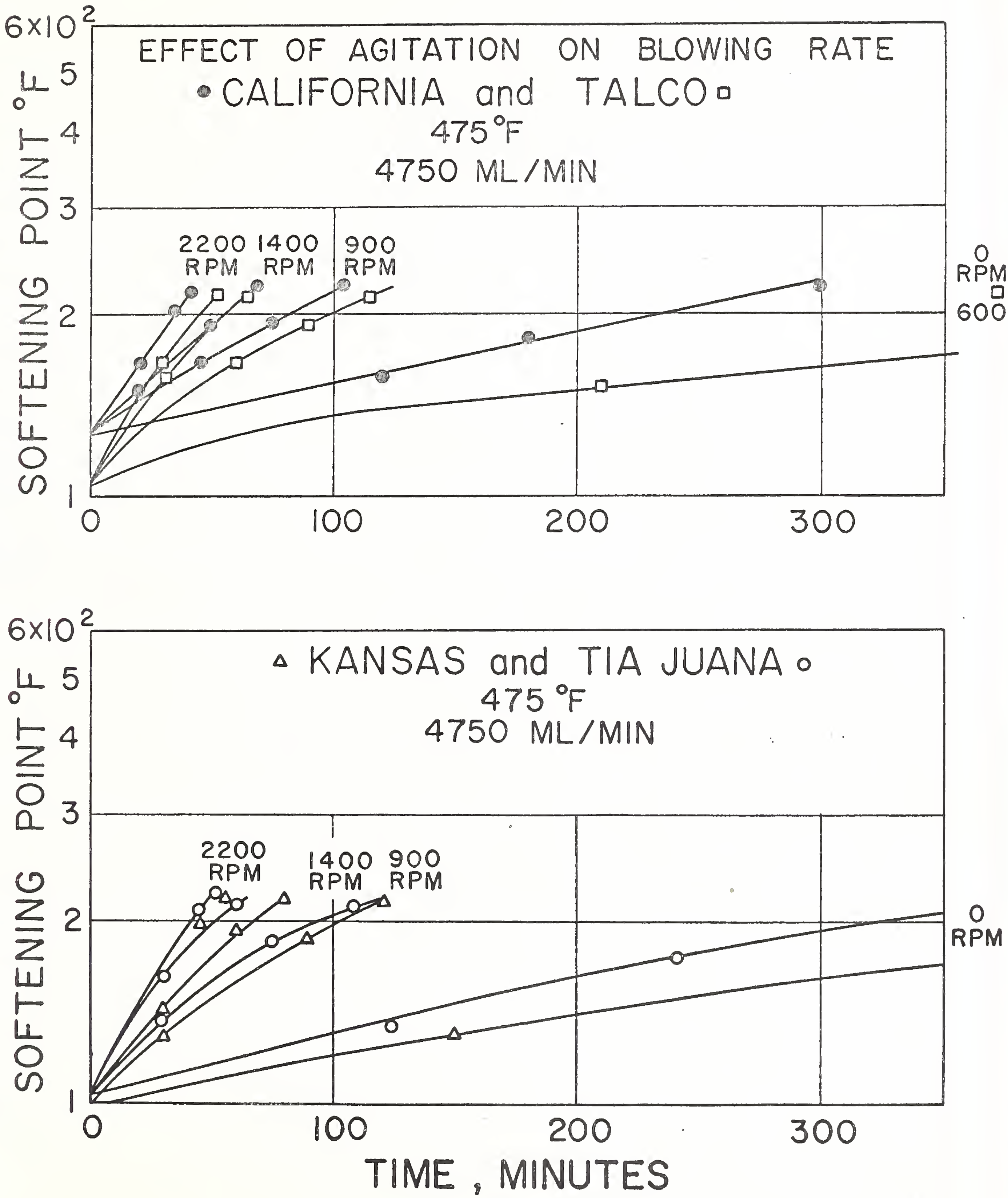

475 °F and 1400 RPM
SOFTENING POINT °F
6x10²
5
4
3
2
1
9500
4750
2400
ML/MIN
0
20
40
60
80
100
120
140
TIME, MINUTES

FIGURE 7



EFFECT OF AIR RATE ON BLOWING RATE
KANSAS and TIA JUANA
475 F and 0 RPM
9500
4750
2400 ML/MIN
SOFTENING POINT °F
6x10²
5
4
3
2
1
0
200
400
600
800
1000
1200
1400
475 °F and 1400 RPM
9500
4750
2400 ML/MIN
SOFTENING POINT °F
6x10²
5
4
3
2
1
0
20
40
60
80
100
120
140
TIME, MINUTES

FIGURE 8



EFFECT OF AGITATION ON BLOWING RATE
• CALIFORNIA and TALCO □
475°F
4750 ML/MIN
2200 RPM
1400 RPM
900 RPM
0 RPM
600
SOFTENING POINT °F
6x10²
5
4
3
2
1
0
100
200
300
△ KANSAS and TIA JUANA ○
475 °F
4750 ML/MIN
2200 RPM
1400 RPM
900 RPM
0 RPM
SOFTENING POINT °F
6x10²
5
4
3
2
1
0
100
200
300
TIME, MINUTES

FIGURE 9

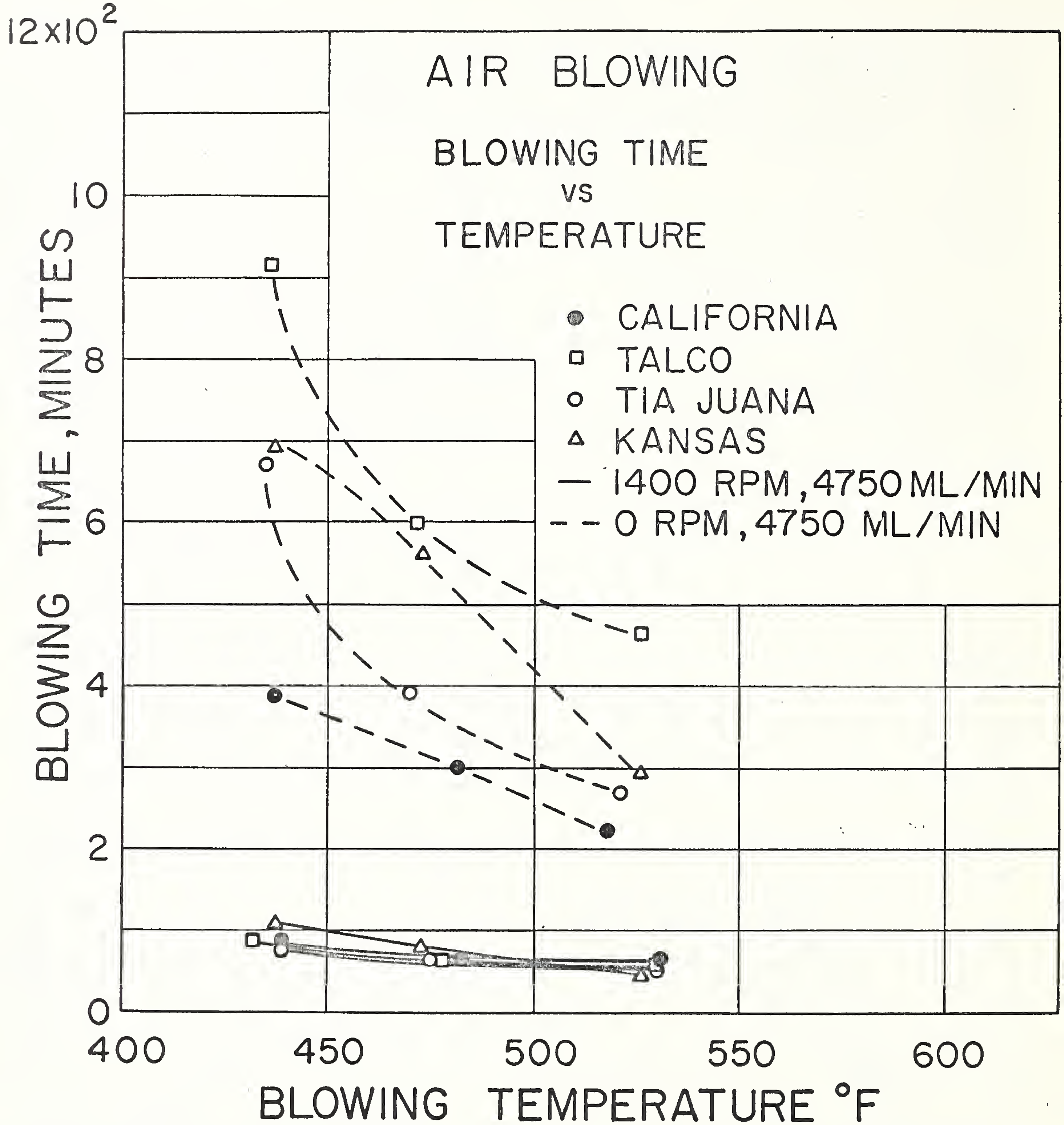

AIR BLOWING
BLOWING TIME
VS
TEMPERATURE
CALIFORNIA
TALCO
TIA JUANA
KANSAS
1400 RPM, 4750 ML/MIN
0 RPM, 4750 ML/MIN
BLOWING TIME, MINUTES
12x10²
10
8
6
4
2
0
400
450
500
550
600
BLOWING TEMPERATURE °F

FIGURE 10

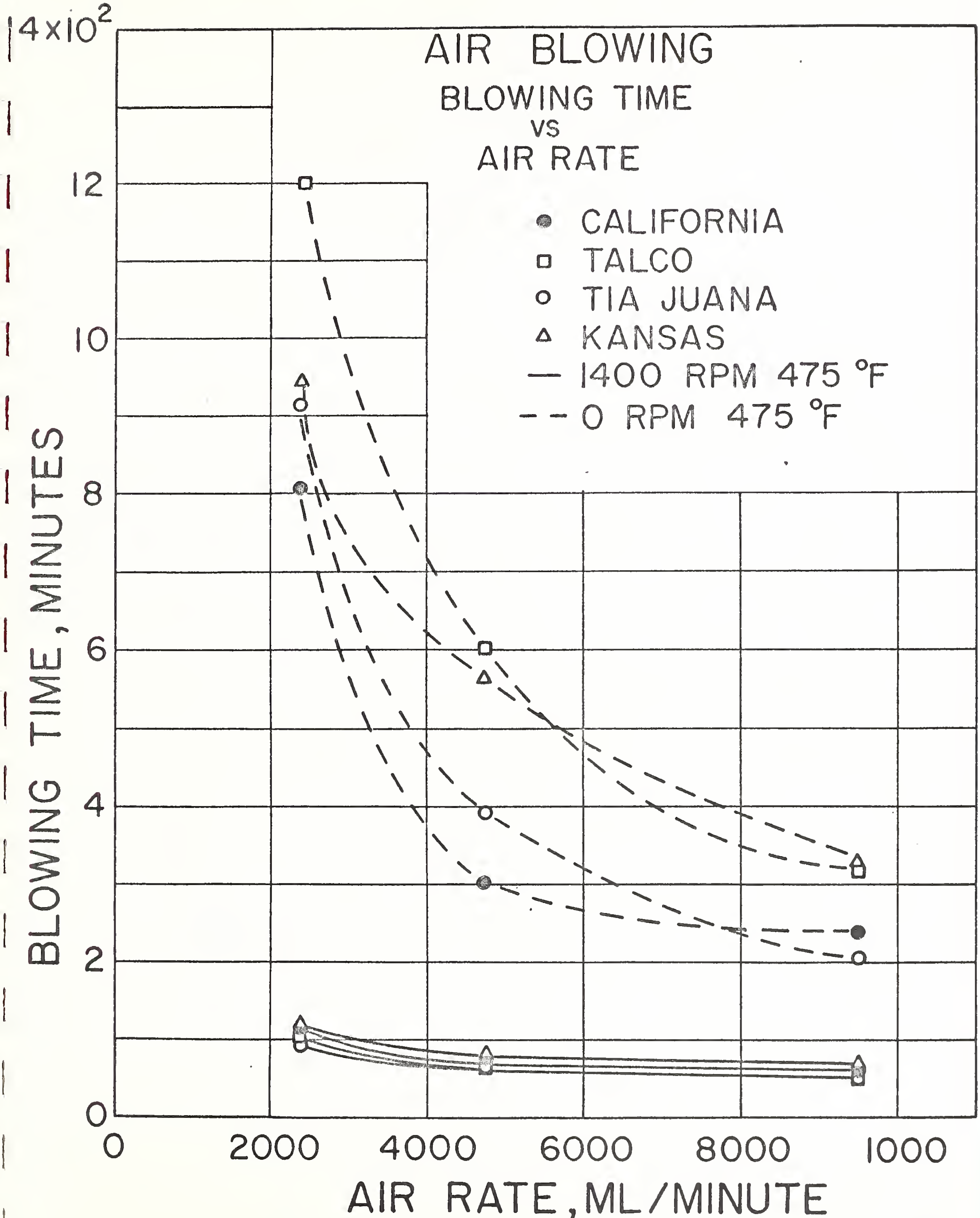

AIR BLOWING
BLOWING TIME
VS
AIR RATE
CALIFORNIA
TALCO
TIA JUANA
KANSAS
1400 RPM 475 °F
0 RPM 475 °F
BLOWING TIME, MINUTES
4x10²
12
10
8
6
4
2
0
0
2000
4000
6000
8000
1000
AIR RATE, ML/MINUTE

FIGURE 11

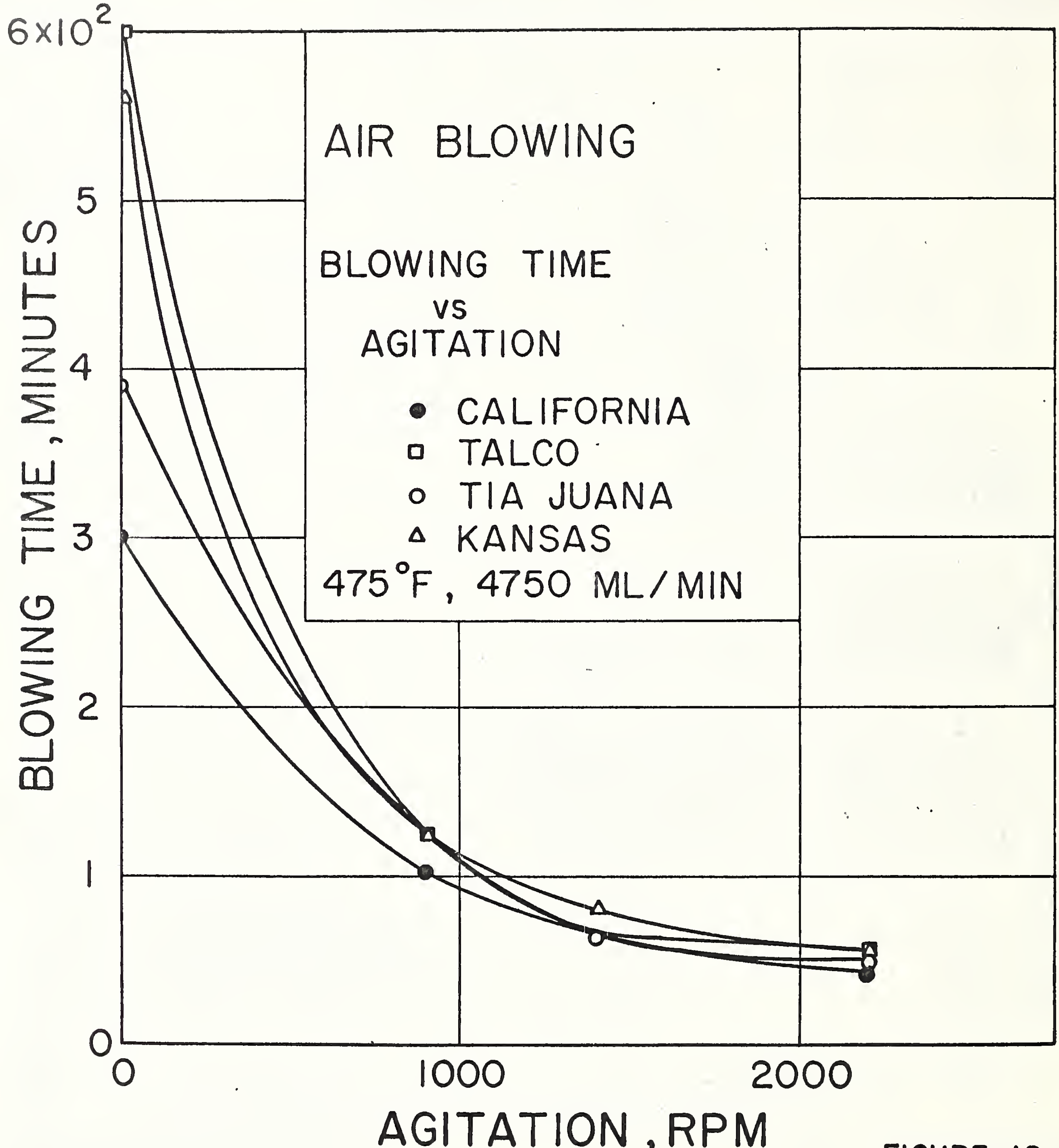

6x10²
5
4
3
2
1
0
BLOWING TIME, MINUTES
0
1000
2000
AGITATION, RPM
AIR BLOWING
BLOWING TIME
VS
AGITATION
CALIFORNIA
TALCO
TIA JUANA
KANSAS
475°F, 4750 ML/MIN

FIGURE 12

The blowing times were considerably different in the absence or presence of agitation. Thus, two sets of standard conditions were adopted: (1) No agitation, 4750 ml/min. (76 ft^3/ton min.) and 475°F. (approx. 245°C.) and (2) 1400 r.p.m., 4750 ml/min. and 475°F. Where conditions were varied, two of these three variables were held constant while the third was changed. Except where agitation was varied, two sets of data were obtained, one for no agitation and one for 1400 r.p.m. These will, of necessity, be discussed separately.

5.1 Effect of Temperature

The effect of temperature on the blowing characteristics and properties of the coating grade asphalts (products) are shown in Table 1 and Figures 3, 4, 5, 6, and 10.

California Coastal.

None of the California asphalts blew into grade (Table 1); i.e., the penetrations were below specification in the softening point range of 217 to 225°F. This condition was expected for asphalts blown from this particular California Coastal flux, because it has a preponderence of low molecular weight oils that are removed by the distillation process used to produce a flux with a minimum flash point of 450°F. The corresponding penetrations were always lower for the runs without agitation than for those with agitation. The penetrations dropped with increasing blowing temperature, even though the residence time in the still dropped as the temperature was increased. Apparently the increased volatility of the oils and decreased viscosity of the asphalt at the higher temperatures was not offset by the decreased residence time in the still.

The blowing loss figures were obtained by difference and were not accurate beyond order-of-magnitude limits. The holdup in the stills varied considerably with the draining temperature and the number of runs that had been made since the stills had been cleaned. Hence, only order-of-magnitude losses will be discussed. The losses with agitation varied from about one-half to one percent. Losses of up to ten percent occurred in runs without agitation.

The blowing curves, shown in Figure 3 on semi-log paper, were straight lines, with no obvious induction periods. In Figure 10, the time required to blow to the 220°F softening-point range can be seen to drop rapidly and linearly with increasing temperature in the unagitated runs. When agitation was used, the blowing time also decreased with temperature, but relatively slowly. (The California and Talco curves almost coincide.) The runs without agitation were about four times as long as the corresponding runs with agitation.

The asphaltene contents of the California asphalts were almost independent of blowing conditions, going up slightly with temperature of blowing. Their range of 42.7 to 44.3% compares well with commercial samples (which, however, are always modified). These latter asphalts usually had about 43-44% asphaltenes.

Talco.

The Talco flux, when blown to the 217-225°F softening point range, produced asphalts that were quite acceptable when agitation was used and borderline in the absence of agitation. The 10 minimum penetration at 32°F is frequently violated in commercial samples by a unit or two, and the two low-temperature runs produced asphalts with low-temperature penetrations of 9. In the absence of agitation, all three runs produced asphalts with penetrations at 77°F that were borderline; but with agitation, the two lower-temperature runs produced asphalts with penetrations well up in the acceptable range. All of the penetrations at 115°F were acceptable.

Just as the penetrations did not decrease appreciably with increasing temperature, so the blowing losses were low and independent of temperature. They were less than 0.5% for all the runs with agitation and between one and two percent in the absence of agitation.

Figure 4 shows that there was no induction period for the blowing of the Talco flux in any of the runs. As blowing progressed, the rate of increase of softening point decreased. This drop in rate was possibly the result of increased viscosity as the asphalt hardened.

In Figure 10 it can be seen that the blowing time dropped extremely rapidly as the temperature of blowing was increased in the absence of agitation. With agitation the decrease was much less. The agitated runs produced coating-grade asphalts in one-eighth to one-tenth of the time required in the corresponding runs without agitation.

The asphaltene contents of the Talco asphalts were all in the 39 to 42% range. Those blown without agitation tended to be toward the top of the range and those blown with agitation nearer the bottom of the range. Commercial Talco coating-grade asphalts had slightly higher asphaltene contents, in the 43 to 45% range.

Tia Juana.

The Tia Juana flux produced coating-grade asphalts within the coating-grade specifications of penetration and softening point under all the conditions investigated. As a matter of fact, the low-temperature penetrations were all within the 12-14 range, the 77°F penetrations were within the 19-22 range, and the high-temperature penetrations ranged from 33-39. There was no systematic variation of these penetrations within their small ranges with any of the variables studied.

The blowing curves for Tia Juana asphalt in Figure 5 show no induction period, but a definite decrease in rate as blowing progressed. From Figure 10 it can be seen that the blowing times dropped off rapidly with temperature, much more so without agitation than with agitation. Agitation reduced blowing time between 80 and 85% over the entire temperature range, the greater reductions being at the lower temperatures.

The asphaltene contents of the laboratory-produced, coating-grade Tia Juana asphalts were somewhat lower than those of commercial Tia Juana asphalts. These latter asphalts had between 38 and 40% asphaltenes. The asphaltene contents of the laboratory-produced asphalts were in the range of 34.3 to 35.2%, which is within the normal variation of the determination.

Kansas.

All of the Kansas coating-grade asphalts met the softening point and penetration specifications. The penetrations were essentially constant at 12 dmm at 32°F, varied from 19-22 dmm at 77°F, and 32-38 dmm at 115°F with agitation, increasing with increasing blowing temperature. In the absence of agitation, the penetrations were essentially constant at each of the three temperatures.

As seen in Figure 6, no induction period existed in any of the blowing runs. The blowing rate decreased as blowing progressed in all instances.

The data in Figure 10 show that blowing time decreased for this Kansas asphalt as the blowing temperature was increased. The acceleration was much greater in the absence of agitation than when agitation was used. It required about six times as long to blow to grade without agitation as it did with agitation.

Blowing losses were very low, of the order of less than 0.5% when agitation was used. In the absence of agitation, the losses were of the order of 1%, with the greater loss occurring at the lowest temperature.

The asphaltene contents of the blown asphalts were in the 32.6-34.5% range, with no temperature dependence indicated. Commercial samples of Kansas asphalt have averaged several percent more asphaltenes, in the 37-38% range.

Comparison and Contrast

The California coating-grade asphalts failed to meet specification grade irrespective of blowing temperature; the penetrations decreased as the blowing temperature was increased and were considerably lower in runs made without agitation. The Talco asphalts met penetration requirements when agitation was used, but were borderline without agitation.

All of the Tia Juana and Kansas asphalts were well within penetration requirements.

Except for the California asphalt, there was no relation between penetration and blowing temperature.

All four asphalts exhibited an appreciable decrease in blowing time as the blowing temperature was increased. The effect was much more pronounced when agitation was not used. In the absence of agitation the Talco asphalt blew most slowly, followed by Kansas, Tia Juana and California in order at all temperatures. When agitation was used, the relative rates changed as the blowing temperature was increased. Kansas was slowest at 430°F and 475°F, and fastest at 530°F. The other three asphalts blew to grade in essentially the same times at 475 and 530°F and within eleven minutes of each other at 430°F. The blowing times were not strictly comparable because all runs were not consumated at exactly the same softening point and the California flux had a softening point some 20°F higher than those of the other three.

The blowing losses were always low, of the order of one percent or less when agitation was used. In the absence of agitation, the losses were about one percent for Kansas and Tia Juana, two percent for Talco, and ten percent for California asphalt.

The asphaltene contents were independent of blowing temperature. They were comparable to commercial asphalts for the California source, but lower by three to four percent for the other three sources.

5.2 Effect of Air Rate

The effects of air rate on blowing time and characteristics and on the properties of the final products were nearly the same for all four asphalts. Therefore, the four asphalts will be discussed as a group, instead of individually as under "Effect of Temperature".

All asphalts in this series were blown at about 475°F. One set with each flux was blown without agitation and one set with the turbos run at 1400 r.p.m. Three air rates were used: 2400, 4750, and 9500 ml/min., corresponding to 38.5, 76, and 152 ft^3/ton min.

Except for the California asphalt, all products easily met the penetration requirements (Table 2). The corresponding penetrations were always lower for the unagitated runs for the California and Talco asphalts and about the same for the Tia Juana and Kansas materials. No dependence of penetrations on air rate was observed.

No induction period was found at any air rate (Figures 7 and 8) and, except for the California asphalt in the unagitated runs, the blowing rates decreased as blowing progressed.

Blowing time was extremely dependent on air rate (Figure 11). As the rate was quadrupled from 2400 to 9500 ml/min., the blowing times were halved when agitation was used and reduced by a factor of 65 to 75% in the absence of agitation. In all instances the major decrease in time was accomplished during the initial increases in air rate. When agitation was used, the acceleration accomplished by going from 4750 to 9500 ml/min. was extremely small.

No consistent pattern of weight loss was observed in this phase of the work. For the California and Talco asphalts the losses with agitation were under one percent, but without agitation they ran from five to ten percent for the California asphalt and, for Talco up to five percent. In contrast, the losses in the Tia Juana runs were in the neighborhood of one percent, decreasing as the air rate was increased. The Kansas losses were greater with agitation than without it. They increased from one-half percent at the lowest air rate to three percent at the highest with agitation and remained under one percent without agitation.

The asphaltene contents of the final products were independent of the air rates (Table 2).

5.3 Effect of Agitation

The effects of agitation during blowing on the blowing characteristics and properties of the coating-grade asphalts were investigated at a blowing temperature of 475°F and air rate of 4750 ml/min. (76 ft^3/ton-min.). Agitation rates up to 2200 r.p.m. were used.

With both the California and Talco asphalts (Table 3), the penetrations at each temperature increased progressively with increasing agitation (decreasing residence time as well). However, none of the California asphalts met coating-grade penetration specifications. The other three asphalts met specifications regardless of agitation. The penetrations of the Kansas and Tia Juana asphalts were independent of agitation.

The blowing curves for the four asphalts in Figure 9 show no induction period and a tapering off of blowing rate as blowing progressed for all but the California asphalt.

As seen in Figure 12, the blowing times dropped rapidly as agitation was increased, but the greatest drop occurred as agitation was introduced. Very little improvement was involved when the speed of the turbo blades was increased from 1400 to 2200 r.p.m. The largest decrease in time was effected for the Talco and the least for the California asphalt. These different improvements in blowing time in the four asphalts are undoubtedly related to the viscosity characteristics of the charges during the blowing process.

No consistent weight loss relation with agitation was found. The California asphalt lost ten percent without agitation and about one percent in the other three runs. The Talco asphalt lost two percent without agitation, four percent at 900 r.p.m. and considerably less than one-half percent in the other two runs. Both the Kansas and Tia Juana asphalts lost about one percent without agitation. With agitation, the losses progressively increased from less than one-half percent at 900 r.p.m. to over two percent at 2200 r.p.m.

The asphaltene contents were independent of agitation.

5.4 Comparisons with Recent Literature

Two articles have appeared in the technical literature in recent years relating directly to the effect of various blowing variables on the blowing characteristics of asphalts and the properties of coating-grade asphalts. Rescorla, et al, [2] in 1956, reported on the effects of agitation up to 1100 r.p.m., air rates up to 50 ft^3/ton-min. and temperature up to 550°F on the time required to blow four different asphalts to a softening point of 220°F. Their still was one foot in diameter and 21 inches high,with four verticle baffles. It was charged to a depth of one foot (H/D = 1) with a 50-pound charge of asphalt. A single 4-inch diameter, flat-bladed impeller was used to agitate the asphalt. The air was introduced through a 4-inch ring just below the agitator containing a series of 1/16-inch holes along its top surface. The essential results of this work indicated that blowing time dropped appreciably as agitation was increased at constant temperature, as temperature was increased at constant agitation, and as air rate was increased at constant temperature. They compared their laboratory results with plant production and found that with no agitation in the laboratory still the penetrations (at 77°F) were considerably lower than plant production. At 880 r.p.m., the agreement was somewhat improved, but the penetrations of the laboratory produced asphalts were still lower than plant production for two Venezuelan asphalts. For another Venezuelan asphalt and a Mexican asphalt, agreement was good.

Because the geometry of the still was different from the one used in this study, the results are not strictly comparable, but the trends are in good agreement.

The second paper was published by Chelton, Traxler, and Romberg in 1959 [3]. It was a study of the effects of temperature, pressure, air rate and liquid level on the blowing characteristics of a Louisiana and a Texas asphalt in an unagitated still. A height-to-diameter ratio of 1.7 was used. The results obtained showed that blowing time decreased as temperature, air rate, pressure and liquid level were increased. Penetrations at 77°F decreased appreciably as temperature increased, but increased as pressure or air rate was increased. All of their blowing rates were constant during the entire runs.

Again a direct comparison is not possible with the present work because of geometry differences in the stills. However, the trends of the variation of blowing time with air rate and temperature are the same. The changes of penetration at 77°F with temperature and air rate are quite different, however. No changes were observed in this study except for a slight drop in penetration with temperature for the California Coastal asphalt.

6. SUMMARY AND CONCLUSIONS

The effects of temperature, air rate and agitation on the blowing characteristics and properties of blown asphalts were investigated on California, Talco, Tia Juana, and Kansas fluxes. The following conclusions may be drawn:

(1) Tia Juana and Kansas fluxes blew into grade under all conditions.

(2) Talco flux blew into grade under standard blowing conditions, but produced marginal products when blown at high or low temperatures.

(3) California flux did not blow into grade under any of the conditions studied.

(4) The final penetrations of Kansas and Tia Juana asphalts were relatively independent of blowing conditions.

(5) The penetrations of the final California products were lower in the absence of agitation and decreased as the blowing temperature was increased.

(6) The 77°F and 115°F penetrations of Talco products decreased as blowing temperature was increased.

(7) Blowing times for all asphalts decreased as temperature, air rate, and agitation were increased.

(8) No induction periods were observed in any of the runs.

(9) Except for the California flux, the blowing rate of which remained constant, the blowing rates on all of the other fluxes decreased as blowing progressed.

(10) Except for the California flux blown without agitation, the blowing losses were relatively low, as follows:

Asphalt	Agitation 0 r.p.m.	900-2200 r.p.m.
	%	%
California	1.5-10.0	0.5-1.5
Talco	0.1- 5.0	0 -2.0
Tia Juana	0.5- 2.0	0 -2.0
Kansas	0 - 2.5	0 -3.0

(11) The asphaltene contents of the blown asphalts were independent of blowing conditions.

(12) The asphaltene contents of the California products were comparable to those of commercial materials, but products made from the other three asphalts were two to four percent lower in asphaltenes than comparable commercial asphalts.

7. REFERENCES

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[2] A. R. Rescorla, W. E. Forney, A. R. Blakey, and M. J. Frino, "Asphalt Oxidation with Agitation", Ind. Eng. Chem. 48, 378-80, 1956.

[3] H. M. Chelton, R. N. Traxler, and J. W. Romberg, "Oxidized Asphalts", Ind. Eng. Chem. 51, 1353-1354, 1959.

8. ACKNOWLEDGMENT

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