NATIONAL BUREAU OF STANDARDS REPORT

8046

THE EFFECT OF BLOWING VARIABLES ON THE

ACCELERATED WEATHERING DURABILITY OF COATING-GRADE ASPHALTS

by

Sidney H. Greenfeld

Not for publication or
for reference.

NBS

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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Sidney H. Greenfeld

Research Associate
Asphalt Roofing Industry Bureau

Organic Building Materials Section
Building Research Division

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ABSTRACT

The effects of varying temperature, agitation and air-rate during the blowing of fluxes from California, Kansas, Tia Juana and Talco fields on the properties and accelerated weathering durability of the coating-grade products were studied. Although the durabilities of asphalts from each source varied with the blowing conditions, the California and Talco fluxes were more dependent on the variables studied than were the Tia Juana and Kansas fluxes. The durabilities of the asphalts produced from the California flux varied from 39 - 68 days inversely with blowing temperature and directly with air rate and agitation. The durabilities of the Talco asphalts varied in the range of 32 to 56 days inversely with the blowing temperature, but were relatively independent of air rate and agitation. The durabilities of the Tia Juana asphalts were highest when they were blown at the lower temperatures and higher air rates with little or no agitation. They varied within the range of 46 to 60 days. The durabilities of the Kansas asphalts ranged from 65 to 87 days. The highest blowing temperatures and air rates produced the least durable Kansas asphalts. The ranges of variables covered were as follows:

Temperature:	430 - 530°F
Air Rate:	38 - 150 cu ft/ton-min.
Agitation:	0 - 2200 rpm

All three variables had pronounced effects on the blowing rates of the four fluxes. However, the final penetrations of the Kansas and Tia Juana asphalts were insensitive to the blowing conditions. The penetrations of the California and Talco asphalts varied to a greater extent with blowing conditions. The California asphalts had penetrations lower than normally specified for coating-grade materials.

The solubility parameters of the asphaltenes and the asphaltene contents of the asphalts were related to the flux source, but did not vary appreciably with the blowing variables.

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1. INTRODUCTION

Controlled oxidation (blowing) has been used to modify the properties of organic materials for almost a century. The historical development and general nature of the changes that occur during the blowing of asphalt have been described in standard references such as Abraham (1)[1/] and Traxler (2). These changes consist of hardening of the asphalt, a decrease in its temperature susceptibility and a shift toward a more gel-like material. While the changes involved are generally similar among many asphalts the exact nature of the changes that occur in each asphalt is related to its composition and, therefore, to its source. The blowing process offers a relatively convenient way for modifying asphalts.

Chelton, Traxler and Romberg (3) have shown that the mechanical properties of two asphalts were functions of blowing temperature, pressure, air rate and liquid level. The magnitudes of the effects were different for the two asphalts studied. The differences between them were attributed to differences in their viscosities at the blowing temperatures. Gun and Gurevich (4) have reported that the mechanical properties of blown road asphalts vary with blowing temperatures.

Rescorla, Forney, Blakey and Frino (5) studied the use of mechanical agitation to increase the efficiency of oxygen utilization in the blowing of asphalts. Mechanical agitation (one impeller) decreased the oxidation time to produce an asphalt with a given softening point. The asphalt produced with agitation was softer than its non-agitated counterpart. Agitation faster than 880 rpm appeared not to shorten the time further. The authors recommended additional impellers to improve oxygen efficiency further.

Eng, Govier and Quon (6), and Muraya et al (7), on the other hand, reported that blowing temperature and air rate had little effect on the softening point-penetration relationships of Redwater and Arabian asphalts, respectively, even though the blowing times were nearly halved. The ranges of temperature and air rate covered were 465 - 525°F and 55 - 75 ft^3/ton-min, respectively.

Others have reported on the effects of blowing on the composition of coating-grade asphalts (8, 9, 10, 11, 12, 13, 14). Lockwood (15) has shown with his own data and the data of Rescorla et al (5) that except at high temperatures and low air rates blowing seems to conform to a first order reaction rate.

[1/] The numbers in parentheses refer to references at the end of this report.

In all these studies, no attention was given to the effect of any of the blowing variables on durability. In this study, however, durability[2/] will be of primary importance and the physical properties will be of secondary consideration. However, comparisons of the "specification properties" among the various asphalts will be made and the sensitivity of these properties to variations in blowing conditions will be discussed.

2. EQUIPMENT

Two 2-liter stainless steel blowing stills were used, as shown in Figure 1. The one on the left contained an agitator, the one on the right an x-member air distributor. Figure 2 is a view of the disassembled stills, showing the 4-inch OD x 21-inch still housing, the four 500-watt strip heaters fastened to the 1/4-inch heat distributing jackets, the air distribution systems and the agitator design. Air was introduced into both stills through 3/8-inch stainless steel tubes, which enter through the tops and carry the air to within one inch of the bottom of the stills. In the agitated still the air was delivered to the center of the lowest impeller, where it was broken into extremely fine bubbles as it was thrown outward by the three impeller blades. The ascending air-asphalt mixture was drawn successively into the second and third impellers, where the air was again subdivided. In the non-agitated still the air was distributed through twelve holes equally spaced in the four arms of the X. The total area of the holes was 1.2 times that of the 3/8-inch tube. This ratio is frequently used in commercial stills.

The asphalt flux was introduced through the 1/2-inch stainless steel 45° elbows on the tops of the stills and was sampled and removed through the stainless steel valves at the bases of the stills.

Compressed air was filtered through glass wool and metered to each still through a rotameter, the meter closest to each still in Figure 1. The other two rotameters were not used in this study.

The heat input was controlled manually through the two variable transformers at the bottom of Figure 1.

All of the asphalts in this study were blown at atmospheric pressure. When a positive pressure appeared on one of the gages a blockage in that air line was indicated and that still was dismantled for cleaning. Cleaning was accomplished by heating the dismantled still and air tubes to about 600°C in a muffle furnace and completely oxidizing the residues in them.

[2/] Durability as used in this report is as determined in a Weather-Ometer.

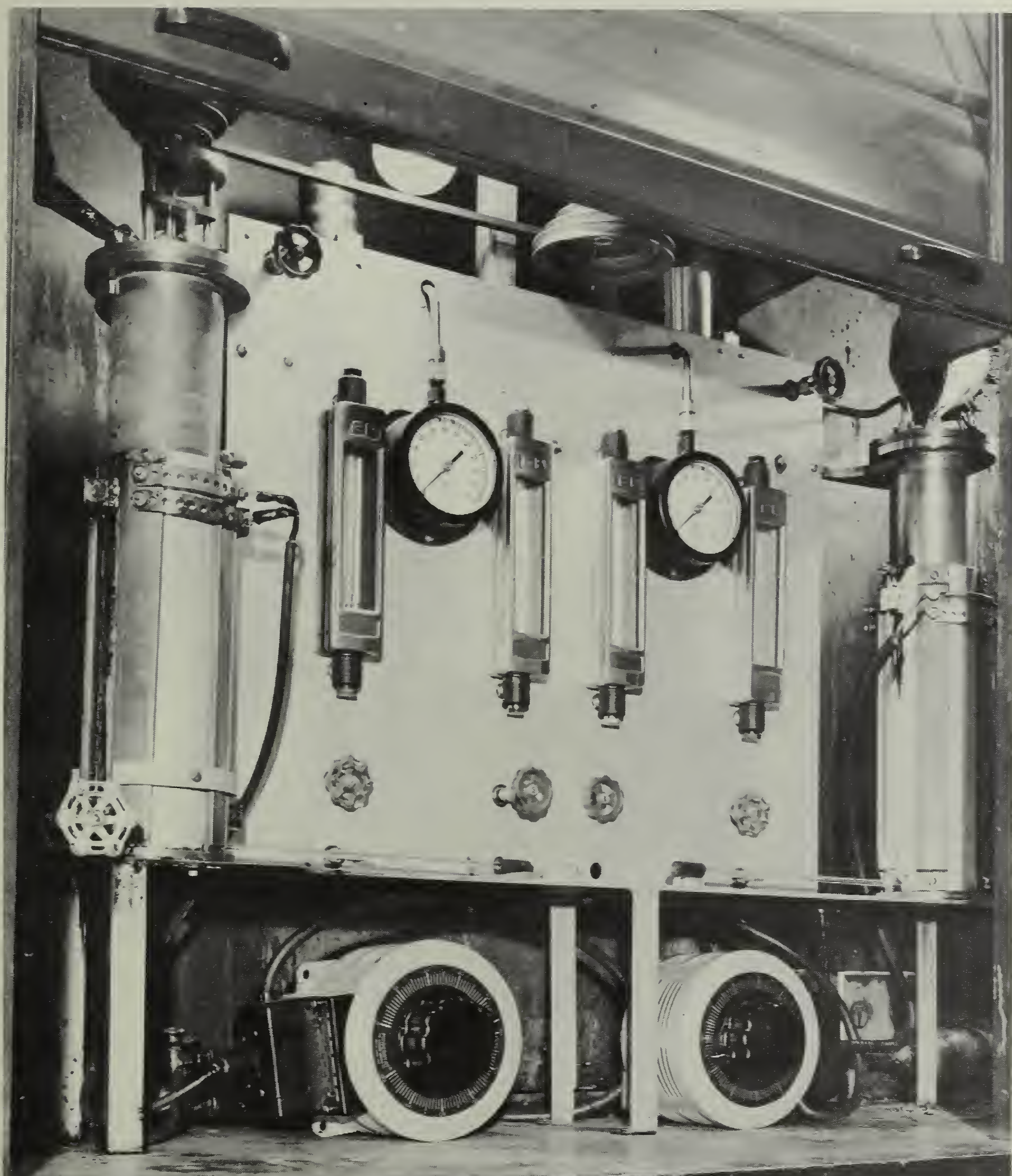


FIGURE 1. ASPHALT BLOWING STILLS.

FIGURE 2. COMPONENTS OF BLOWING STILLS.

3. MATERIALS

Based on a survey of crudes being used for the production of coatings for shingle and roll roofing products in the United States, four fluxes were selected. These asphalts represented a large fraction of the tonnage of asphalt used for roofing manufacture. The properties of these fluxes are listed in Table 1.

4. PROCEDURE

4.1 Blowing

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, the air was turned off and samples were taken for softening point determinations. The air was turned on immediately afterwards. When the expected softening point was over 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 products.

4.2 Exposure and Evaluation

A pint can of each asphalt was heated on a thermostated electric hot plate to just over 400°F (205°C) and maintained at that temperature until the asphalt surface was essentially free of air. (Those asphalts blown with agitation usually contained more finely divided air than those blown without agitation.) Four exposure specimens were made of each asphalt by the hydraulic press method (ASTM D1669-59T). However, Teflon reinforced with glass fibers was used as the parting material in place of the dextrin-coated paper. The Teflon was stripped from each specimen by a firm pull when the specimen had cooled to room temperature.

TABLE 1.

Properties of Fluxes

Source	California Coastal	Kansas	Talco	Tia Juana
Softening Point, °F	122	101	104	104
Penetration @ 32°F, 1/10 mm	20	42	44	44
Penetration @ 77°F, 1/10 mm	64	249	190	152
Penetration @ 115°F, 1/10mm	TS 1/	TS	TS	222
Specific Gravity at 77°F	1.030	0.994	1.020	1.004
Flash Point (C.O.C.), °F	460	635	555	575

1/ too soft

The specimens were exposed the following morning, two in each of two Weather-Ometers, to the 51-9C cycle (ASTM D529-59T Cycle A). All specimens were made on Mondays and exposed on Tuesdays in order to keep the weekends, periods during which the Weather-Ometers were not operated, in the same relative places in the exposures of all specimens.

Failure was determined by a high-voltage probe (ASTM D1670-59T) at the end of a dry part of a cycle. The durabilities reported are the average of the four specimens at the 50% failure level as measured with a 60-square grid.

4.3 Properties

Softening points were measured by a slight modification of ASTM Method D36-26. The penetrations, by ASTM D5-61. The softening point determination was modified in three respects: (1) A shouldered ring was used, as described in ASTM E28-58T. (2) The ring holder assembly was similar to the one described in ASTM E28-58T, but contained holes for four rings instead of two. (3) A gentle stream of air was bubbled through the glycerine during the higher softening point determinations to provide the agitation required for a uniform temperature rise.

The asphaltene-content and solubility parameter determinations were as described by Greenfeld and Wright (16).

5. RESULTS

The data obtained in this study may conveniently be summarized under the three principal independent variables investigated: (1) Temperature, (2) agitation and (3) air rate. These summaries are presented as Tables 2, 3, and 4, respectively.

Data are presented on the changes of softening points with blowing time for each of the asphalts under the various blowing conditions. Figures 3, 4, 5, and 6 show the effect of blowing temperature on the change of softening point with blowing time (blowing rate) for California, Talco, Tia Juana and Kansas asphalts, respectively.

Figures 7 and 8 contain data showing the effect of air rate on blowing rate for California and Talco and Kansas and Tia Juana asphalts, respectively.

Figure 9 is a summary of the effect of agitation on blowing rate for all four asphalts.

TABLE 2. BLOWN ASPHALT CHARACTERISTICS

EFFECT OF TEMPERATURE

Flux Identification	Durability (51-9c), Days	Blowing Conditions: Time, min.	Blowing Conditions: Agitation, r.p.m.	Blowing Conditions: Temp., °F	Blowing Conditions: Air Rate ft^3/ton-min.	Weight Loss*, %	S.P., °F	Penetrations: at 32°F	Penetrations: at 77°F	Penetrations: at 115°F	Asphaltenes %	Solubility Parameter of Asphaltenes
Calif.Coastal	68	84	1400	439	75	0.5	221	7	15	28	43.1	7.97
" "	62	69	1400	483	75	1.0	221	7	12	26	42.7	7.95
" "	50	64	1400	531	75	1.0	223	6	10	22	44.0	8.02
" "	53	390	0	437	75	10.0	219	5	10	22	42.8	7.97
" "	44	300	0	482	75	11.0	223	4	9	17	43.7	7.98
" "	41	225	0	518	75	11.0	225	3	6	12	44.3	7.99
Talco	50	87	1400	432	75	0.5	217	9	21	34	39.2	7.79
"	48	65	1400	478	75	0.5	217	13	20	34	39.7	7.81
"	41	60	1400	530	75	0.5	226	10	16	28	40.7	7.80
"	38	919	0	436	75	2.0	225	9	16	28	41.1	7.80
"	45	600	0	472	75	2.0	219	11	17	33	40.0	7.80
"	32	453	0	526	75	1.0	223	10	16	26	41.6	7.82
Tia Juana	53	76	1400	439	75	0.5	217	14	22	38	34.6	7.88
" "	50	64	1400	475	75	1.5	217	13	21	37	35.2	7.83
" "	47	52	1400	529	75	0.5	219	12	20	37	35.2	7.80
" "	58	670	0	435	75	2.0	217	12	20	37	35.2	7.81
" "	59	390	0	470	75	1.0	218	13	21	38	34.3	7.78
" "	46	270	0	521	75	0.5	217	12	19	34	35.2	7.82
Kansas	81	110	1400	437	75	0.0	221	12	19	35	33.2	7.76
"	81	81	1400	473	75	1.0	219	12	21	36	34.5	7.79
"	78	46	1400	526	75	0.0	217	13	22	38	32.6	7.78
"	85	690	0	437	75	4.0	220	12	20	34	34.1	7.76
"	87	562	0	473	75	1.5	217	12	21	34	33.8	7.74
"	75	295	0	526	75	1.0	219	12	21	32	33.8	7.78

* Approximately to nearest 0.5%

TABLE 3. BLOWN ASPHALT CHARACTERISTICS

EFFECT OF AGITATION

Flux Identification	Durability (51-9c), Days	Blowing Conditions: Time, min.	Agitation, r.p.m.	Temp., °F	Air Rate ft^3/ton-min.	Weight Loss*, %	S.P., °F	Penetrations: at 32°F	at 77°F	at 115°F	Asphaltenes %	Solubility Parameter of Asphaltenes
Calif.Coastal	44	300	0	482	75	11.0	223	4	9	17	43.7	7.98
" "	54	104	900	484	75	1.0	221	7	12	24	42.9	7.95
" "	62	69	1400	483	75	1.0	221	7	12	26	42.7	7.95
" "	67	42	2200	479	75	1.5	217	7	14	27	42.0	7.96
Talco	45	600	0	473	75	2.0	219	11	17	33	40.0	7.80
"	44	125	900	472	75	4.0	219	10	20	29	40.4	7.82
"	48	65	1400	478	75	0.5	217	13	20	34	39.7	7.81
"	47	56	2200	472	75	0.0	217	14	21	36	39.6	7.80
Tia Juana	59	390	0	470	75	1.0	218	13	21	38	34.3	7.78
" "	58	124	900	470	75	0.5	221	12	19	34	35.4	7.81
" "	50	64	1400	475	75	1.5	217	13	21	37	35.2	7.83
" "	55	52	2200	472	75	2.0	219	12	20	36	35.6	7.83
Kansas	87	562	0	473	75	1.0	217	12	20	34	33.8	7.74
"	81	124	900	473	75	0.5	219	12	22	36	32.4	7.78
"	81	81	1400	473	75	1.5	219	12	21	36	34.5	7.79
"	82	55	2200	473	75	2.0	220	12	22	36	32.9	7.76

* Approximately to nearest 0.5%

TABLE 4. BLOWN ASPHALT CHARACTERISTICS

EFFECT OF AIR RATE

Flux Identification	Durability (51-9c), Days	Blowing Conditions: Time, min.	Blowing Conditions: Agitation, r.p.m.	Blowing Conditions: Temp., °F	Blowing Conditions: Air Rate ft^3/ton-min.	Weight Loss*, %	S.P., °F	Penetrations at 32°F	Penetrations at 77°F	Penetrations at 115°F	Asphaltenes %	Solubility Parameter of Asphaltenes
Calif.Coastal	61	117	1400	480	38	0.5	223	6	14	25	42.5	7.97
" "	62	69	1400	483	75	1.0	221	7	12	26	42.7	7.95
" "	67	61	1400	479	150	-	223	6	16	25	42.7	7.97
" "	39	810	0	472	38	-	223	4	8	18	44.7	7.94
" "	44	300	0	482	75	11.0	223	4	9	17	43.7	7.98
" "	44	240	0	468	150	5.0	217	4	8	18	44.1	7.98
Talco	49	107	1400	471	38	0.5	217	14	23	33	40.1	7.81
"	48	65	1400	478	75	0.5	217	13	20	34	39.7	7.81
"	47	55	1400	473	150	1.0	219	12	20	32	40.4	7.81
"	49	1200	0	473	38	5.0	217	11	18	33	40.9	7.82
"	45	600	0	472	75	2.0	219	11	17	33	40.0	7.80
"	56	317	0	471	150	1.0	219	12	19	36	40.0	7.77
Tia Juana	51	96	1400	472	38	3.0	217	12	22	35	35.4	7.86
" "	50	64	1400	475	75	1.5	217	13	21	37	35.2	7.83
" "	52	56	1400	472	150	1.0	217	13	22	37	35.1	7.83
" "	51	915	0	472	38	1.0	221	12	19	33	36.8	7.77
" "	59	390	0	470	75	1.0	218	13	21	38	34.3	7.78
" "	60	206	0	472	150	0.5	217	14	22	39	35.6	7.82
Kansas	81	119	1400	472	38	0.5	222	13	20	34	32.8	7.77
"	81	81	1400	473	75	1.0	219	12	21	36	34.5	7.79
"	73	68	1400	472	150	3.0	221	12	20	34	33.4	7.79
"	80	944	0	472	38	0.0	217	13	20	35	33.3	7.73
"	87	562	0	473	75	1.5	217	12	21	34	33.8	7.74
"	65	330	0	475	150	0.0	221	11	19	29	34.6	7.77

* Approximately to nearest 0.5%

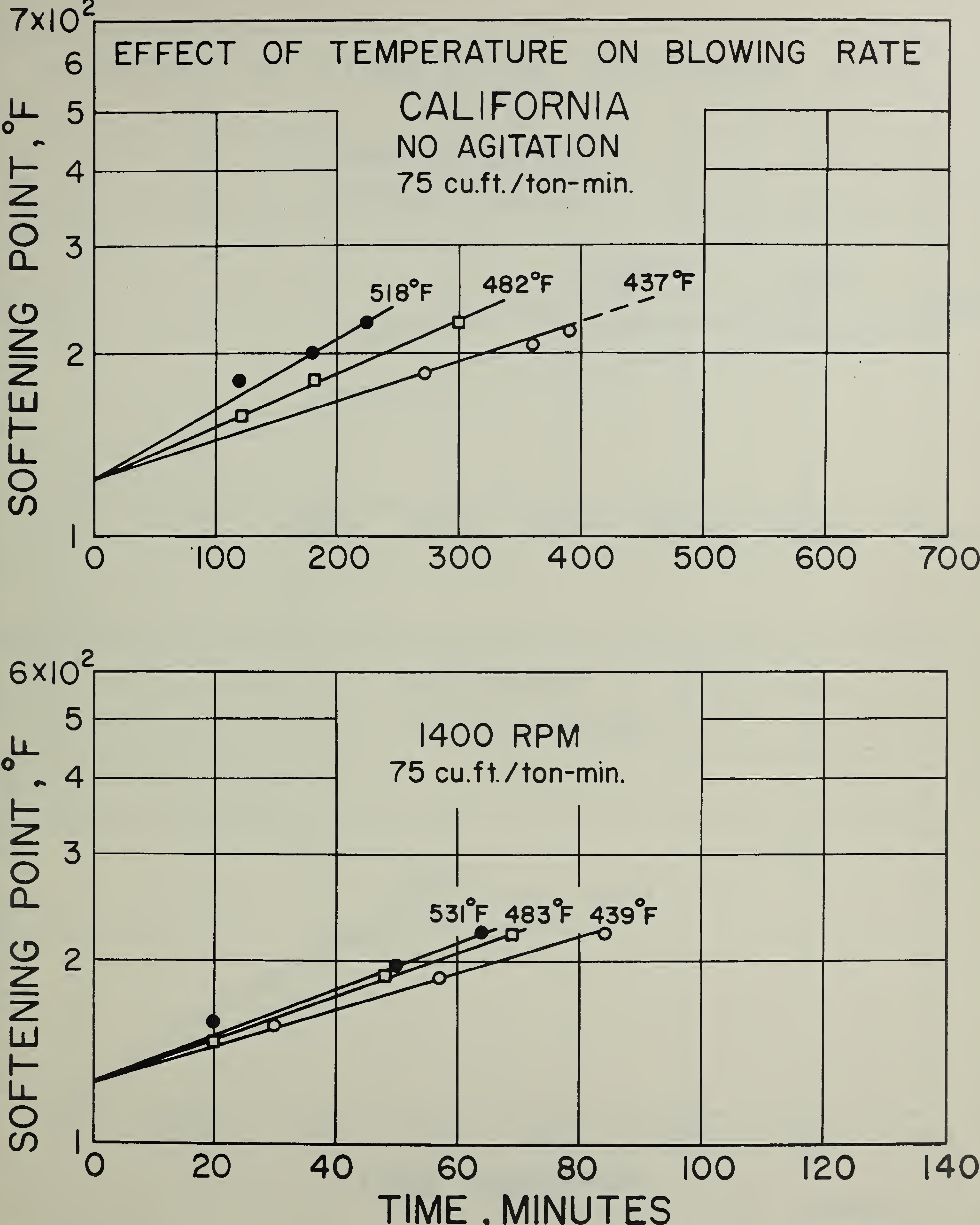

EFFECT OF TEMPERATURE ON BLOWING RATE
CALIFORNIA
NO AGITATION
75 cu.ft./ton-min.
518°F
482°F
437°F
SOFTENING POINT, °F
7x10²
6
5
4
3
2
1
0
100
200
300
400
500
600
700
1400 RPM
75 cu.ft./ton-min.
531°F
483°F
439°F
SOFTENING POINT, °F
6x10²
5
4
3
2
1
0
20
40
60
80
100
120
140
TIME, MINUTES

FIGURE 3

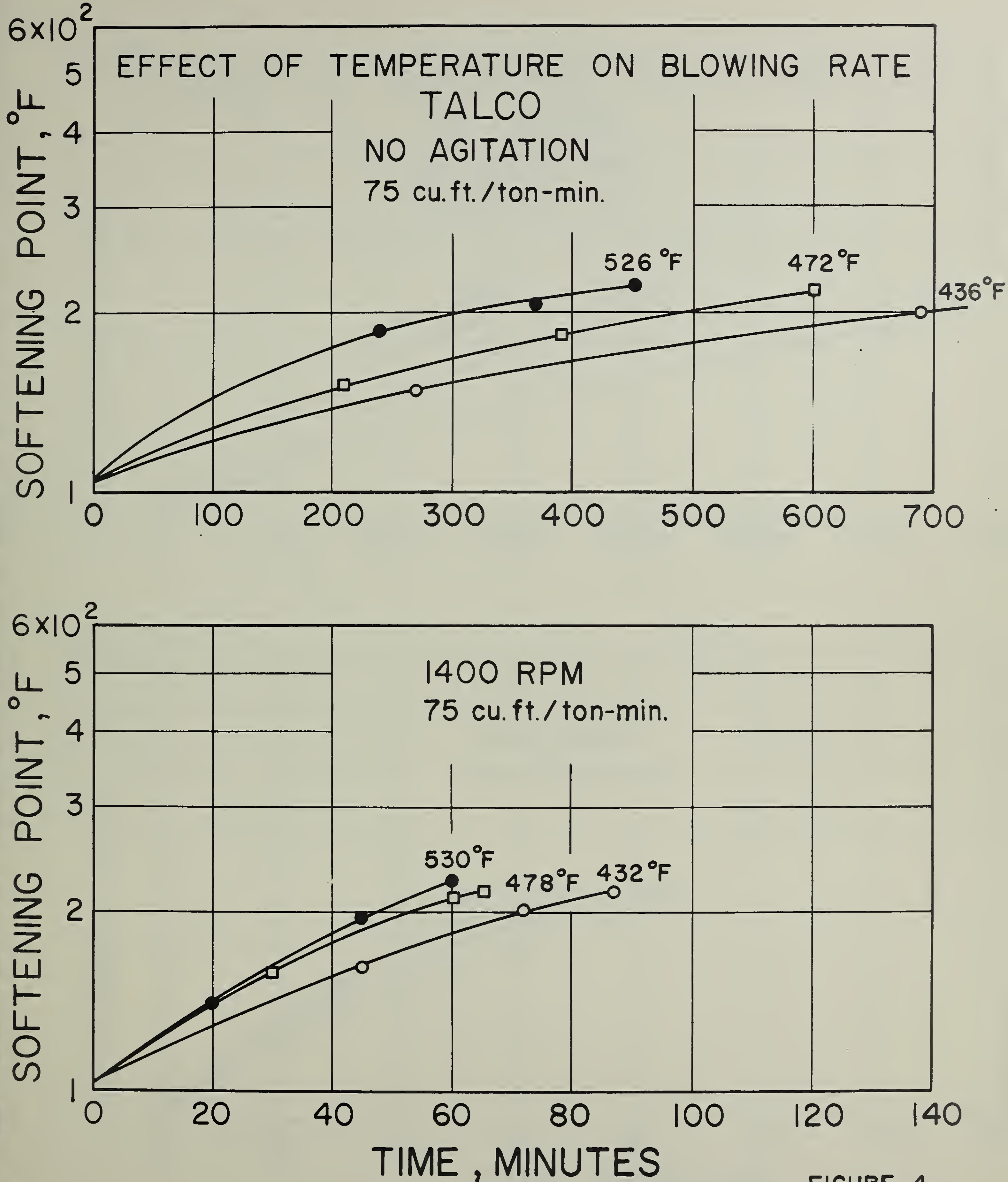
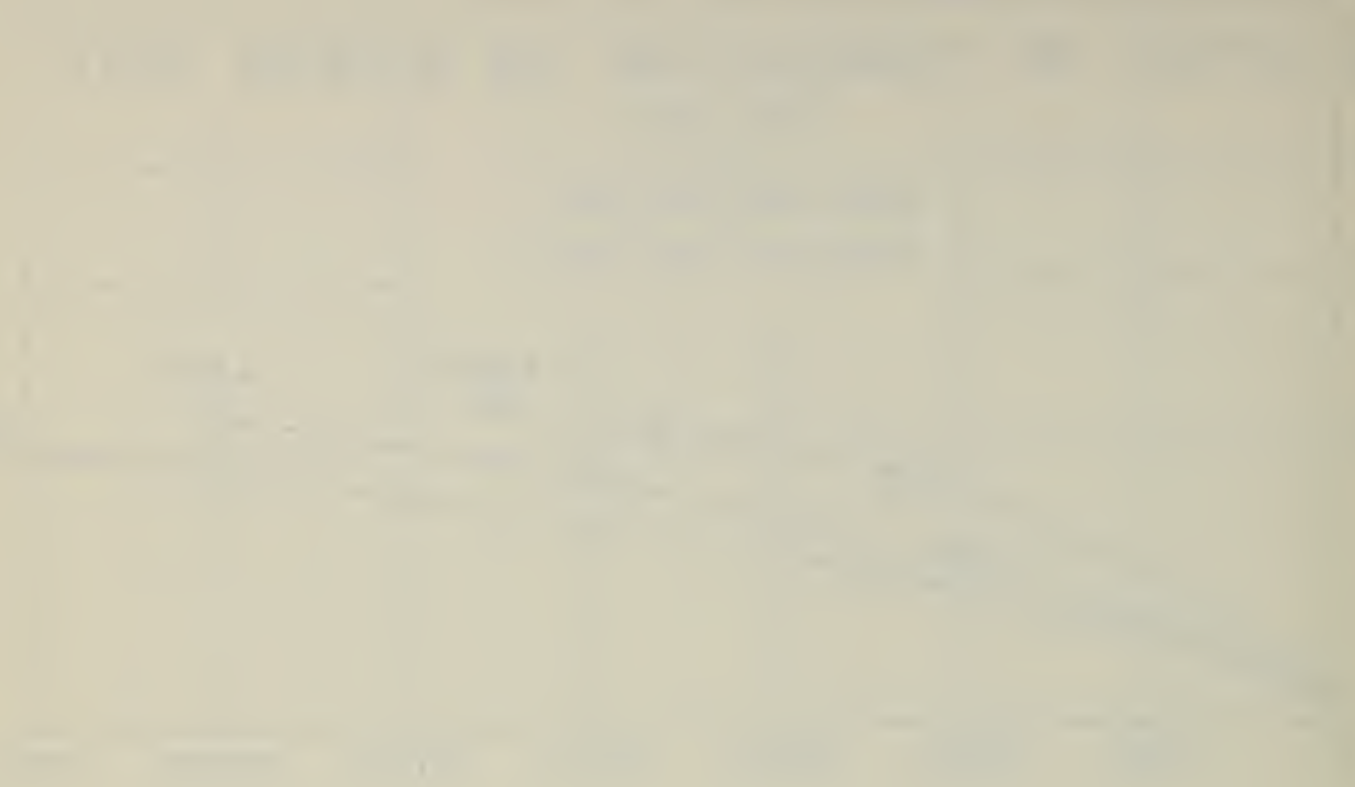
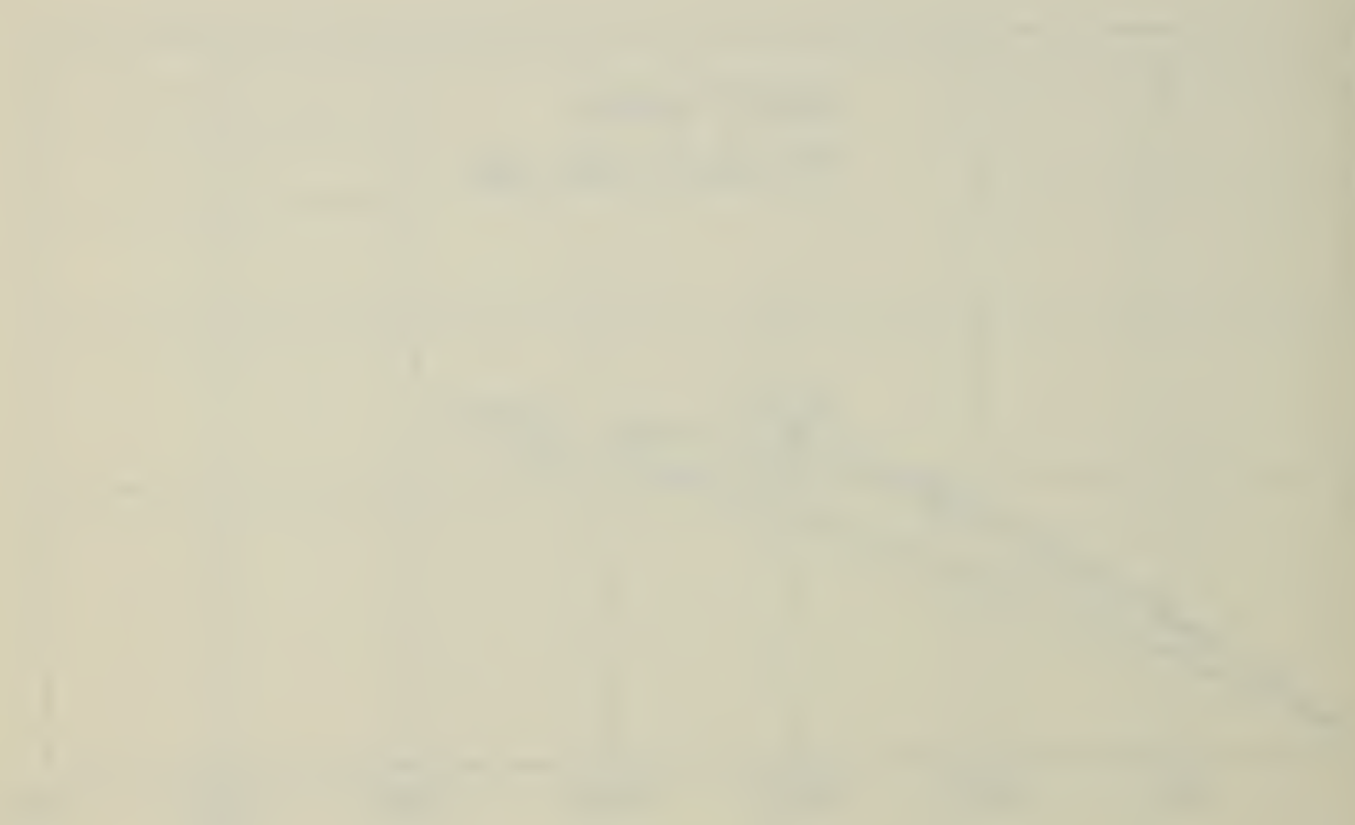
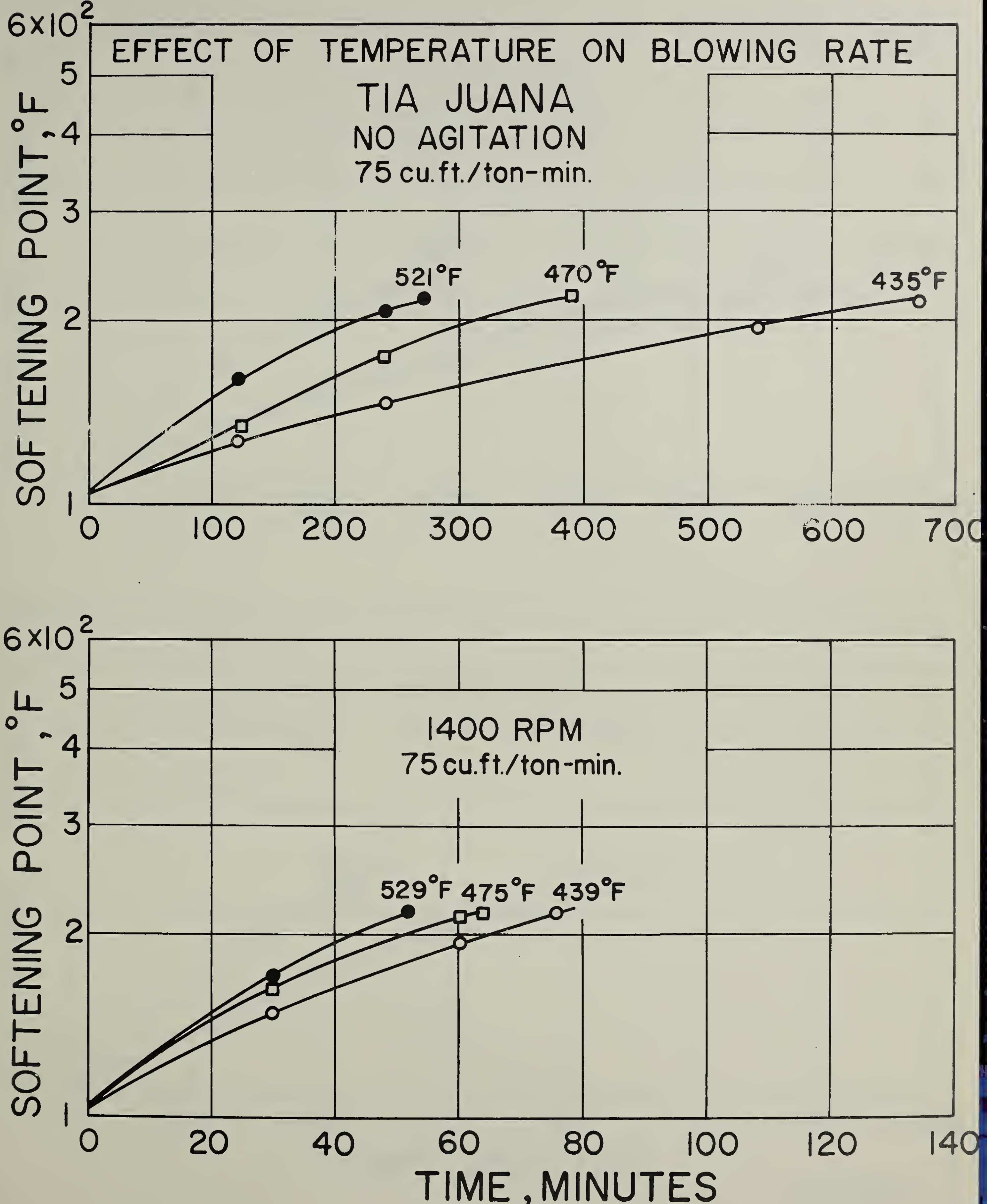

EFFECT OF TEMPERATURE ON BLOWING RATE
TALCO
NO AGITATION
75 cu.ft./ton-min.
526 °F
472 °F
436 °F
SOFTENING POINT, °F
6x10^2
5
4
3
2
1
0
100
200
300
400
500
600
700
1400 RPM
75 cu.ft./ton-min.
530 °F
478 °F
432 °F
SOFTENING POINT, °F
6x10^2
5
4
3
2
1
0
20
40
60
80
100
120
140
TIME, MINUTES

FIGURE 4







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

FIGURE 5

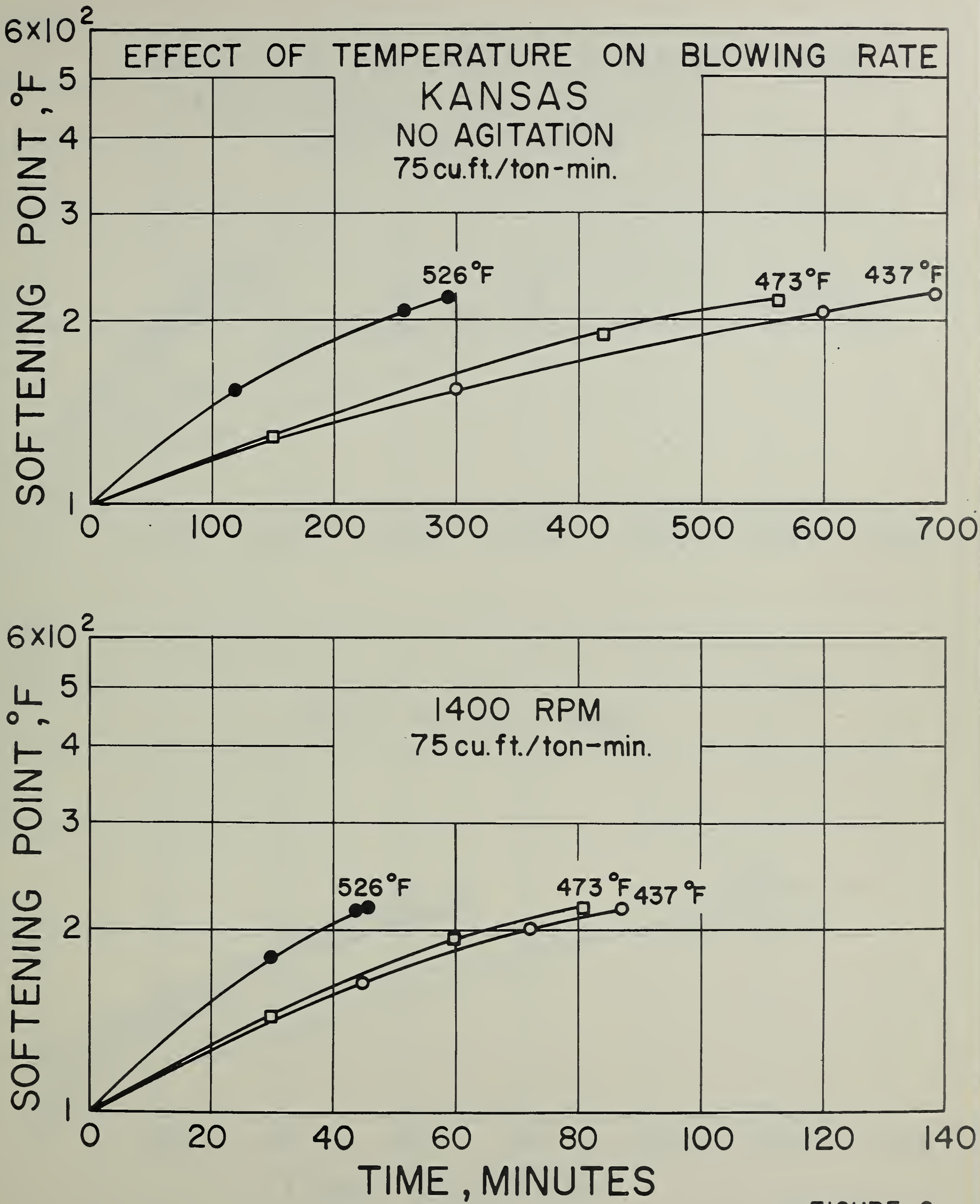

EFFECT OF TEMPERATURE ON BLOWING RATE
KANSAS
NO AGITATION
75 cu.ft./ton-min.
SOFTENING POINT, °F
6x10²
5
4
3
2
1
526°F
473°F
437°F
0
100
200
300
400
500
600
700
1400 RPM
75 cu.ft./ton-min.
SOFTENING POINT, °F
6x10²
5
4
3
2
1
526°F
473°F
437°F
0
20
40
60
80
100
120
140
TIME, MINUTES

FIGURE 6

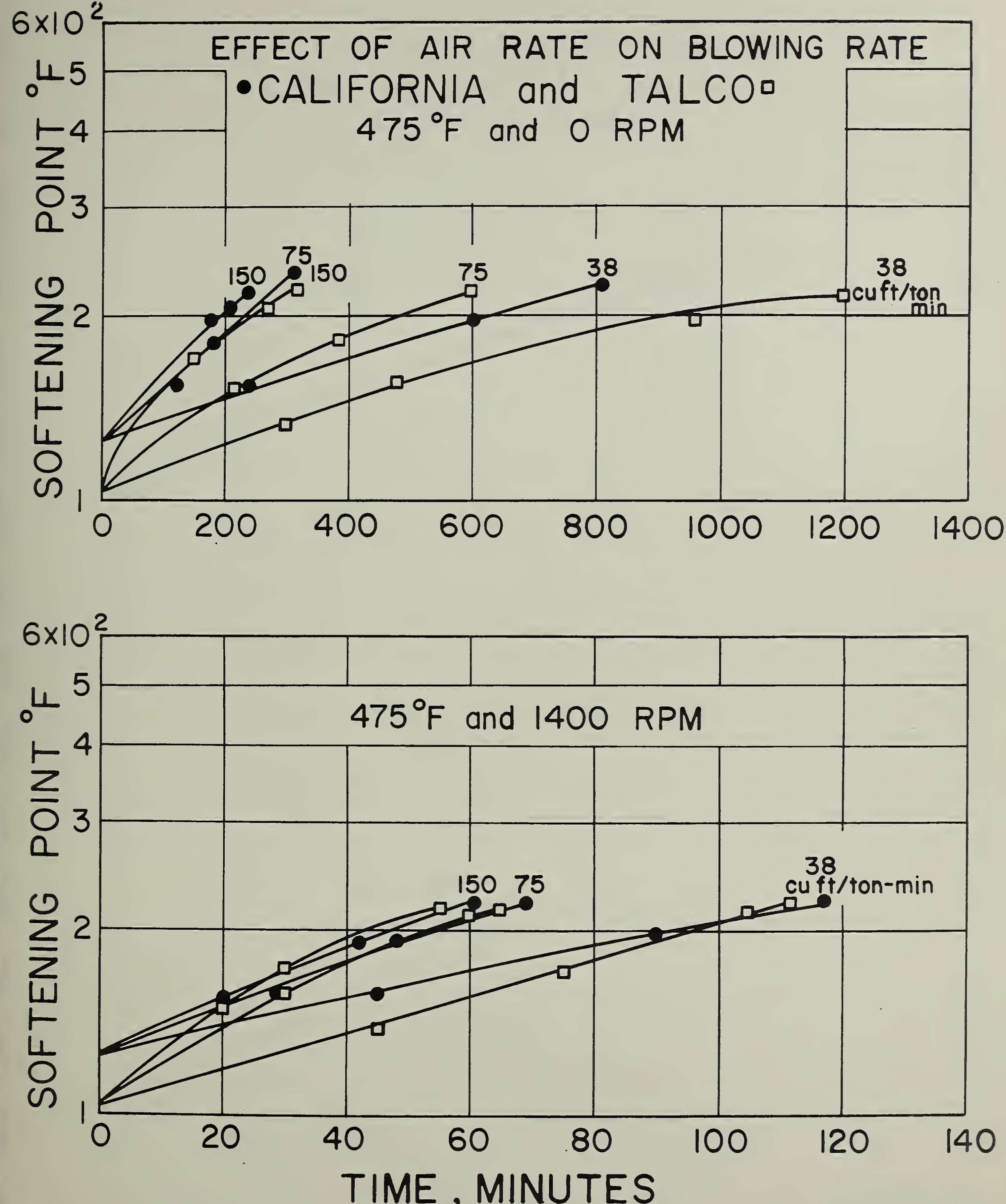
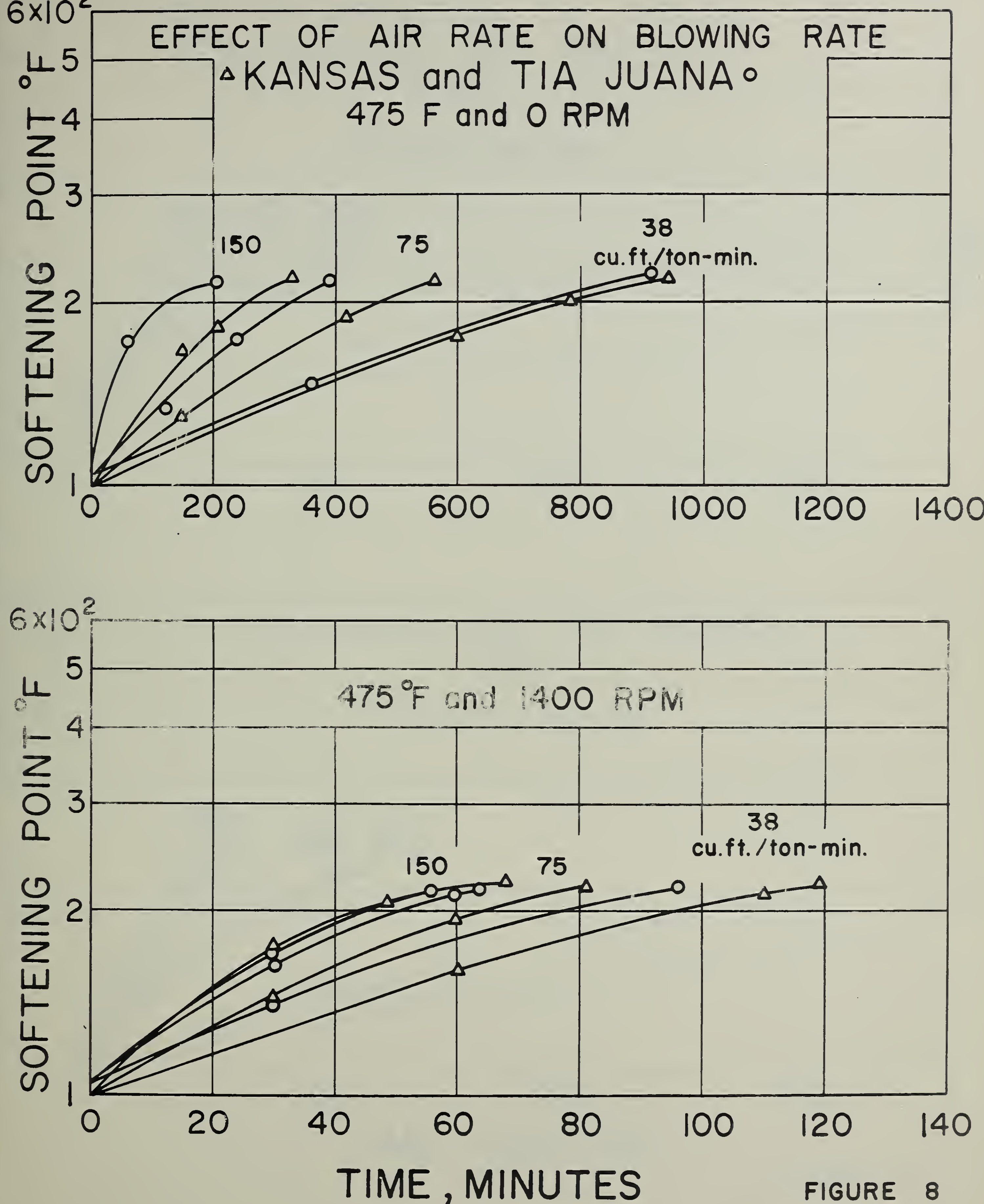
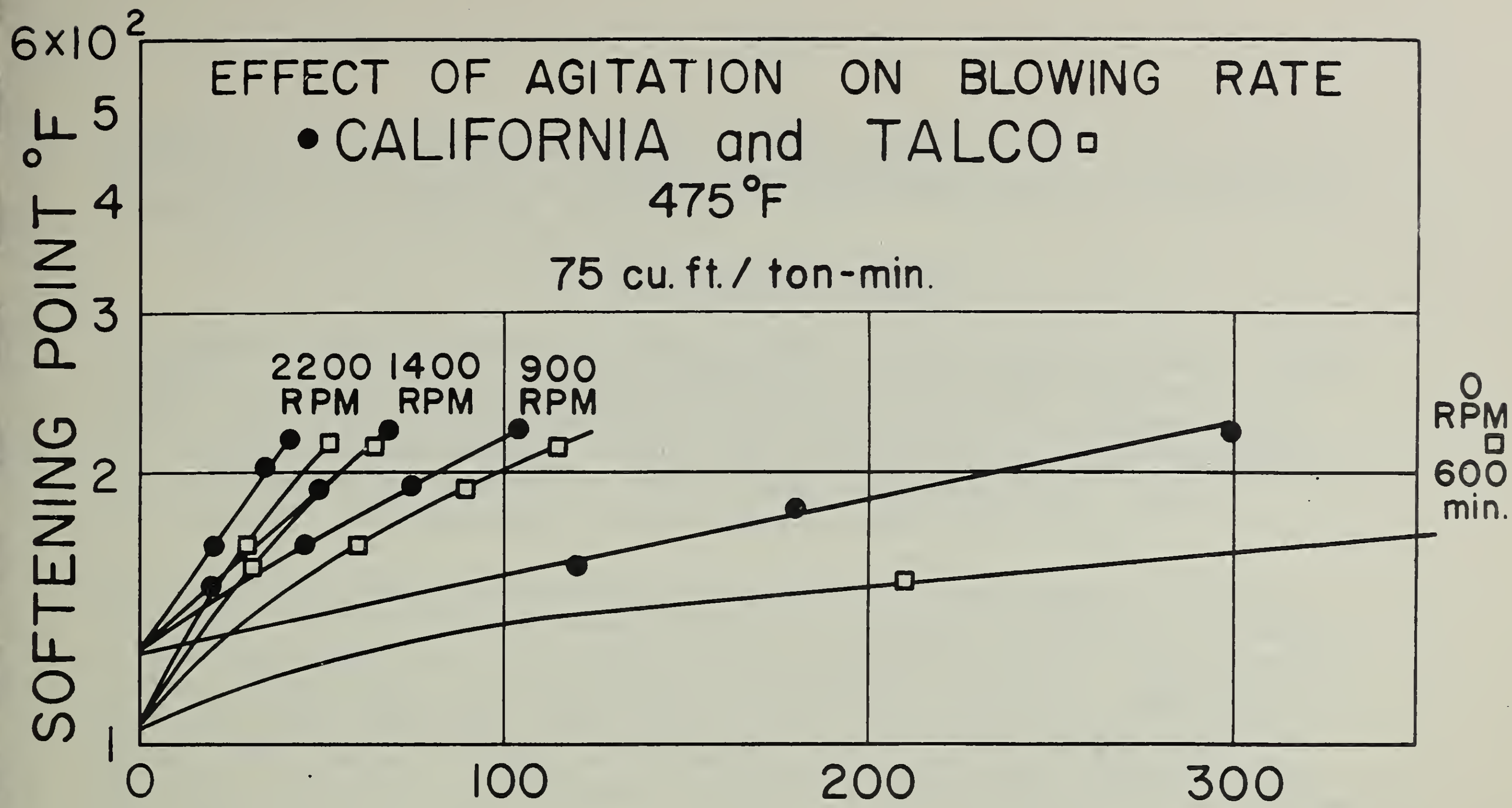

EFFECT OF AIR RATE ON BLOWING RATE
•CALIFORNIA and TALCO□
475 °F and 0 RPM
SOFTENING POINT °F
6x10²
5
4
3
2
1
0
200
400
600
800
1000
1200
1400
150
75
150
75
38
38
cu ft/ton
min
475 °F and 1400 RPM
SOFTENING POINT °F
6x10²
5
4
3
2
1
0
20
40
60
80
100
120
140
150
75
38
cu ft/ton-min
TIME, MINUTES

FIGURE 7


EFFECT OF AIR RATE ON BLOWING RATE
△ KANSAS and TIA JUANA ○
475 F and 0 RPM
SOFTENING POINT °F
6x10²
5
4
3
2
1
150
75
38
cu.ft./ton-min.
0
200
400
600
800
1000
1200
1400
475 °F and 1400 RPM
SOFTENING POINT °F
6x10²
5
4
3
2
1
150
75
38
cu.ft./ton-min.
0
20
40
60
80
100
120
140
TIME, MINUTES
FIGURE 8


EFFECT OF AGITATION ON BLOWING RATE
• CALIFORNIA and TALCO □
475 °F
75 cu. ft. / ton-min.
SOFTENING POINT °F
6x10²
5
4
3
2
1
2200 RPM
1400 RPM
900 RPM
0 RPM
□ 600 min.
0
100
200
300

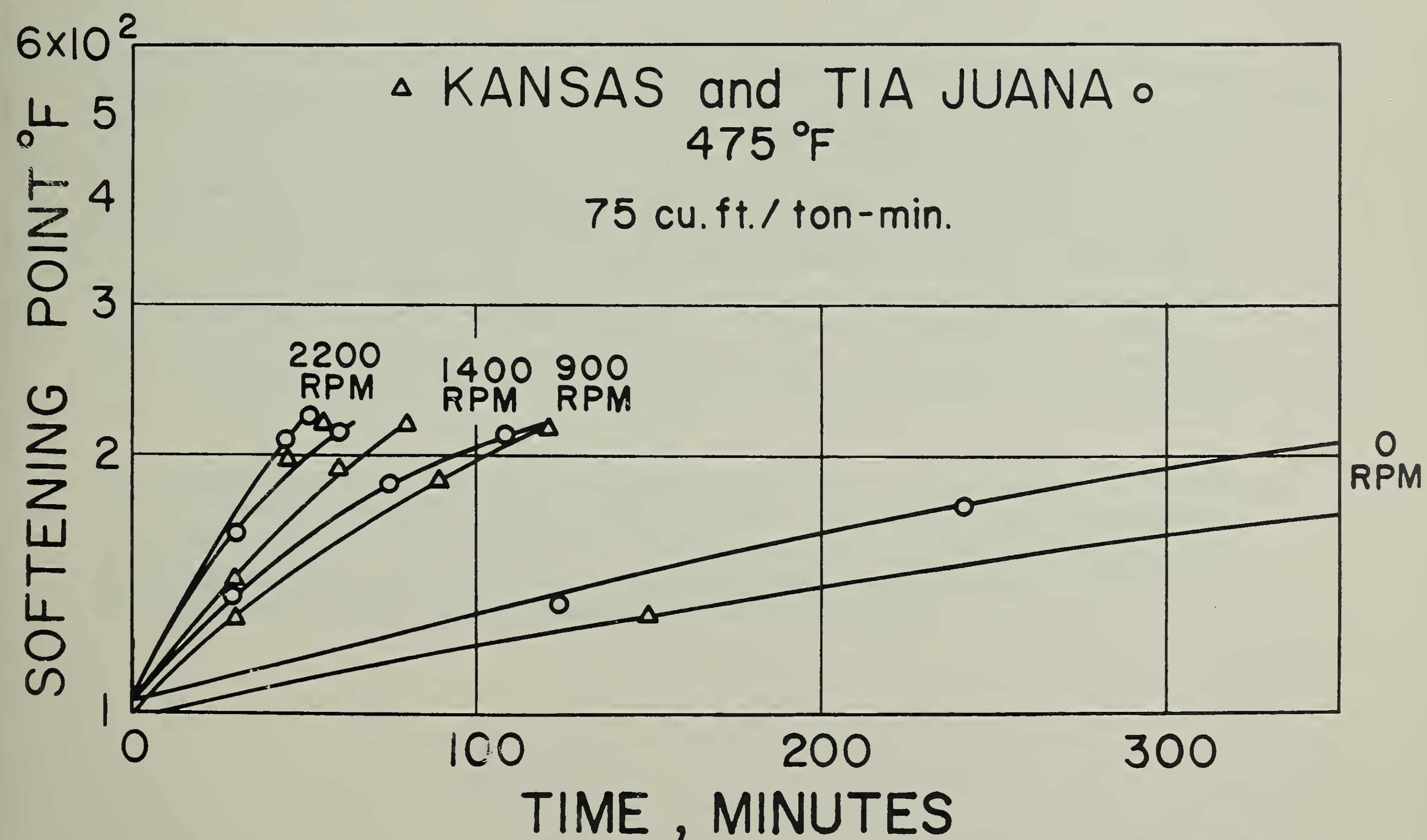

△ KANSAS and TIA JUANA ○
475 °F
75 cu. ft. / ton-min.
SOFTENING POINT °F
6x10²
5
4
3
2
1
2200 RPM
1400 RPM
900 RPM
0 RPM
0
100
200
300
TIME, MINUTES

FIGURE 9

Figures 10, 11, and 12 are graphical presentations of the time required to blow each of the fluxes to coating grade softening points as functions of temperature, air rate and agitation.

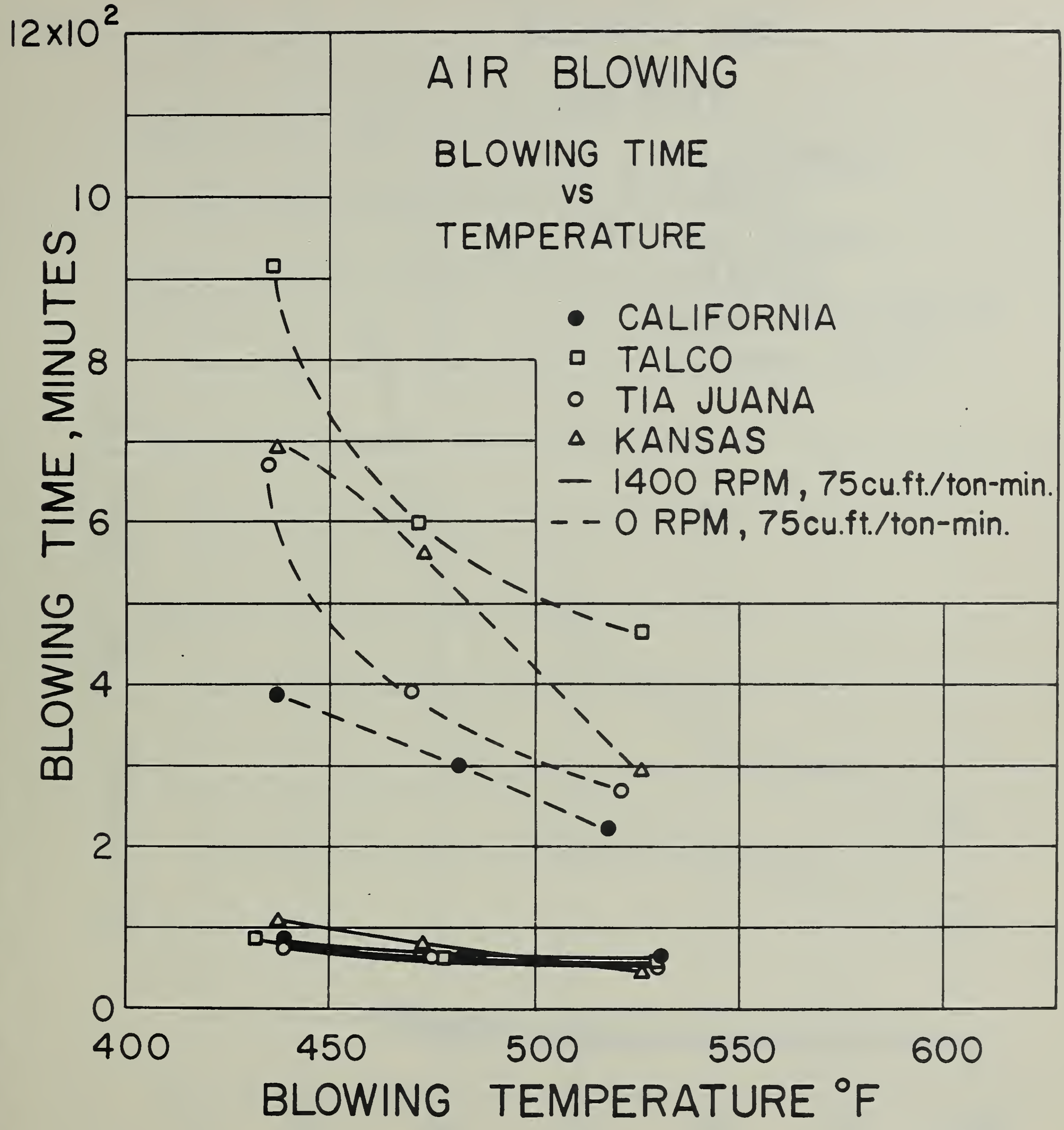
6. DISCUSSION OF RESULTS

No general specification for coating-grade roofing asphalts exists. Each company that purchases these asphalts has its own specifications. However, these cover narrow ranges of softening points and penetrations. For purposes of discussion, a broad specification will be assumed which will encompass the majority of the company specifications. Such a hypothetical specification is reproduced in Table 5.

The softening point-penetration relationships of the asphalts will be discussed relative to this specification. As seen in Tables 2, 3, and 4, all of the Kansas and Tia Juana asphalts complied with the specification; their penetrations were essentially independent of blowing conditions. Only the low-temperature penetration of the two Talco asphalts blown at about 430°F failed to meet this specification, and they failed by only one unit. None of the California asphalts met the penetration requirements. The necessity of a flash point in excess of 450°F precluded the production of a flux from this source which could be blown to grade without modification.

Unlike the Kansas and Tia Juana fluxes, those from California and Talco fields produced products the penetrations of which varied with the conditions under which they were blown. As the blowing temperature was increased from 430°F to 530°F the penetrations of the California products blown to approximately a 220°F softening point decreased. Those blown without agitation had lower penetrations than the corresponding products blown with agitation. These lower values were probably related to the much longer residence times in the stills during the unagitated runs and the correspondingly higher weight losses. As seen in Tables 2, 3, and 4, the weight losses for the unagitated runs with the California flux were of the order of 10%, while the California asphalts blown with agitation lost 1.5% or less.

The penetrations of the Talco asphalts increased as the speed of agitation was increased. Except for the 900 rpm run, the weight losses decreased correspondingly. Temperature, in the absence of agitation, had little effect on penetration; but with agitation, the penetrations at 77°F dropped appreciably as the temperature was increased.



AIR BLOWING
BLOWING TIME
VS
TEMPERATURE
CALIFORNIA
TALCO
TIA JUANA
KANSAS
1400 RPM, 75cu.ft./ton-min.
0 RPM, 75cu.ft./ton-min.
BLOWING TIME, MINUTES
12x10^2
10
8
6
4
2
0
400
450
500
550
600
BLOWING TEMPERATURE °F

FIGURE 10

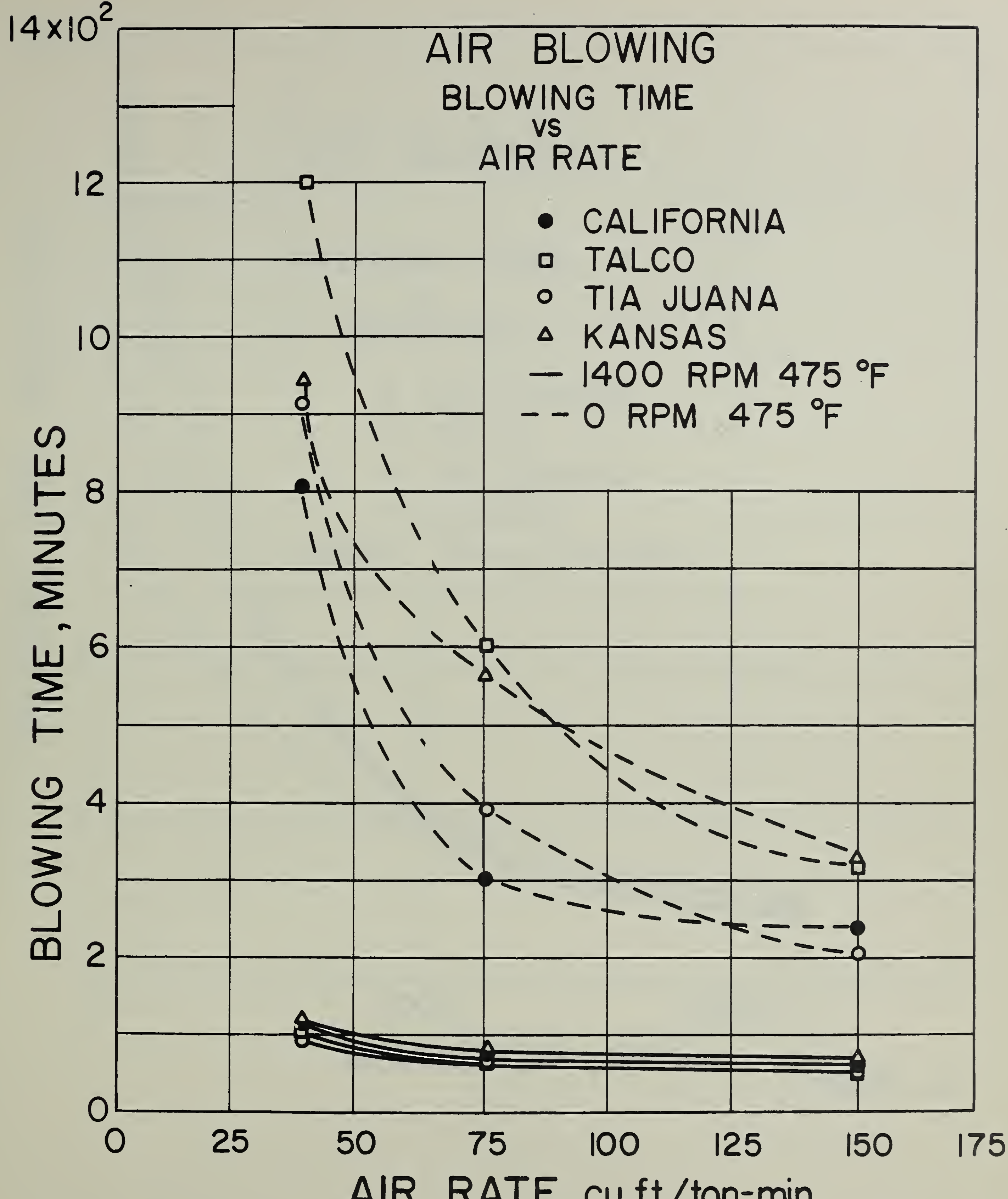
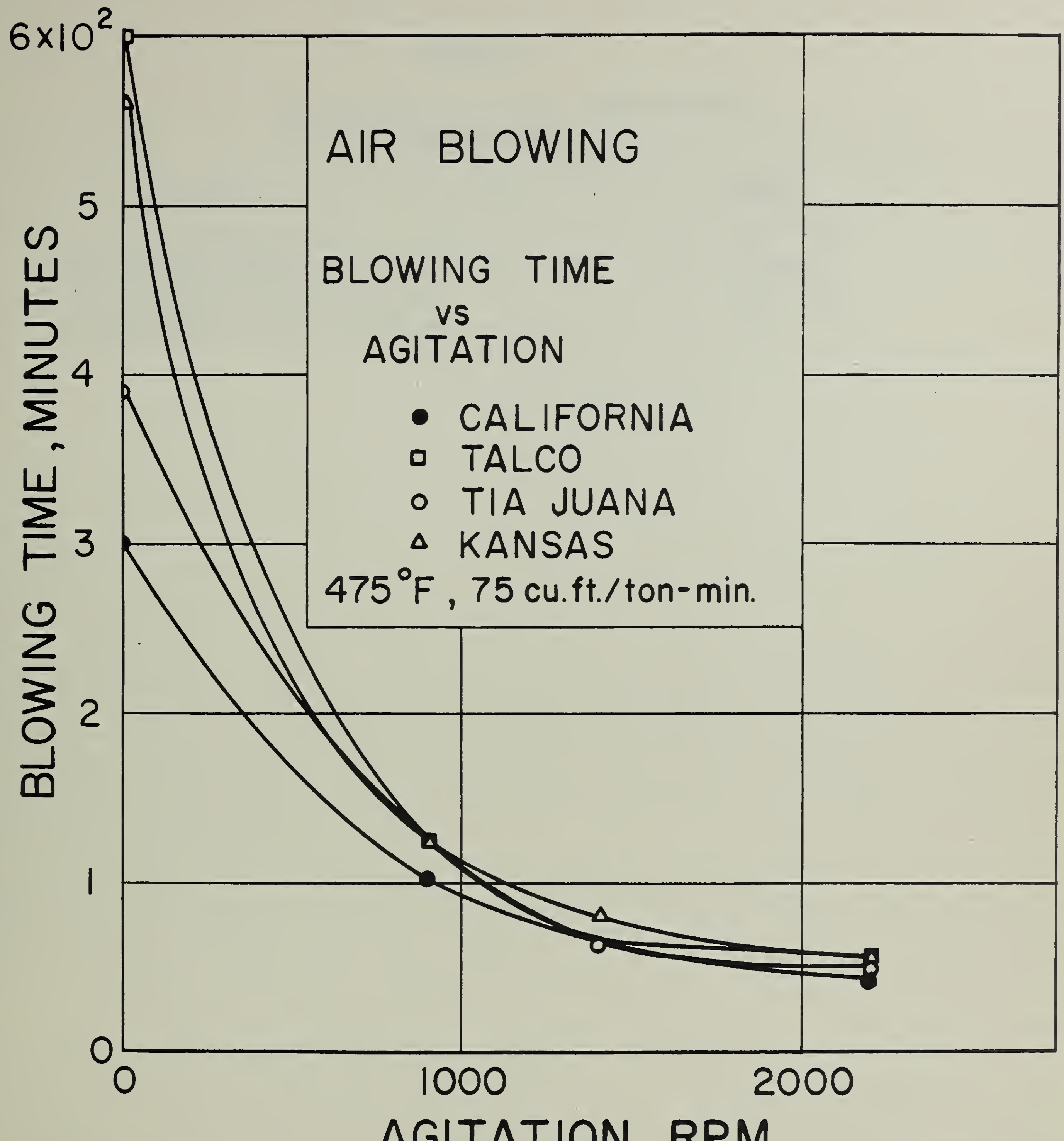

AIR BLOWING
BLOWING TIME
VS
AIR RATE
CALIFORNIA
TALCO
TIA JUANA
KANSAS
1400 RPM 475 °F
0 RPM 475 °F
14x10^2
12
10
8
6
4
2
0
BLOWING TIME, MINUTES
0
25
50
75
100
125
150
175
AIR RATE, cu. ft./ton-min.

FIGURE 11



AIR BLOWING
BLOWING TIME
VS
AGITATION
CALIFORNIA
TALCO
TIA JUANA
KANSAS
475°F , 75 cu.ft./ton-min.
6x10²
5
4
3
2
1
0
BLOWING TIME, MINUTES
0
1000
2000
AGITATION, RPM

FIGURE 12

TABLE 5.

Hypothetical Coating - Grade Asphalt Specification

Property	Range
Softening Point, °F	215 - 225
Penetration @ 32°F, 1/10 mm	10 min
Penetration @ 77°F, 1/10 mm	15 - 25
Penetration @ 115°F, 1/10 mm	25 - 45

The weight losses for all of the runs except those involving California asphalt without agitation were reasonably low. These losses were a little erratic because of the variable holdup in the stills, which was largely dependent on the temperature at which the asphalts were discharged. The first run in a completely clean still always had a high holdup; this situation was corrected in the approximate loss figures reported. In two of the runs some undeterminable transfer loss occurred and, consequently, no loss figures are reported.

In 1959, Greenfeld, Mertens, and John (17) published an article pertaining to the statistical considerations involved in the comparison of the durabilities of asphalts exposed in Weather-Ometers. Since that time, modifications in the evaluation procedure have occurred. The statistics, therefore, are not strictly applicable to the results obtained in this study. However, for discussion purposes, it will be assumed the variability of these results is not greater than for the results analyzed and, thus, the average of one set of four specimens must be at least 12 percent greater than that of another set of four specimens in order for the first material to be truly more durable than the second.

The durabilities of the blown asphalts produced from the four fluxes covered a range of 32 to 87 days. The Kansas flux produced the more durable asphalts while the Talco flux produced the least durable ones. However, there was an overlapping of the California, Talco and Tia Juana products. From the California flux, asphalts were produced which were more durable than the best Tia Juana product and others which were less durable than the least durable Tia Juana.

The durabilities of all of the asphalts varied with the temperatures at which they were blown. In general, the durability was lowest for the asphalts blown at about 525°F and highest for those blown at about 435°F. There was usually less difference between the asphalts blown from a single source at 435 and 475°F than between those blown at 475 and 525°F.

The largest effect of blowing temperature was observed in the California asphalts (Table 2). The durability was highest for the product produced at the low blowing temperature with agitation and lowest for the asphalt produced at the high temperature without agitation. The former, with a durability of 68 days, was two-thirds more durable than the latter (41 days).

A similar set of results was obtained for the asphalts blown from the Talco source. The most durable, 50 days, was produced at the lowest temperature with agitation, and the least durable was produced at the highest temperature without agitation (32 days).

The Tia Juana and Kansas fluxes were less sensitive to blowing temperature than the California and Talco asphalts. In both of these the most durable asphalts were produced by blowing at the intermediate (475°F) temperature without agitation and the least durable products were produced at the highest blowing temperature without agitation. All of the products blown with agitation were of intermediate durabilities. When agitation was used, the durabilities of asphalts made from either of these fluxes were independent of blowing temperature and in the middle of the durability ranges of the respective products blown without agitation.

As seen in Table 3, durability was independent of the degree of agitation for three of the four asphalts; only for California asphalt was durability found to vary with the degree of agitation during blowing. Durability increased from 44 days when blown without agitation to 67 days when blown with the agitators rotating at 2200 rpm. While none of the California products had penetrations high enough to meet the minimum requirements of a coating-grade material, the most durable product had the highest penetrations.

As seen in Table 4, the durabilities of the asphalts blown with agitation were independent of the air rate in the range of 38 to 150 ft^3/ton-min. In the absence of agitation the durabilities of the California and Talco asphalt products were again independent of air rate. However, the durabilities of the products blown from Tia Juana asphalt were higher at the higher air rate. The durability of the Kansas product blown at the highest air rate, conversely, was lowest.

Each flux possessed an optimum set of blowing conditions with which asphalts of higher durability could be produced. For some, the ranges of variables were quite large; for others, they were rather narrow. The conditions under which the more durable products were made from each of the four fluxes studied are summarized in Table 6.

There was no general correlation of either solubility parameter or asphaltene content with durability. Both of these variables seemed to be related to the asphalt source and little affected by the processing. Even the loss of as much as 11% of the flux through volatilization that occurred during the blowing of the California asphalt did not noticeably affect these variables. Mertens (18) and Greenfeld and Wright (16) reported correlations of durability with solubility parameter of the asphaltenes and Wilkinson, Striker, and Traxler (19), and Greenfeld and Wright (16) with asphaltene content. Greenfeld and Wright (16) pointed out the tendency of the various asphalts from the same area to group together in these correlations. Apparently, the variations in the processing of the commercial samples used in these studies contributed to the differences in durability, but the inherent solubility parameters and asphaltene contents remained essentially unmodified.

TABLE 6.

BLOWING CONDITIONS PRODUCING MORE DURABLE ASPHALTS

Source	Durability, (51-9c), Days	Temperature °F	Agitation rpm	Air Rate ft^3/ton-min.
California	62-68	439-483	1400-2200	75-150
Kansas	80-87	437-475	0-2200	38-75
Talco	50-56	432	0	150
Tia Juana	58-60	435-470	0-900	75-150

7. SUMMARY AND CONCLUSIONS

The effects of varying the temperature, air rate and amount of agitation during blowing on the physical properties and durability of asphalts produced from four fluxes were studied. Each flux responded differently.

The California flux was most sensitive to changes in these parameters. The durability of its products decreased as the blowing temperature was increased from 430 to 530°F, was independent of the air rate in the range of 38 to 75 cubic feet per ton minute and increased as the rate of mechanical agitation was increased from zero to 2200 rpm by three, three-bladed turbo-mixer type impellers.

The Talco flux was next most sensitive to the variables. The durability of its asphalts decreased as the blowing temperature was increased and was independent of air rate and agitation.

The Tia Juana flux was relatively unresponsive to blowing temperature, but the blown products did suffer a loss of durability at highest blowing temperatures (520°F) in the absence of agitation. Tia Juana produced the more durable products at the lower rates of agitation and, in the absence of agitation, at the higher air rates.

The durabilities of the Kansas asphalts were relatively independent of all three variables in the lower two-thirds of their ranges, but the asphalts produced at the highest blowing temperature and the highest air rate were less durable than the others.

The ranges of durability of the asphalts produced from the four fluxes were as follows:

California	39 - 68 days
Kansas	65 - 87 days
Talco	32 - 56 days
Tia Juana	46 - 60 days

The blowing time to produce asphalts of approximately 220°F softening point varied from 42 to 125 minutes with agitation and 225 to 1200 minutes without agitation. The blowing times decreased as the temperature, air rate or agitation was increased.

The Kansas and Tia Juana asphalts produced under all conditions had penetrations within the normal coating-grade ranges. The penetrations of the Talco asphalts were within the coating-grade ranges under most conditions, but tended toward the lower limits when blown at the highest and lowest

temperatures. The California flux always produced asphalts with penetrations lower than desirable. When agitation was used, however, the penetrations were higher than for the asphalts produced under corresponding conditions without agitation.

Neither the asphaltene content nor the solubility parameter of the asphaltenes correlated with durability; they seemed to be more characteristic of the source than of the processing variations.

8. ACKNOWLEDGMENT

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