NATIONAL BUREAU OF STANDARDS REPORT

6523

Study of the Properties of Rubber Base Calking Materials
(Progress Report No. 1)

by

Arthur Hockman

Report to
the Departments of
the Air Force, the Army, and the Navy

NBS

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

THE NATIONAL BUREAU OF STANDARDS

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NATIONAL BUREAU OF STANDARDS

Identification of Samples of Rubber Base Calkings (Tri-Service Project)

Sample No.	Producer	Type	Color	Class
1-A	Pecora Paint Company	Polysulfide Thiokol	Neutral	Non-sag
1-B	" " "	"	Aluminum	"
1-C	" " "	"	Brown	"
1-D	" " "	"	Gray	"
1-E	" " "	"	Black	"
1-F	" " "	"	Neutral	"
1-G	" " "	"	Aluminum	"
1-H	" " "	"	Black	"
2-A	Parr Paint and Color Co.	Polysulfide Thiokol	Aluminum	Non-sag
2-B	" " " " "	"	Gray	"
2-C	" " " " "	"	Tan	"
2-D	" " " " "	"	Black	"
2-E	" " " " "	"	Aluminum	"
2-F	" " " " "	"	Tan	"
2-G	" " " " "	"	Black	"
3-A	L. Sonneborn Company	Polysulfide Thiokol	Aluminum	Non-sag
3-B	" "	"	Gray	"
3-C	" "	"	Tan	"
3-D	" "	"	Black	"
3-E	" "	"	Aluminum	"
3-F	" "	"	Black	"
3-G	" "	"	Gray	"
3-H	" "	"	Tan	"
3-I	" "	"	Aluminum	"
3-J	" "	"	Gray	"
3-K	" "	"	Tan	"
3-L	" "	"	Black	"

Sample No.	Producer	Type	Color	Class
4-A	A. C. Horn Company	Polysulfide (Thiokol)	Gray	Non-sag
4-B	" " "	"	"	Non-sag (hard)
4-C	" " "	"	Aluminum	Non-sag
4-D	" " "	"	White	"
4-E	" " "	"	Black	"
4-F	" " "	"	Red	"
4-G	" " "	"	Aluminum	"
4-H	" " "	"	Gray	"
4-I	" " "	"	Black	"
5-A	Steelcote Mfg. Co.	Polysulfide (Thiokol)	Tan	Non-sag
5-B	" " "	"	Gray	Non-sag
5-C	" " "	"	Aluminum	"
5-D	" " "	"	Black	"
6-A	Products Research Co.	Polysulfide (Thiokol)	Neutral	Non-sag
6-B	" " "	"	"	Flow
6-C	" " "	"	Gray	Non-sag
6-D	" " "	"	"	Flow
6-E	" " "	"	Aluminum	Non-sag
6-F	" " "	"	Black	"
6-G	" " "	"	Black	Flow
6-H	" " "	"	Neutral	Non-sag
6-I	" " "	"	Aluminum	"
6-J	" " "	"	Gray	"
6-K	" " "	"	Black	"
6-L	" " "	"	Gray	"
6-M	" " "	"	Aluminum	"
6-N	" " "	"	Neutral	"
6-O	" " "	"	Black	"
6-P	" " "	"	Gray	Flow
7-A	Atlas Mineral Products	Polysulfide (Thiokol)	Black	Non-sag
8-A	Tremco Mfg. Co.	Polysulfide (Thiokol)	Aluminum	Non-sag (soft)
8-B	" " "	"	"	Non-sag (hard)
8-C	" " "	"	Brown	Non-sag (soft)
8-D	" " "	"	Black	Non-sag (soft)
8-E	" " "	"	"	Non-sag (hard)

Sample No.	Producer	Type	Color	Class
9-A	Presstite-Keystone Co.	Polysulfide (Thiokol)	Aluminum	Non-sag
9-B	"	"	Tan	"
9-C	"	"	Aluminum	"
9-D	"	"	"	Flow
9-E	"	"	Tan	"
9-F	"	"	Black	"
10-A	David E. Long	"	Gray	Non-sag
10-B	"	"	"	"
10-C	"	"	Aluminum	" (soft)
10-D	"	"	Black	"
10-E	"	"	"	Pourable
10-F	"	"	Gray	Non-sag
11-A	Dicks-Armstrong Pontius	"	"	"
11-B	"	"	"	Flow
11-C	"	"	Tan	Non-sag
11-D	"	"	Black	"
11-E	"	"	Gray	"
11-F	"	"	Aluminum	"
11-G	"	"	Tan	"
11-H	"	"	Black	"
12-A	Coast Pro-Seal Co.	"	Gray	"
12-B	"	"	Tan	"
12-C	"	"	Black	"
12-D	"	"	"	Flow
12-E	"	"	Tan	Non-sag
12-F	"	"	Black	"
12-G	"	"	Aluminum	"
12-H	"	"	Gold	"
13-A	Minnesota Mining and Manufacturing Co.	"	Tan	"
13-B	"	"	Gray	"
13-C	"	"	White	"
13-D	"	"	Aluminum	"
13-E	"	"	Light Gold	"
13-F	"	"	Black	"
13-G	"	"	Green	"
13-H	"	"	White	"
13-I	"	"	Gray	"
13-J	"	"	Aluminum	"

Sample No.	Producer	Type	Color	Class
14-A	Servicised Prod. Co.	Polysulfide (Thiokol)	White	Non-sag
14-B	"	"	Gray	"
14-C	"	"	Black	"
15-A	Armstrong Cork Co.	"	Aluminum	"
15-B	"	"	"	"
15-C	"	"	Black	"
15-D	"	"	"	"
15-E	"	"	Aluminum	"
16-A	Gera Manufacturing Co.	"	"	"
16-B	"	"	Black	"
17-A	Dow Corning	Silicone Rubber	Pink	"
17-B	"	"	White	"
18-A	Sika Chemical Corp.	Epoxy Resin	Tan	"
19-A	Stay-Tite Products Co.	Neoprene Rubber	Aluminum	"
20-B	Pittsburgh Plate Glass Co.	Butene Rubber	Black	"

US-COMM-NBS-DC

Study of the Properties of Rubber Base Calking Materials
(Progress Report No. 1)

Arthur Hockman

A study of the properties of rubber base joint sealers (calking compounds) for use in buildings and other types of structures is in progress at the National Bureau of Standards. For this purpose 130 samples from 19 producers have been obtained to date. These include 125 samples of the polysulfide synthetic rubber base, 2 silicone rubber, and 1 each of neoprene rubber, butene rubber and epoxy-resin formulation.

Description of test specimens, procedures, results, and discussion are given for the following properties: 1) staining effects and color changes, 2) slump, 3) hardness, 4) application life, 5) bond-ductility, 6) effect of outdoor exposure.

Of 74 samples (17 producers) tested, 93% stained mortar or marble or both. In the polysulfide group no stain appeared where mortar was absent. Fifty seven percent developed color changes varying from minor to extreme, such as light tan or dark brown. Of 65 non-sag type samples from the above group, 21% slumped 1/8 to 5/16 in. when tested in a trough 4- by 1- by 3/8-in. at 122°F. Nine percent slumped 3/8 in. or more. Shore A hardness values obtained on 53 samples from 17 producers ranged from 10 to 58 at 74°F, with an average of 34. The average hardness increased by 56% when tested at 0°F, and 8.8% at 158°F. Residual increase in the hardness after the 0°F treatment was 2.9% and after 158°F it was 29.5%.

Application life tests made with the Brookfield Viscometer indicated 41% of the 29 samples tested as relatively rapid curing compounds (less than 3 hr pot life). Bond-ductility tests made on 28 samples from 12 producers with 4 accessory materials (concrete, concrete block, brick, and wood) showed an exceptionally high degree of failure for primed brick. Results showed no significant differences between primed and unprimed concrete or block; there were no significant differences either between compounds of different colors.

Seventy four samples from 17 producers were exposed outdoors for nine months in primed and unprimed concrete joints. Joints were stretched every three months to a total of 33% of the original width. Thirty six samples developed at least one bond break including twelve which leaked under 1 in. head of water. Primed joints generally showed somewhat poorer performance than the unprimed joints. Bond failures in outdoor exposure tests showed good correlation with laboratory hardness tests and fair correlation with bond-ductility test results.

The investigation will be continued with emphasis on the performance and durability properties such as bond-ductility, hardness and effects of outdoor exposure. The width-depth ratio of the joint in the bond test will be increased to produce a lower incidence of bond failure for all samples, and new accessory materials such as aluminum, glass, steel, and ceramic coated metals will be added. A purchase specification will be written after enough dependable laboratory test data is compiled.

1. INTRODUCTION

At the request of the member agencies of the Tri-Service Program, the National Bureau of Standards is investigating the properties of rubber base calking materials for sealing joints in concrete, brick, stone and other types of masonry construction. The study will include the development of laboratory test methods which will be eventually incorporated in a purchase specification.

The new type of rubber base calking (also referred to as sealer, sealant and elastomer) differs from the "conventional" oleo-resinous base plastic compound in that it consists mainly of a synthetic rubber base liquid combined with suitable fillers and with little or no volatile component. The base material is mixed with an accelerator immediately before application. The application or pot life normally varies from 2 to 6 hr.

For the past ten years polysulfide synthetic rubber base compounds have been used by the aircraft industry for sealing integral fuel tanks. The compound proved effective by maintaining good adhesion to the aluminum frame-work and by remaining flexible over the temperature range of -65°F to + 180°F. It also has been used to seal wooden decks of aircraft carriers.

The successful use of this type of mastic in aircraft suggested its use as a joint sealer in masonry structures around windows, between panels in curtain wall and concrete construction, for sealing flashing and expansion joints, and in any joint where some movement is expected and watertightness is required. For the past few years the polysulfide synthetic rubber compounds have been used by private industry in curtain wall construction and to a lesser extent by Government agencies in sealing masonry structures.

One of the primary purposes of this study is to develop new laboratory test methods and, if feasible, apply certain existing test methods which will successfully predict the behavior of the joint sealers in actual service. Since it is expected that a rubber base joint sealer should function effectively for a minimum of ten years, the performance and durability tests are being designed with this goal in mind.

The properties investigated thus far and described in this progress report are as follows: 1) staining effects and color changes, 2) slump or flow, 3) hardness, 4) application life, 5) bond-ductility, 6) effect of outdoor exposures.

For this portion of the investigation the accessory building materials used in the various tests of the calkings were restricted to the porous types only such as concrete, concrete block, brick, and wood. White marble was used for staining and color change tests.

In view of the fact that some of the tests described herein are somewhat exploratory in nature it will be necessary to make changes in procedures, as well as add new ones as the investigation progresses.

2. DESCRIPTION OF SAMPLES

To date, 130 samples of rubber base calkings have been obtained from 19 producers. Of this group, 125 samples are of the polysulfide synthetic rubber base (Thiokol) type, 2 silicone rubber, 1 butene rubber, 1 neoprene rubber, and 1 epoxy-resin formulation.

At the start of the study an attempt was made to buy the samples directly from the producers since no retail outlets were available. About half of the total samples were obtained in this way. Later it was found more advantageous to both producer and the Bureau to have the samples submitted without charge. In this way samples were delivered more promptly than when purchased.

All samples, except the neoprene and butene base compounds, consisted of two separate components, generally referred to as base and accelerator, activator, or curing agent.

All samples were classified by the producers as either non-sag or flow; the latter type specified for horizontal joints only. Some producers submitted samples labeled "hard" or "soft" designating the relative hardness of the cured rubber.

The polysulfide samples (in the cured state) consisted of several colors including white, natural (or neutral), aluminum, gray, tan, brown, red, and black. Most producers had at least three colors available and black was sold by all producers of the polysulfide type. The two silicone rubber compounds were white and pink, respectively; the neoprene was aluminum colored, epoxy-resin, tan, and the butene rubber, black.

For report purposes each sample was given a laboratory designation in the form of a number and letter, the number specifying the producer and the letter, the compound. Some producers submitted as many as 16 compounds over a year's period, although many of them are duplicate formulations.

3. PHYSICAL TESTS

3.1 Staining and Color Changes

3.1.1 Test Specimens and Procedure

This test was made on 74 samples of calkings submitted by 17 producers. The sample distribution by type, class, and color, is given in Table 1.

Each test specimen consisted of a calked joint, approximately 1 1/2 to 2 in. long, 1/2 in. deep and 3/8 in. wide placed between two 3- by 2- by 1-in. white marble blocks (either Georgia or Vermont marble was used for this test). The calking was backed by a layer of white cement mortar and below the mortar was a 2- by 1/2- by 3/8-in. aluminum spacer cemented between the two blocks. (Figure 1).

In preparation of the test specimen, a workable mortar mixture made of white cement, hydrated lime and standard graded Ottawa sand (1:0.5:3, by volume) was placed in the bottom of the joint, above and in contact with the spacer. After allowing the mortar to set for 4 hr at 74°F the remaining top portion of the joint was filled with freshly mixed calking.

A duplicate test specimen of each sample was made, omitting the mortar backing. Ten to fourteen days after filling, the test specimens were fastened to an aluminum rack and placed in a vertical position on the cylinder wall of a standard type Atlas Weatherometer (machine described in ASTM Standard D529-39T, 1958, part 4). One half of the joint (calking and marble) was covered with a sheet of aluminum, the latter shielding that portion of the joint from the light rays. (Figures 2, A, B). During the test each specimen was exposed to 500 hr of light from the carbon arc and periodic water spray (water, 9 min in each hr). The air temperature at the specimen during the test was 140° ± 5°F and the water temperature maintained at 75 ± 2°F.

3.1.2 Test Results

Table 2 summarizes the results obtained on the 74 samples after 500 hr in the Weatherometer.

All stains formed on the specimens during the exposure, were shades of red, or brown or mixtures of the two colors, and were rated as slight, moderate or deep. (Figure 1, A, B). Sixty seven of the 72 polysulfide calking specimens developed stains in the mortar but only 43 of these specimens developed stains in the marble.

No stains appeared in the marble blocks containing polysulfide calking without mortar.

Of the two silicone rubber samples, one developed a deep oily stain in the marble with no stain in the mortar. The second sample did not stain either the marble or mortar.

Table 2 also summarizes the color change effects in the calkings after the 500 hr exposure test.

Of the 74 samples tested, 42 showed noticeable changes in color when the latter was compared with the original color of the covered half of the calking joint. (Figure 2, A). Eleven of the 42 compounds turned lighter and the remaining 31 turned darker including such changes as from light tan to dark brown, and from white to dark gray. After examination, the test specimens were placed on the roof of the Mineral Products Building for further observations.

3.2 Slump or Flow Test

3.2.1 Test Specimens and Procedure

This test was made on the same samples as those tested for staining effects and color changes (except the flow type compounds). A portion of the freshly mixed sample was placed in an aluminum trough 4-in. long, 1-in. deep and 3/8 in. wide. The trough was immediately suspended in a vertical position in an oven for 24 hr at 122°F ± 2°F. At the end of this period the amount of slump was measured from the lower end of the trough to the lowest point assumed by the compound. Measurements were made to the nearest 1/16 inch.

3.2.2 Test Results

Table 2 includes the results of the slump tests for each of the 65 samples tested. The distribution of the values obtained are shown in Table 3.

Of the six samples with slump ratings over 3/8 in., three were regarded as "complete" failures. In such cases portions of the calking materials dropped out of the trough during the test.

3.3 Hardness

3.3.1 Test Specimens and Procedure

This test was made on 53 samples obtained from 17 producers. The instrument used for making the determinations was the Shore Durometer (Model A) described in Federal Test Method for Rubber No. 601, Method 3021, and also in ASTM Standard D676-55T, 1958, part 9. All readings were taken by the instantaneous method.

The test procedure was as follows: A portion of a freshly mixed compound was placed into a polyethylene mold with inside dimensions of 4- by 1 1/2- by 1/4-in. After striking off the top surface with a spatula, the specimen was stored in the laboratory at 74°F. (At least two specimens were made for each sample). When the compound lost its tackiness (usually two or three days), it was removed from the mold, placed on a 3- by 6-in. thin aluminum plate and allowed to cure for a total of 10 to 14 days. (Figure 4, A).

After the curing period, Shore hardness values were obtained on two specimens of each sample, at 74°F. An average of eight readings were taken on the two specimens, and this value was regarded as the "original value" listed in Table 4. One specimen was then exposed in an oven at 158°F for 72 hr and the other in a freezer at 0°F for the same period. At the end of the cold and hot exposure periods hardness values were determined at the respective temperatures and also after a recovery period for 3 hr at 74°F.

3.3.2 Test Results

Table 4 gives the hardness values obtained on all samples at 74°F, 0°F, and 158°F. The recovery values and respective percentage changes from the original values after the cold and hot exposures are also given. Figures 5 and 6 illustrate the distribution of hardness values for the three temperatures for all samples.

It follows from the data given in figure 5 that the average hardness value at 0°F was 56% higher than the average at 74°F. However, the residual increase in hardness after recovery to 74°F was only 2.9%.

On the other hand, the data in figure 6 show that the hot treatment (158°F) caused an immediate increase in hardness of only 8.8% over the original but was followed by a residual hardness, after recovery, of 29.5%.

The samples of group 6 had a hardness value of 20 which was the lowest original value. Except for the epoxy-resin sample 18 A, with a hardness of 53, the samples of groups 9 and 10 had the highest original values of 50.

3.4 Application or Pot Life

3.4.1 Test Specimens and Procedure

This property was determined on 29 samples of calkings (non-sag type) submitted by 9 producers. A Brookfield Viscometer, model RVF fitted with a No. 7 spindle, rotating at 2 rpm was used to make the determinations. (Figure 7). The test procedure was as follows:

After mixing thoroughly about 300 g of base and accelerator of the sample under test, the compound was placed into a gill capacity can (with friction rim removed), slightly overfilling the can. The calking was struck off flat with the edge of a spatula. The spindle was slowly brought down (by means of rack and pinion arrangement) into the compound to the level of the standard immersion mark indicated on the spindle. Any voids immediately around the spindle were filled in with the aid of a small spatula. The viscometer motor was turned on and readings on the scale (0 to 100) were taken every minute for the first 5 min and at 5, 10, or 20 min intervals thereafter. The motor was stopped either at the end of 3 hr, or when the pointer reached the 100 mark depending on which came first. (No attempt was made to translate the readings into absolute viscosity values).

3.4.2 Test Results

For 12 of the 29 samples tested the pointer reached the scale limit of 100 before the 3 hr time limit, indicating relatively rapid curing compounds. For the remaining 17 samples, the test ran the full three hours.

At this stage of the investigation it seemed desirable to have a single numerical value (or factor) which could be used as a measure of the application life of each sample, using the data obtained with the viscometer. For this purpose an index, P, was calculated as follows:

$$P = \frac{3V}{t}$$

where, P = application life factor

V = scale reading at end of test (either 100, or specific reading at 3 hr)

t = duration of test in hours (3 hr or less).

The formula indicates that the factor P of a sample is less than 100 when the pointer fails to reach 100 within 3 hours. In such a case t = 3, and P = V.

The factor P is greater than 100 when the pointer reaches 100 in less than 3 hours. In such case $v = \frac{300}{t}$.

The smaller the P factor the longer is the application life.

Table 5 gives the minimum and maximum viscometer scale readings, duration of test in hours and the P factor for the 29 samples tested. The P factor ranged from 30 to 1500 with an average of 194 for all samples. The results indicate that one producer had consistently higher than average P factors such as No. 3-E, F, G, H, and L. All samples of producers 1, 2, 11, 14, 15, and 18 had P factors smaller than average. Values for sample group 6 ranged from 49 to 428 and values for group 12 ranged from 63 to 196.

Figures 8, 19, 10, and 11 show graphically the relationship between viscometer readings and time over a 3 hr period for 25 samples from 9 producers. The curves indicate that in 11 tests, viscometer readings reached the limit of the scale in less than 3 hours. These tests included 5 samples with test duration of less than 1 hour.

3.5 Bond-Ductility

3.5.1 Test Specimens and Procedure

This test, designed to evaluate the calking compounds for use in joints where movement is expected, was made on 28 compounds submitted by 12 producers. Twenty six samples were polysulfide rubber base, 1 silicone rubber, and 1 epoxy resin formulation.

A joint sealer stretching machine accommodating three test specimens was used to make the tests. The machine, with some changes in design, is similar to the one described in Fed. Spec. SS-R-406C, Road and Paving Materials, Method 223.11. (Figures 12, 13).

The test specimens, shown in figure 14-A, were prepared with calking, accessory masonry materials and aluminum spacers. The accessory materials used thus far in this study were brick, concrete, concrete block and wood with the following descriptions:

			24 hr absorption,%
Concrete	-	Prepared in accordance with Fed. Spec. SS-R-406,Method 223.11	6.6
Brick	-	Sanford Center Tunnel Kiln, Smooth	8.0
Concrete block	-	Expanded slag, high pressure steam cured	9.0
Wood	-	Douglas fir 1 in. plywood	no test

The accessory blocks, except concrete, were cut to the standard size of 3- by 2- by 1-in. The concrete blocks were fabricated in brass gang molds.

The test joint was prepared by first making a "sandwich" of two similar blocks separated by two aluminum spacers and held together by rubber bands or a clamp. Freshly mixed calking was carefully placed between the spacers forming a filled joint 2- by 1- by 3/8 in. After a curing period of at least 14 days the spacers were separated from the calking but left in place between the blocks until they were tested.

The tests were made in triplicate with accessory materials both primed and unprimed, except in the case of the wood which was tested primed only. The primers used were those submitted by the producers. If the producers submitted no primer for his samples, another producer's primer was used. A total of 21 test specimens were prepared for each sample.

The bond-ductility tests were combined with a heating, soaking and freezing cycle in the following sequence:

(a) Test specimens were heated for 16 to 24 hr at 158°F (with the aluminum spacers between the blocks).

(b) Cooled to room temperature (74°F).

(c) Soaked in distilled water for 3 to 5 hr.

(d) The saturated specimens frozen at 0°F for a minimum of 24 hr.

(e) The joint extended 50% of its original width at the rate of 1/8 in. per hr at 0°F.

(f) The joint held in the stretched position with 9/16 in. spacers for 16 to 24 hr at 0°F, followed by 4 hr at 74°F.

(g) Test specimens placed with original 3/8 in. spacers in oven and the cycle repeated until a total of 5 cycles were completed.

3.5.2 Rating of Failure in Bond-Ductility Test

After step (f) in the cycle was completed, the 3 test specimens (with spacers removed) were examined for bond or cohesion breaks and an estimate made of the loss in bonded area, in square inches, for each of the 3 specimens. The loss in bonded area of each specimen was estimated as the sum of the losses on both sides of a given joint.

Almost all of the failures occurred as bond breaks. A complete bond failure in a specimen was defined as a failure resulting in area loss of 2 sq in., regardless of whether this figure represented the loss on a single side or the combined loss on both sides of a joint. When two or three of the specimens showed complete bond failure (total loss of 4 or 6 sq in., respectively) the test was stopped regardless of the number of cycles completed. In all other cases, the tests were continued through five cycles.

The following formula for rating bond (or cohesion) failure was used which evaluated area of bond loss as well as the number of cycles completed:

Bond Loss Factor (L1) = A (6-C)

where, A = bond loss for each specimen in sq in.

C = number of cycles completed

For example, when the three specimens showed complete bond failure after the first cycle, the Bond Loss Factor was calculated as follows:

L1 = A (6-C) = 2 (6-1) = 10

For three specimens, L = 3 x 10 = 30

In a second example where complete bond failure occurred on all three specimens after the fourth cycle, the Bond Loss Factor was calculated as follows:

L1 = A (6-C) = 2 (6-4) = 4

For three specimens, L = 4 x 3 = 12

3.5.3 Test Results

Table 6 gives the bond loss factors obtained on 27 samples submitted by 12 producers.

The results indicate that the specimens fabricated with primed brick gave the highest bond losses (average 26.1) among all samples tested. This is in contrast to the average bond loss factor of 12.0 obtained on the unprimed brick specimens.

Priming of the concrete and concrete block specimens apparently did not cause any significant changes in bond failure, compared to the unprimed specimens.

The average bond loss factors (exclusive of the unprimed brick) for all specimens and all accessory materials ranged from 10.7 (primed wood) to 13.7 (unprimed concrete).

Table 7 lists the bond loss factors in relation to various colors of the compounds (white not included). The differences obtained among the four color groups do not appear significant at this stage of the investigation.

Table 8 lists the bond loss factors in relation to producer groups. The results show an exceptionally low average bond loss factor for the samples of group 6. However, it should be noted (table 6) that there were numerous air bubbles present in the calking of the concrete specimens of this group which were not included in the calculation of the bond loss factors.

3.6 Outdoor Exposure Tests of Calkings in Concrete Joints

3.6.1 Test Specimens and Procedure

This test was devised to obtain information on performance and possibly on the durability properties of the calkings under natural weather conditions.

For this purpose reinforced concrete troughs were fabricated, each 14 in. long, 8 in. wide and 1 3/4 in. deep and containing two joints, each 11 in. long 3/4 in. deep and 3/8 in. wide. (Figure 15, A). The troughs were so designed that 1 in. head of water could be placed over the filled joints to test them for leakage.

After the two joints were thoroughly cleaned with a stiff fiber brush and methyl ethyl ketone, one joint was given a coat of primer and the latter allowed to dry. Freshly mixed calking was then placed into the joints, the surface struck off with a putty knife and the compound left to cure for at least 14 days in the laboratory. Following the curing period, saw cuts 3/32 in. wide were made in the concrete at the ends of the joints making it possible to place aluminum wedges in the saw cuts which stretched the joints to any desired width. In this manner each of the two joints was stretched 1/32 in. and then placed on the coping of the roof of the Mineral Products Building and exposed to the weather for nine months (Figure 15, B).

During the nine months exposure period each joint was extended 1/32 in. every three months following the initial extension of 1/32 in. This made a total extension of 1/8 in. or 33% of the original width. At the end of nine months, the total length of bond breaks (or cohesion) regardless of depth, was measured and each joint tested for leakage.

3.6.2 Outdoor Exposure Test Results

Table 9 gives the results of observations and tests made on 74 samples of calkings exposed to the weather for nine months.

Figures 16 to 20 illustrate some of the bond and cohesion failures obtained in both primed and unprimed joints after six months exposure.

As shown in Table 9, 49% of the samples developed at least one break in either the primed or unprimed joint. Almost all breaks were in bond. Of this group 12 samples developed leakage through either one or both joints when tested with 1 in. head of water.

The results in Table 9 also show a total of 135 in. of bond breaks in the primed joints as compared with 93 in. in the unprimed joints.

3.7 Discussion of Results

3.7.1 Staining and Color Changes

It is obvious from the test results that staining of mortar and light colored masonry in contact with mortar is characteristic of most polysulfide rubber base (Thiokol) calkings. It is not known if the staining affects in any way the performance or durability of the sealer itself. However, it is an established fact that extensive stains, caused by calking, can seriously mar the appearance of a marble, limestone or concrete facade of a building. Such examples are the United Nations Building in New York City and the Arlington Amphitheater in Virginia, both badly stained by polysulfide rubber base calkings in the joints. Several other instances of staining caused by calkings have come to the attention of the National Bureau of Standards.

To date no method has been found to remove such stains from the masonry. Some observations made at NBS on stained marble test specimens indicate that the stains tend to lighten, and in some cases disappear entirely after the specimens have been exposed to the sun's rays over a period of six months.

It has also been observed in a limited number of tests that a coat of primer applied to the surfaces of a joint, including the mortar backing, prevents staining. However, the primer itself if applied carelessly can cause a yellowish stain on the masonry.

The "Rubber and Plastic Adhesive and Sealant Manufacturers Council" in a proposed specification for rubber base sealers, suggests a staining test that is simple and effective. Briefly, it consists of placing a mortar mixture in the form of a 3 in. diameter ring (doughnut shape) on a glass plate and a pat of freshly mixed calking over and in contact with

the mortar. After being suspended over water in a closed desiccator for 14 days, the test specimen is examined for stains. No marble or other accessory material is required for the test.

All staining tests made at the National Bureau of Standards with polysulfide calkings have shown that no stain occurs when there is no cement mortar in contact with the calking.

Although the test results indicated some extreme color changes after 500 hr in the Weatherometer this effect is not as serious as staining. In installations where white or neutral calkings are required, a test for color change should be a requirement in a specification.

3.7.2 Slump

Slump or flow in a rubber base calking is an important handling property of the material. On the job, a slumping compound, applied to a vertical joint can cause poor workmanship, unsightly appearance and delay in installation.

The slump test results reported herein are significant only for the specific size of trough, period of exposure, and the temperature used in these tests.

In future studies troughs of other dimensions will be used such as 6- by 3/4- by 3/8 in., and 6- by 1/2- by 1/2 in. before a standard test is developed.

3.7.3 Hardness

The main objective of the study of the hardness properties of the rubber base compounds is to accumulate enough data on this property which would form the basis for a laboratory durability test.

Experience has shown that hardness is of relatively minor importance in a calking installed in a joint in which there is little or no movement. This has been demonstrated by the behavior of various fillers installed in joints of an experimental concrete panel structure on the NBS grounds. After two years exposure to the weather, the joints that were filled with cement mortar prevented leakage of rain water as effectively as the rubber base calkings.

However, in installations where large movements are expected the stresses developed in the bond between calking and masonry may be directly related to the hardness (or modulus of elasticity) of the joint filler.

The test results obtained thus far indicate (with some exceptions) that relatively high hardness values tend to cause bond failures. Sample groups 9 and 10 which had the highest average hardness values (except sample 18-A) also had the highest average bond loss factors. Furthermore, the seven samples of groups 9 and 10 developed bond breaks in the exposure test totaling 44.5 and 53.5 in., respectively.

In the case of the epoxy-resin sample, 18-A, the high hardness value of 82 at 0°F, caused failure in the bond test, by breaking the accessory blocks when the joints were stretched.

Nevertheless, all bond failures were not caused by excessive hardness of the calking. This is shown by sample 14-A which developed complete bond failure on both primed and unprimed specimens after six months exposure in concrete joints. The sample had an initial hardness value of 36.

The study of hardness properties will be continued in the investigation. An attempt will be made to develop an accelerated test which can predict the extent of hardening of a calking with time. The use of heating and freezing cycles as well as exposures in the Weatherometer will be tried. These test results will be correlated with the results obtained on specimens exposed to the weather.

3.7.4 Application Life

The application or pot life of the two part rubber base calkings may be regarded as the most important of the handling properties. It can sometimes be the single factor causing failure in a calking installation when an attempt is made to force a rapidly curing compound into a joint.

The use of the Brookfield Viscometer proved to be an accurate laboratory method for determining the rates of curing of the calkings over a 3 hr period at constant temperature. Unfortunately the tests were made without control of relative humidity. However, a repeat test made on a sample at two different relative humidities of 20 and 40% did not produce significant differences in the results.

Another method for measuring this property, as suggested by the Rubber and Plastic Adhesive and Sealant Manufacturers Council, involves the use of a standard "Semco" cartridge or equivalent, operated at 50 psi. The maximum application life is reached when the time required to discharge 6 fluid oz exceeds 1 min for flow type compounds and 2 min for the non-sag type.

In continuing the study of application life, some tests will be made by the method described above, and the results compared with those obtained with the Viscometer.

3.7.5 Bond-Ductility

The results in Table 6 give the impression that most of the calking samples tested would be ineffective sealers in calking installations where the joints were subjected to large movements. This high incidence of failure shown in the bond-ductility test might be due to the relatively deep joint used in the test, or more specifically, the low width-depth ratio of the calking, .38, in this case.

W. H. Kuenning[1/] in his study of joint sealers for concrete pavements also found a high degree of bond and cohesion failures of rubber base calkings tested with low width-depth ratios. The number of failures in the tests were reduced by lessening the depth of the calking in the joint (in effect, increasing the width-depth ratio).

Studies of bond-ductility will be continued in this investigation. The test joints will be made up in various width-depth ratios ranging from 0.5 to 1.0 to determine the optimum width-depth ratio.

Heretofore, the bond-ductility test procedure included stretching of the joint with recovery to its original width. In future tests, it is planned to study the effect of compression of the calking along with extension.

The effect of accessory materials such as aluminum, steel, glass and ceramic coated materials on bond will also be studied.

3.7.6 Outdoor Exposure Tests

The outdoor exposure tests afford an excellent opportunity to observe the over-all performance of the calkings in concrete joints under natural weather conditions. For this reason, the exposure tests will be continued on the 74 samples reported and on as many new samples as possible.

As in the bond-ductility tests, the results of the exposure test indicated that priming of the concrete joints, generally, did not improve the performance of the calkings, although several exceptions are noted in Table 9.

Of the 74 samples exposed to the weather only 14 were included in the bond-ductility tests. An attempt was made to correlate the bond loss factors (concrete) of these 14 samples with the length of bond breaks occurring after nine months exposure in the concrete joints. The results obtained for the 14 samples are given in Table 10.

1/ Kuenning, William H., Laboratory Tests of Sealers for Sawed Joints, Highway Research Board, Bulletin 211, (1959).

As indicated in Table 10, eight of the fourteen samples show good correlation between the two tests. It is possible that by increasing the width-depth ratio in future bond-ductility tests, better correlation between the bond loss factors and the bond losses in the outdoor exposure tests will be obtained. In the tests reported, the width-depth ratio of the joints in the exposure test was .50 as compared to .38 in the bond-ductility test.

As mentioned in section 3.7.3 in the discussion of hardness results, the two sample groups 9 and 10 with the highest average hardness values developed exceptionally large bond breaks in the exposure tests. The study of the relationship between outdoor exposure effects and hardness will be continued on the old as well as new samples.

Table 1. Distribution of samples in staining, color change and slump tests.

Type	Number of samples tested			
	Total	Non-sag	Flow	Color
Polysulfide Rubber	72	66	8	Black - 22 Gray - 16 Alum. - 15 Tan - 12 White - 4 Brown - 2 Red - 1
Silicone Rubber	2	2	none	White - 1 Pink - 1

Table 2. Staining, color change and slump properties of 74 joint sealers.[1]

Sample No.[2]	Color (mix)	After 500 hr in Weatherometer			Slump rating 122°
		Stain [3]		Calking color change	
		Mortar	Marble		
					in.
1-A	Light tan	Moderate	Moderate	Lighter	1/8
1-B	Aluminum	do	do	do	1/8
1-C	Brown	do	do	Darker	1/8
1-D	Gray	do	do	Lighter	1/4
1-E	Black	do	do	None	1/4
2-A	Aluminum	Moderate	Moderate	Lighter	None
2-B	Gray	do	do	do	do
2-C	Tan	do	do	Darker	do
2-D	Black	do	Slight	None	1/8
3-A	Aluminum	Moderate	Moderate	None	None
3-B	Gray	do	do	Lighter	1/8
3-C	Tan	do	do	Darker	None
3-D	Black	do	do	None	do
4-A	Gray	Moderate	Moderate	Lighter	1/16
4-B	Gray[4]	do	do	Brownish gray	1/16
4-C	Aluminum	do	Slight	None	1/8
4-D	White	Slight	do	Tan	None
4-E	Black	do	None	None	3/16
4-F	Red	do	Slight	Darker	1/16
5-A	Tan	Moderate	Moderate	Brown	None
5-B	Gray	do	None	Brownish gray	do
5-C	Aluminum	do	do	Darker	do
5-D	Black	do	do	None	do
6-A	Light tan	None	None	Brown	None
6-B	Light tan[5]	do	do	Dark brown	No test
6-C	Gray	Slight	Slight	None	None
6-D	Gray [5]	None	None	Darker	No test
6-E	Aluminum	Slight	do	None	1/16
6-F	Black	None	do	do	None
6-G	Black[5]	Slight	do	do	No test

Table 2. Staining, color change and slump properties of 74 joint sealers.[1] (Continued)

Sample No.[2]	Color (mix)	After 500 hr in Weatherometer: Stain [3]: Mortar	Stain [3]: Marble	Calking color change	Slump rating 122°
					in.
7-A	Black	Moderate	Slight	None	None
8-A	Aluminum[4]	Moderate	Moderate	None	1/16
8-B	Aluminum[6]	do	Slight	do	None
8-C	Brown [6]	do	Moderate	Lighter	do
8-D	Black [6]	do	do	None	do
8-E	Black [4]	Slight	None	None	1/16
9-A	Aluminum	Slight	None	Brownish gray	None
9-B	Tan	Moderate	Slight	Dark brown	1/16
9-C	Black	do	do	None	None
9-D	Aluminum[5]	do	Deep	Brown	No test
9-E	Tan [5]	do	Moderate	Dark brown	No test
9-F	Black [5]	do	Slight	None	No test
10-A	Gray	Moderate	None	Grayish tan	None
10-B	Gray	Slight	do	Dark gray	do
10-C	Aluminum[6]	Moderate	do	None	Complete
10-D	Black	Slight	Slight	do	1/8
10-E	Black [5]	Moderate	Moderate	do	No test
10-F	Gray	Slight	None	Dark gray	None
11-A	Gray	Slight	None	Grayish brown	None
11-B	Gray[5]	Moderate	do	Darker	No test
11-C	Tan	Slight	do	Dark brown	None
11-D	Black	do	do	None	do
12-A	Gray	Slight	Slight	Darker	None
12-B	Tan	do	None	Dark brown	do
12-C	Black	Moderate	do	None	1/16
12-D	Black[5]	do	Slight	do	No test
13-A	Tan	Moderate	None	Dark brown	None
13-B	Gray	Moderate	Slight	Darker	1/16
13-C	White	None	None	Dark gray	None
13-D	Aluminum	Moderate	Moderate	Lighter	1/16
13-E	Light tan	Moderate	None	Darker	3/8
13-F	Black	Slight	Deep	None	3/8
13-G	White	Slight	None	Dark gray	None

Table 2. Staining, color change and slump properties of 74 joint sealers.[1/] (Continued)

Sample No. [2/]	Color (mix)	After 500 hr in Weatherometer			Slump rating 122°
		Stain [3/]		Calking color change	
		Mortar	Marble		
					in.
14-A	White	None	None	Buff	None
14-B	Gray	Moderate	Slight	Brownish gray	None
14-C	Black	Slight	None	None	None
15-A	Aluminum	Slight	None	Lighter	Complete
15-B	Aluminum	Moderate	Moderate	do	3/16
15-C	Black	do	do	None	3/8
15-D	Black	do	do	do	Complete
16-A	Aluminum	Moderate	Moderate	None	None
16-B	Black	do	do	do	do
17-A	Pink[7/]	None	Deep (oily)	None	1/8
17-B	White[7/]	do	None	do	None

1/ Samples are polysulfide rubber base compounds, non-sag type, unless specified in a footnote.
2/ NBS sample designation: the number represents a producer, and the letter, an individual sample.
3/ Color of stain is shade of red or brown or mixture of both unless specified in a footnote.
4/ Designated by producer as "hard" type.
5/ Flow type compound. No slump test made on these samples.
6/ Designated by producer as "soft" type.
7/ Silicone rubber compound.

Table 3. Distribution of slump ratings.
(slump in inches)

	None or < 1/16	1/16	1/8	3/16	1/4	5/16	3/8 or more
No. of samples	37	10	8	2	2	2	6

Table 4. Shore "A" Hardness Values

Sample No.	Specimen 1 (cold exposure)					Specimen (hot exposure)				
	Orig. value	Exposure, 3 days		Recovery		Orig. value	Exposure, 3 days		Recovery	
	74°F	0°F	Change %	To 74°F	Change %	74°F	158°F	Change %	To 74°F	Change %
1-F	32	51	+59	40	+25	32	32	0	40	+25
1-G	32	52	+63	36	+12	32	32	0	44	+38
1-H	34	59	+73	36	+ 6	34	34	0	42	+24
Avg.	33	54	+65	37	+14	33	33	0	42	+29
2-E	42	55	+31	41	- 2	42	42	0	47	+12
2-F	38	50	+32	40	+ 5	38	32	-19	39	+ 3
2-G	34	51	+50	35	+ 3	34	38	+12	43	+26
Avg.	38	52	+38	39	+ 2	38	37	- 2	43	+14
3-E	32	49	+53	32	0	32	24	-25	41	+28
3-F	33	53	+61	34	+ 3	33	35	+ 6	40	+21
3-G	46	56	+22	41	-11	46	44	- 4	43	- 7
3-H	33	51	+55	34	+ 3	33	41	+24	46	+39
3-I	30	61	+103	33	+10	30	38	+27	48	+60
3-J	38	56	+ 47	36	- 5	38	39	+ 3	46	+21
3-K	31	56	+81	34	+10	31	36	+16	46	+48
3-L	25	48	+92	26	+ 4	25	29	+16	42	+68
Avg.	34	54	+64	34	+ 2	34	36	+ 8	44	+35
4-G	39	64	+64	46	+18	39	40	+ 3	51	+31
4-H	22	56	+155	35	+59	22	39	+77	46	+109
Avg.	30	60	+110	41	+39	31	40	+40	49	+ 70
6-A	15	40	+167	16	+ 7	15	7	-53	16	+ 7
6-H	20	37	+ 85	20	0	20	15	-25	24	+20
6-J	16	34	+112	22	+38	16	12	-25	16	0
6-K	16	43	+169	19	+19	16	12	-25	20	+25
6-L	16	53	+231	18	+12	16	15	-6	22	+37
6-M	35	58	+ 66	34	- 3	35	27	-23	37	+ 6
Avg.	20	44	+138	22	+12	20	15	-26	23	+16

Table 4. Shore "A" Hardness Values (Continued)

Sample No.	Specimen 1 (cold exposure) Orig. value 74°	Exposure, 3 days 0°F	Exposure, 3 days Change %	Recovery To 74°F	Recovery Change %	Specimen (hot exposure) Orig. value 74°F	Exposure, 3 days 158°F	Exposure, 3 days Change %	Recovery To 74°F	Recovery Change %
7-A	49	60	+ 22	48	- 2	49	48	- 2	56	+14
8-A	10	37	+270	10	0	10	12	+20	24	+140
8-B	27	52	+ 92	29	+ 7	27	34	+26	48	+ 78
8-D	16	44	+175	13	-19	16	15	- 6	21	+ 31
8-E	47	61	+ 30	47	0	47	53	+13	54	+ 15
Avg.	25	49	+142	25	- 3	25	29	+13	37	+ 66
9-A	49	59	+ 20	52	+ 6	49	50	+ 2	53	+ 8
9-B	45	56	+ 25	44	- 2	45	45	0	50	+ 11
9-C	50	60	+ 20	49	- 2	50	52	+ 4	54	+ 8
9-F	55	59	+ 7	52	- 5	55	56	+ 2	58	+ 5
Avg.	50	59	+ 18	49	- 1	50	51	+ 2	54	+ 8
10-A	36	57	+ 58	36	0	36	36	0	42	+ 17
10-B	58	67	+ 16	57	- 2	58	58	0	62	+ 7
10-D	57	63	+ 10	64	+12	57	60	+ 5	60	+ 5
Avg.	50	62	+ 28	32	+ 3	50	51	+ 2	55	+ 10
11-A	33	54	+ 64	34	+ 3	33	47	+42	53	+ 61
11-E	40	58	+ 45	37	- 8	40	46	+15	51	+ 28
11-F	17	46	+171	16	- 6	17	32	+88	40	+135
11-G	41	57	+ 39	41	0	41	43	+ 5	52	+ 27
11-H	44	60	+ 36	46	+ 5	44	50	+14	58	+ 32
Avg.	35	55	+ 71	35	- 1	35	44	+33	51	+ 57
12-E	41	54	+ 32	42	+ 2	41	44	+ 7	50	+ 22
12-F	39	56	+ 44	37	- 5	39	40	+ 3	47	+ 20
12-G	28	52	+ 86	32	+14	28	35	+25	46	+ 64
12-H	45	60	+ 33	46	+ 2	45	48	+ 7	57	+ 27
Avg.	38	56	+ 49	39	+ 3	38	42	+11	50	+ 33

Table 4. Shore "A" Hardness Values (Continued)

Sample No.	Specimen 1 (cold exposure)					Specimen (hot exposure)				
	Orig. value 74°F	Exposure, 3 days		Recovery		Orig. value 74°F	Exposure, 3 days		Recovery	
		0°F	Change %	To 74°F	Change %		158°F	Change %	To 74°F	Change %
13-I	24	54	+125	26	+ 8	24	30	+25	43	+79
13-J	21	42	+100	20	- 5	21	28	+33	36	+71
13-K	24	44	+ 83	26	+ 8	24	33	+37	43	+79
Avg.	23	47	103	24	+ 3	23	30	+32	41	+76
14-A	36	56	+ 56	36	0	36	31	-14	41	+14
15-B	46	57	+ 24	46	0	46	48	+ 4	51	+11
15-E	41	54	+ 32	52	+27	41	46	+12	40	- 2
15-F	16	42	+162	15	- 6	16	26	+62	36	+125
Avg.	34	51	+ 73	38	+ 7	34	40	+26	40	+45
17-C	40	40	0	40	0	40	44	+10	42	+ 5
18-A	53	92	+ 74	55	+ 4	53	64	+21	82	+55
19-A	20	26	+ 30	25	+25	20	21	+ 5	35	+75

Table 5. Results of application life tests with the Brookfield Viscometer.

Sample No.	Viscometer Readings (0-100 scale)		Time (t) (hr)	"P" factor
	Minimum	Maximum		
1-F	49.0	56.5	3	56.0
1-G	55.4	81.0	3	81.0
1-H	80.7	92.7	3	93.0
2-E	48.0	98.2	3	98.0
2-F	41.4	100	2.75	109
2-G	53.0	100	2.33	129
3-E	46.0	100	0.97	212
3-F	70.0	100	.57	525
3-G	50.7	100	.47	638
3-H	69.8	100	.20	1500
3-L	77.7	100	.25	1200
6-H	51.5	100	2.50	120
6-I	37.0	49.2	3	49
6-J	60.6	100	.70	428
6-K	45.5	100	1.33	226
11-E	63.0	82.8	3	83
11-F	58.0	61.9	3	62
11-G	53.0	61.0	3	61
11-H	67.5	100	2.08	144
12-E	55.3	62.8	3	63
12-F	57.0	70.5	3	71
12-G	54.8	60.5	3	61
12-H	23.3	76.2	1.53	196
14-H	51.7	59.0	3	59
14-I	34.9	51.3	3	51
14-J	52.7	60.4	3	60
15-E	24.7	29.5	3	30
15-F	46.5	84.4	3	84
18-A	30.5	50.5	3	51

Table 6. Results of Bond-Ductility Tests

Sample	Color	Bond-Loss Factor						
		Concrete		Brick		Block		Wood
		Primed	Unprimed	Primed	Unprimed	Primed	Unprimed	Primed
1-F	Light tan	4.5	4.0	20.9	.88	.65	0	0
1-G	Aluminum	26.0	30	23.1	30.	27.0	no test	15.0
1-H	Black	14.3	12.7	30.0	.68	0.06	0.15	0.06
Avg.		14.9	15.6	24.7	10.5	9.2	.1	5.0
2-C	Tan	26.0	20.0	30.0	19.5	no test	no test	no test
2-E	Aluminum	30.0	11.5	30.0	16.8	30.0	11.4	30.0
2-F	Tan	14.2	3.1	30.0	0.48	3.5	1.5	0.73
2-G	Black	25.0	6.5	30.0	14.0	5.2	1.4	7.5
Avg.		23.8	10.3	30.0	12.7	12.9	4.8	12.7
3-A	Aluminum	11.2 1/	13.2 1/	30.0	10.0 1/	no test	no test	15.0 1/
3-D	Black	0.18	no test	30.0	0.38	0.38	no test	no test
Avg.		5.7	13.2	30.0	5.2	.38		15.0
6-H	Light tan	0.26	0.15 2/	30.0 2/	0.36	0	0.06	0.03
6-I	Aluminum	0.14	0.24	30.0	0.86	0.46	0.06	0.24
6-J	Gray	1.2 2/	0.03 2/	30.0	0.15	0.09	0.28	0.24
6-K	Black	.06 2/	0.05 2/	30.0	1.0	0.02	0.27	0.09
Avg.		.4	.1	30.0	.6	.1	.2	.15
7-A	Black	18.4	30.0	30.0	30.0	18.0	30.0	22.5
8-A	Aluminum	0 2/	8.0 1,2/	0	0.25 1,2/	0	no test	0
8-B	Aluminum	18.0 1/	14.1 1/	8.8 1/	3.1 1/	2.5 1/	2.1 1/	2.2 1/
8-D	Black	0 2/	0 2/	0 2/	0 2/	0 2/	0 2/	0 2/
8-E	Black	2.9 1/	30.0	30.0	30.0	20.8	30.0	30.0
Avg.		5.2	13.0	9.7	8.3	5.8	8.0	8.1

Table 6. Results of Bond-Ductility Tests (Continued)

Sample	Color	Bond-Loss Factor						
		Concrete		Brick		Block		Wood
		Primed	Unprimed	Primed	Unprimed	Primed	Unprimed	Primed
9-A	Aluminum	21.2	30.0	30.0	30.0	10.0	20.0	30.0
9-E	Tan	1.1	16.9	30.0	30.0	30.0	22.5	Broke block
9-F	Black	30.0	27.5	30.0	1.2	30.0	15.8	30.0
Avg.		17.4	24.8	30.0	20.4	23.3	19.4	30.0
10-A	Gray	18.0	2.6	21.0	1.5	0	1.6	1.8
10-B	Light gray	22.5	30.0	30.0	30.0	30.0	30.0	no test
10-D	Black	30.0	25.0	30.0	30.0	30.0	30.0	30.0
Avg.		23.5	19.2	27.0	20.5	20.0	20.5	10.6
11-A	Gray	1.2	3.6	30.0	1.8	2.6	2.8	18.0
14-A	White	1.6	22.8	30.0	30.0	2.6	30.0	.72
17-B	White	13.5 1/	no test	30.0	no test	no test	no test	12.0 1/
18-A	Tan	no test	broke block	no test	broke block	no test	broke block	no test
Grand Averages		12.3	13.7	26.1	12.0	10.2	11.0	10.7

1/ Bond and cohesion failure.
2/ Air bubbles in calking.

Table 7. Bond loss factor in relation to color
(Polysulfide type compounds)

Color	No. of samples	Concrete		Brick		Block		Wood	Averages
		Primed	Unprimed	Primed	Unprimed	Primed	Un-primed	Primed	
Aluminum	7	15.3	15.3	21.7	13.0	11.7	8.4	13.2	14.1
Tan	5	9.2	8.0	28.2	10.2	8.5	6.0	0.3	10.0
Gray	4	10.7	9.1	27.8	8.4	8.2	8.7	6.7	11.4
Black	9	13.4	16.5	26.7	11.9	11.6	13.5	15.0	15.5

Table 8. Bond loss factors in relation to producer groups

Producer Group	Number of samples tested 1/	Bond Loss Factor (average)
1	3	11.4
2	4	15.3
3	2	10.9
6	4	4.7 2/
7	1	25.6
8	4	8.3
9	3	23.6
10	3	20.2
11	1	8.6
14	1	16.8
17	1	18.5

1/ Includes tests with different accessory materials: concrete, brick, block and wood.

2/ Many air bubbles were observed in the calking of the concrete specimens. These were not included in the calculation of bond loss factor.

Table 9. Results of 9 months' exposure to weather of 74 calkings in concrete joints.[1]

Sample No.	Total loss in bond to concrete (inches)[2]			
	Primed joint		Unprimed joint	
	Top	Bottom	Top	Bottom
1-A	None	None	None	3/16
1-B	None	1	None	None
1-C	None	None	None	None
1-D	None	None	None	None
1-E	None	None	None	None
2-A	None	None	None	None
2-B	1/4	None	None	None
2-C	None	None	None	None
2-D	None	3/4	None	None
3-A	3	1/2	None	None
3-B	None	None	None	None
3-C	None	None	None	None
3-D	None	None	None	None
4-A	None	None	None	None
4-B	None	None	None	None
4-C	5/8	None	None	None
4-D	1/4	6	5 [5]	5 [5]
4-E	None	None	None	None
4-F	None[9]	None	None [9]	None
5-A	None	None	None	None
5-B	1/2	None	1/4 [3]	None
5-C	None	1 1/2	None	None
5-D	None	None	None	None
6-A	None	None	None	None
6-B [4]	3 [5]	5 [5]	None	None
6-C	3 [5]	4 [5]	None	None
6-D [4]	None[6]	None[6]	None	None
6-E	None	1/4	None	None
6-F	2 1/2	3/4	None	None
6-G	None	None	None	None
7-A	None	6	1 [5]	8 [5]

Table 9. Results of 9 months' exposure to weather of 74 calkings in concrete joints. [1] (Continued)

Sample No.	Total loss in bond to concrete (inches)[2]			
	Primed joint		Unprimed joint	
	Top	Bottom	Top	Bottom
8-A	None	None	None	None
8-B [7]	None	None	4 [3]	None
8-C [8]	None	None	None	None
8-D [8]	None	None	None	None
8-E [7]	None	9	None	None
9-A	None	None	None	1/2
9-B	9 [5]	5 [5]	None	None
9-C	4	3	2	None
9-D [4]	None	1 1/2	None	None
9-E [4]	1	None	3 1/2 [5]	2 [5]
9-F [4]	4 [5]	8 [5]	3 [5]	7 [5]
10-A	4 1/2	None	None	None
10-B	4 [5]	1 [5]	9 [5]	3 [5]
10-C [8]	None	None	None	None
10-D	Complete[5]	Complete[5]	1/4	1/2
10-E [4]	None	None	None	None
10-F	None	None	None	3/4
11-A	None	2 1/2	None	2
11-B [4]	None	None	None	None
11-C	None	None	None	None
11-D	None	None	None	None
12-A	None	1/4	None	None
12-B	None	None	None	None
12-C	None	None	None	None
12-D [4]	None	None	None	None
13-A	None	3 1/2	None	None
13-B	None	None	None	None
13-C	2	6	1/4	None
13-D	1/2 [5]	1/2 [5]	1/4 [3]	None
13-E	None	None	None	None
13-F	1/4	None	None	None
13-G	None	None	None	None
14-A	Complete[5]	Complete[5]	Complete[5]	Complete[5]
14-B	None	None	None	None
14-C	None	None	None	1 1/2

Table 9. Results of 9 months' exposure to weather of 74 calkings in concrete joints. 1/ (Continued)

Sample No.	Total loss in bond to concrete (inches) 2/			
	Primed joint		Unprimed joint	
	Top	Bottom	Top	Bottom
15-A	None 5/	None	None	None
15-B	3 1/2	2 5/	5 5/	6 /5
15-C	None	None	None	1/4
15-D	None	None	None	None
16-A	None	None	None	None
16-B	None	None	None	None
17-A 10/	None	None	None	None
17-B 10/	None	None	None	None

1/ Calkings are polysulfide rubber base, non-sag type, except where specified in a footnote.
2/ Figures (in inches) denoting loss of bond are for bond breaks, except where specified by a footnote.
3/ Cohesion (or ductility) break.
4/ Flow type compound.
5/ Joint leaked.
6/ Small air holes on surface.
7/ Compound classed as "hard" by producer.
8/ Compound classed as "soft" by producer.
9/ Large closed air pockets.
10/ Silicone rubber.

Table 10. Relationship between bond loss factor and exposure test results.

Sample No.	Bond-Loss 1/ Factor (Avg.)	Exposure Test (9 mo.) Bond Loss, in. 1/	Correlation
2-C	23	0	Poor
3-A	12.2	3.5	Good
3-D	.2	0	Good
7-A	24.2	15 (leaked)	Good
8-A	4.0	0	Good
8-B	16.0	4	Good
8-D	0	0	Good
9-A	25.6	.5	Poor
9-E	9.0	6.5 (leaked)	Poor
10-A	10.3	4.5	Poor
10-B	26.2	17 (leaked)	Good
10-D	27.5	11 (leaked)	Good
11-A	2.4	4.5	Poor
14-A	12.2	complete (leaked) failure	Poor

1/ Results are the values obtained on primed and unprimed specimens.

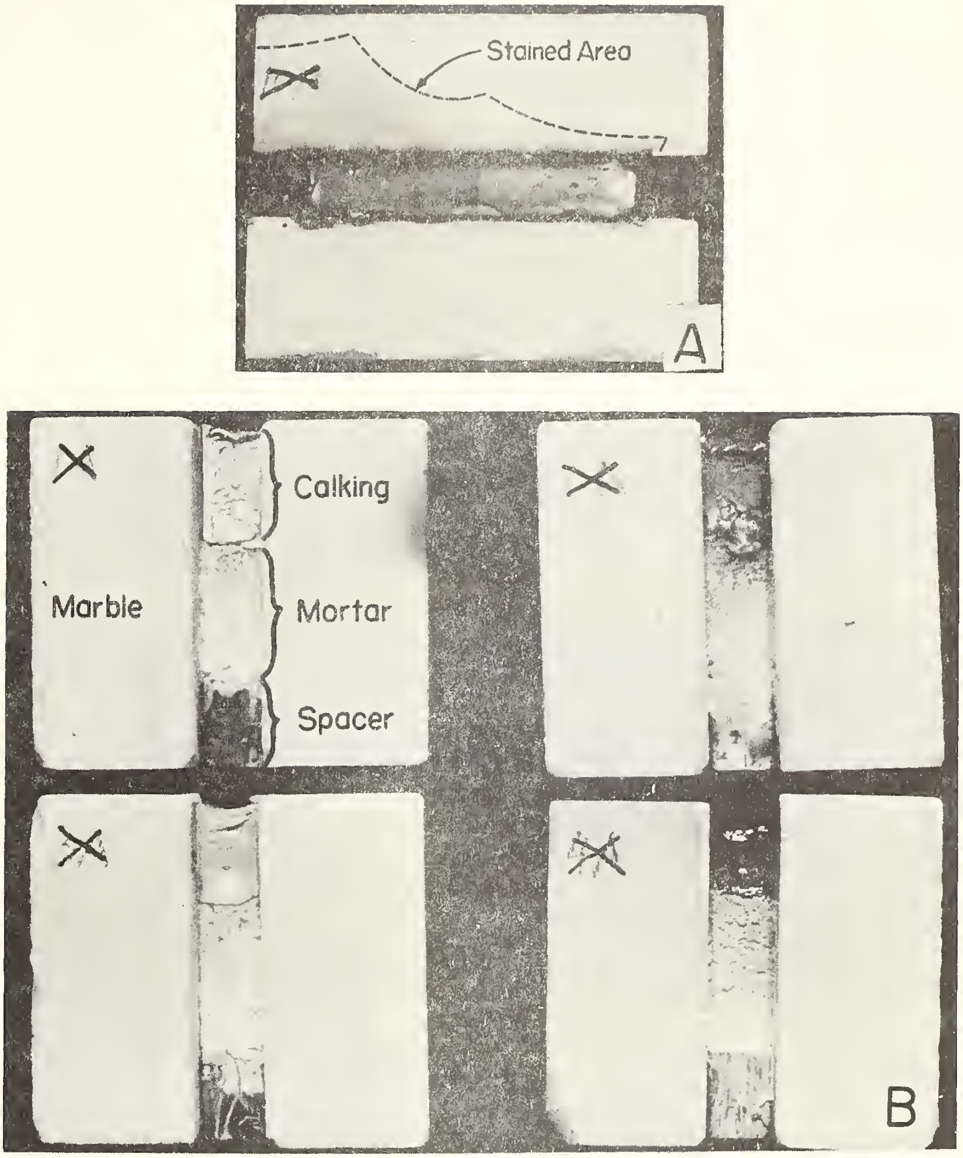


Fig. 1. Staining of marble and mortar caused by contact with Thiokol base calking compounds:
(A) Example of stain (appears red) on white marble after 500 hours exposure in a Weatherometer. Calking joint is 1 1/2 in. long, 1/2 in. deep and 3/8 in. wide, backed by 3/4 in. white cement mortar and 1/2 in. aluminum spacer.
(B) End views of 4 specimens of different calkings with mortar and spacers as in (A), showing prominent stains (red and brown) in mortar after 500 hours exposure in the Weatherometer.

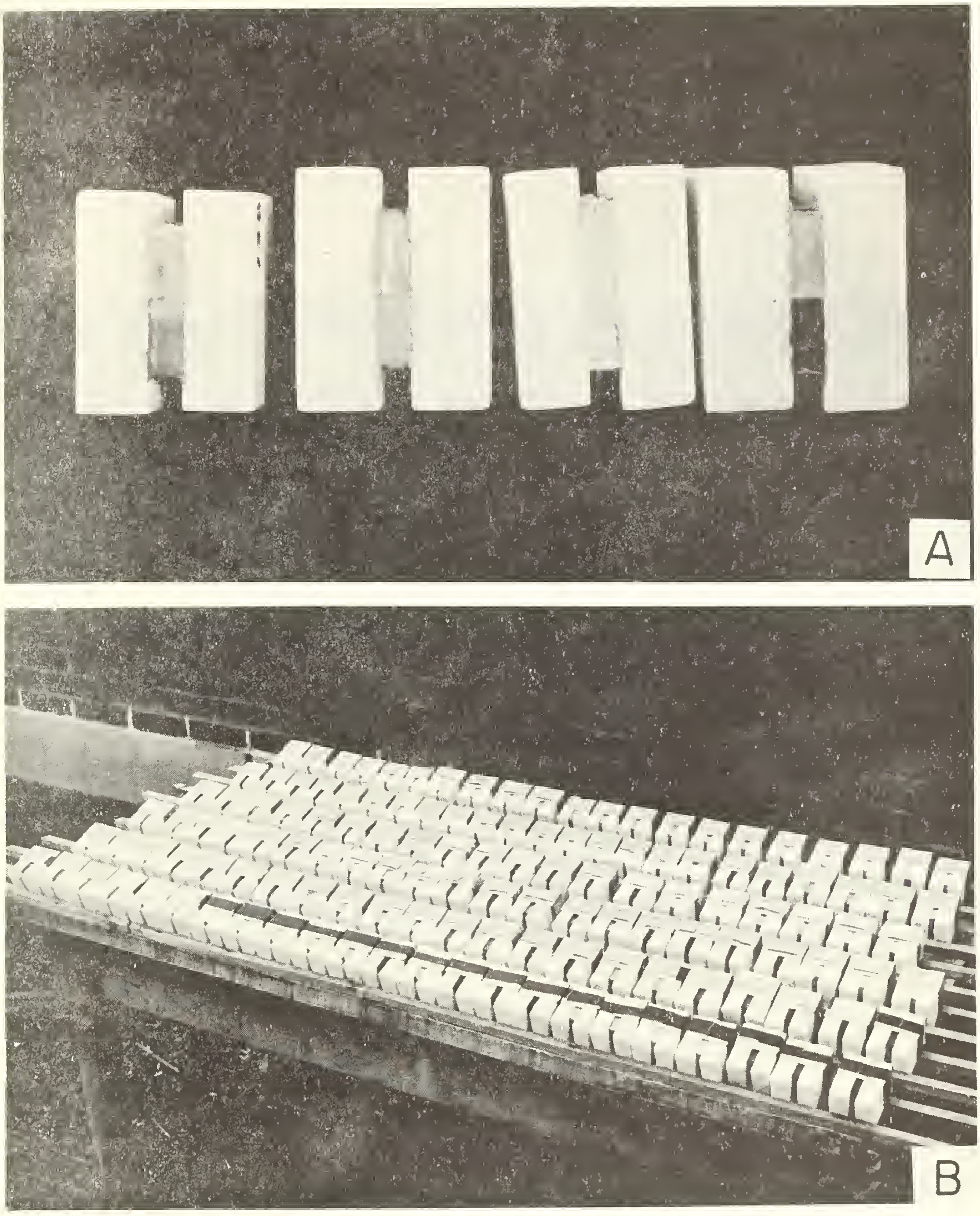


Fig. 2. Test specimens of rubber base calkings fabricated like those in Fig. 1, for study of staining of masonry and color changes in the calkings.

(A) Examples of color changes in calkings after 500 hours in Weatherometer. Top half of joint was covered during test.

(B) After exposure in the weatherometer, specimens are exposed on the roof of the building with one half of each joint covered by a strip of aluminum.

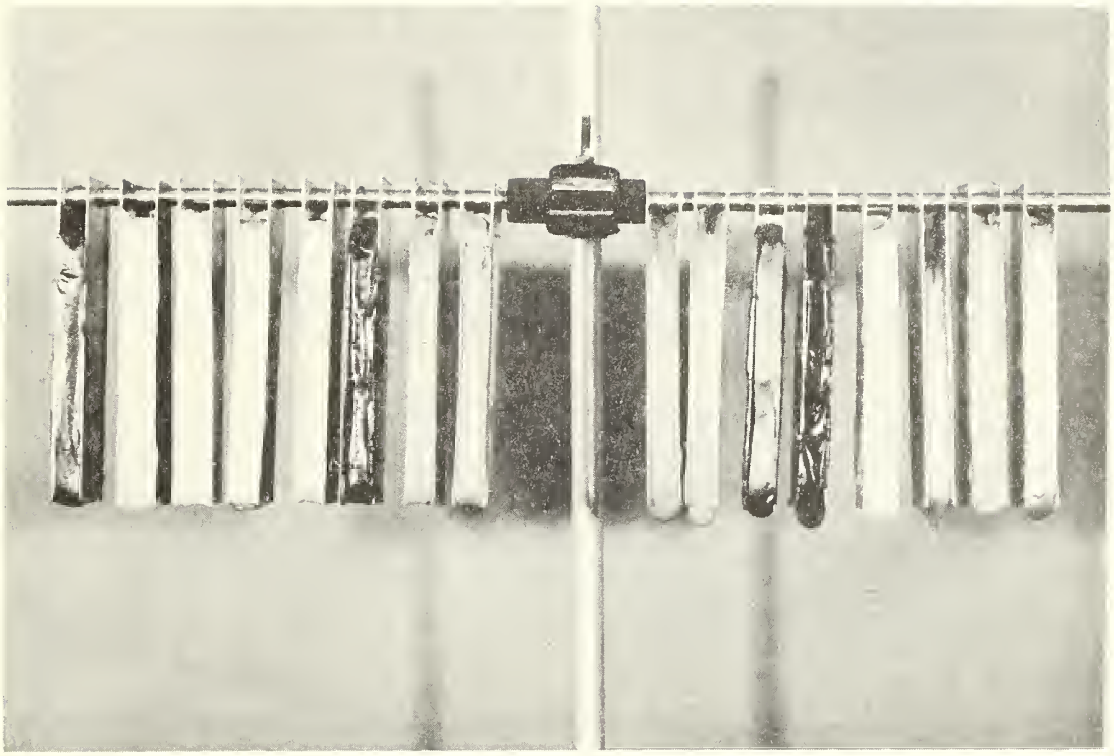


Fig. 3. Laboratory test of slump or flow property of rubber base calkings. Freshly mixed compounds are placed in aluminum troughs, 4 in. by 1 in. by 3/8 in. and are suspended for 24 hrs at ~~74° ± 2° for 24 hrs.~~ 122 ± 2° F. The eight specimens on the right side show varying degrees of slump while those on the left show none.

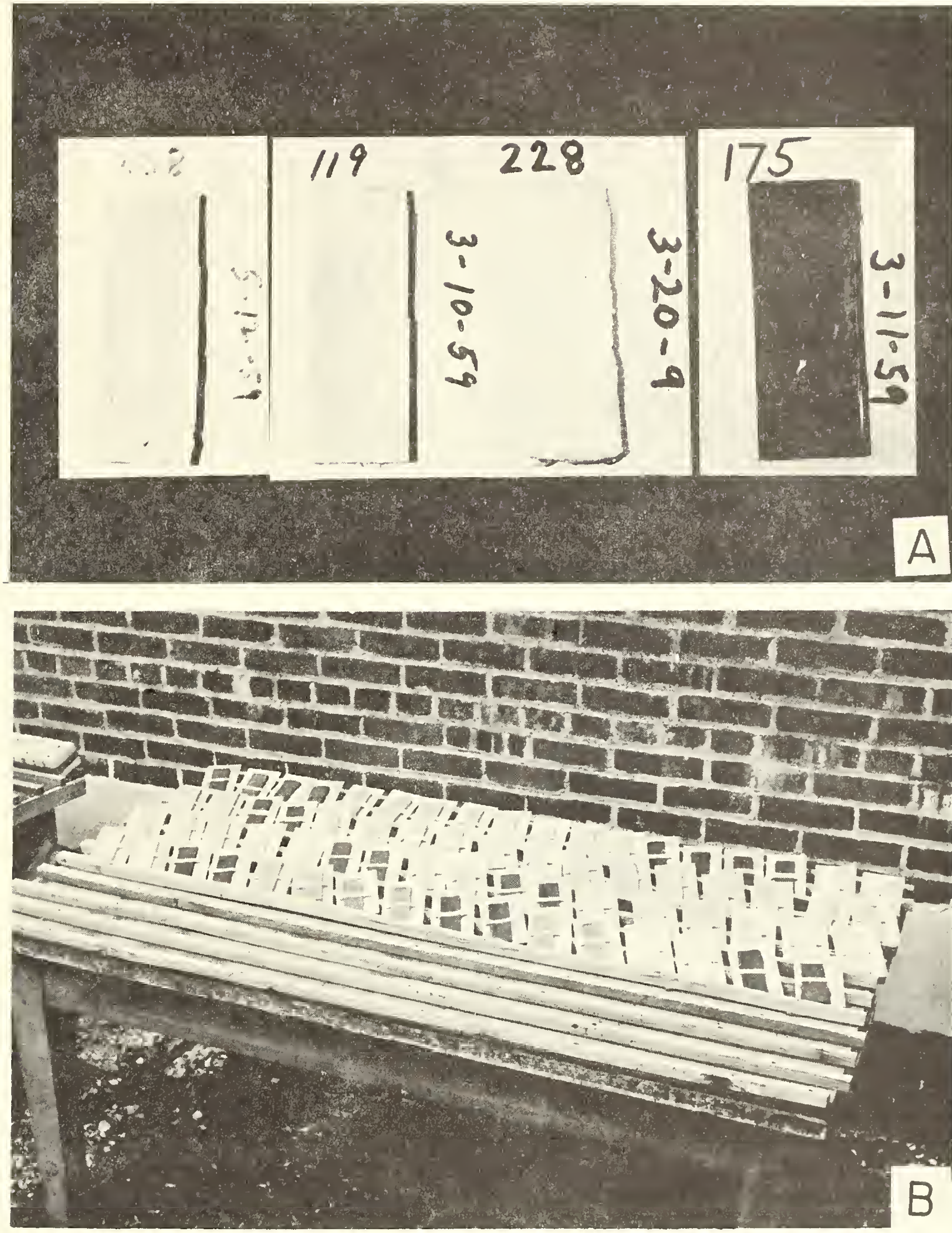


Fig. 4. Calking specimens used for the determination of Shore A hardness.
(A) Hardness specimens, 4 in. by 1 1/2 in by 1/4 in. are placed on aluminum panels and tested 10 to 14 days after compounds are mixed.
(B) Hardness specimens exposed on the roof of the building for future study.

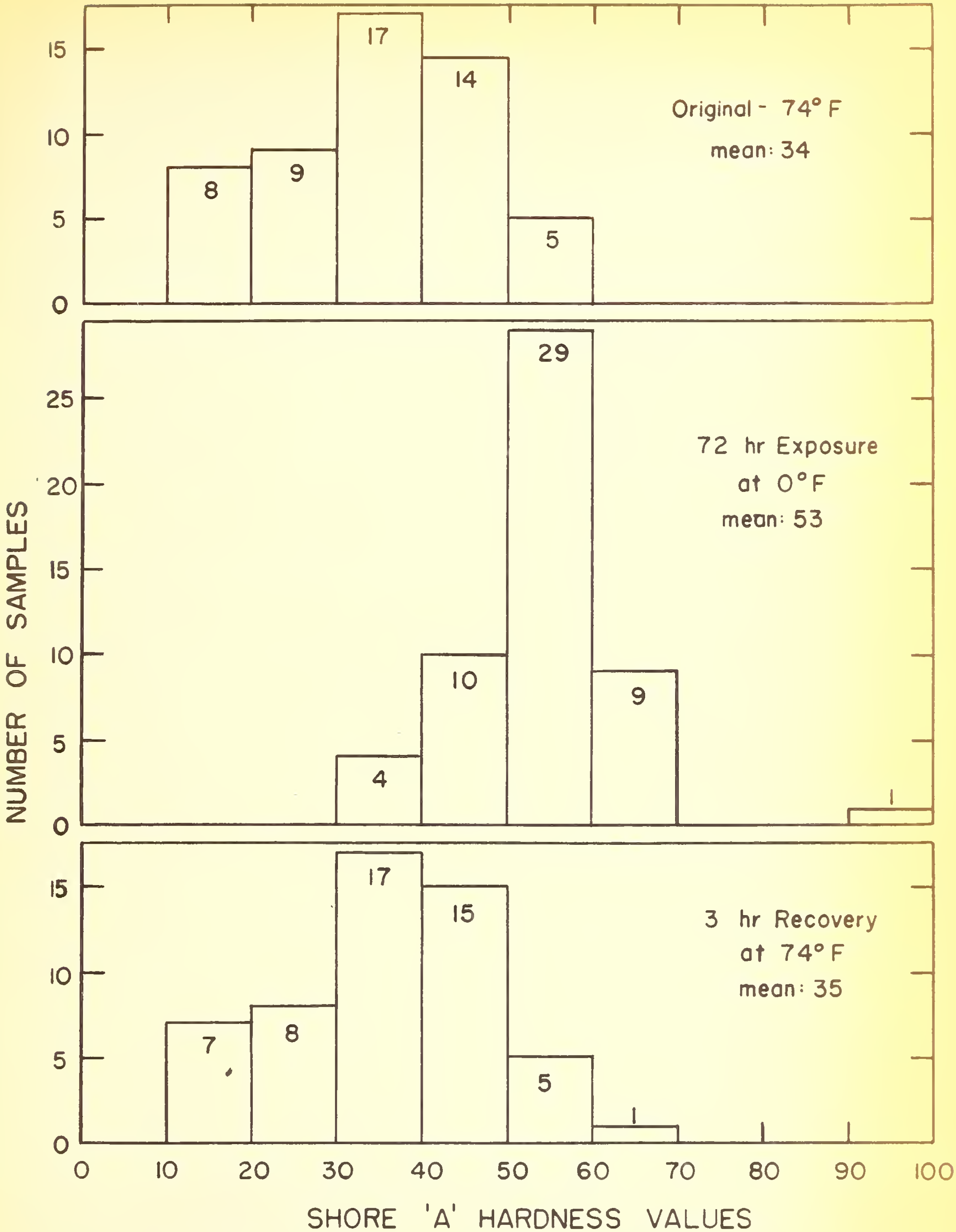


FIG. 5 DISTRIBUTION OF SHORE 'A' HARDNESS VALUES OBTAINED ON 53 SPECIMENS OF RUBBER BASE CALKINGS.

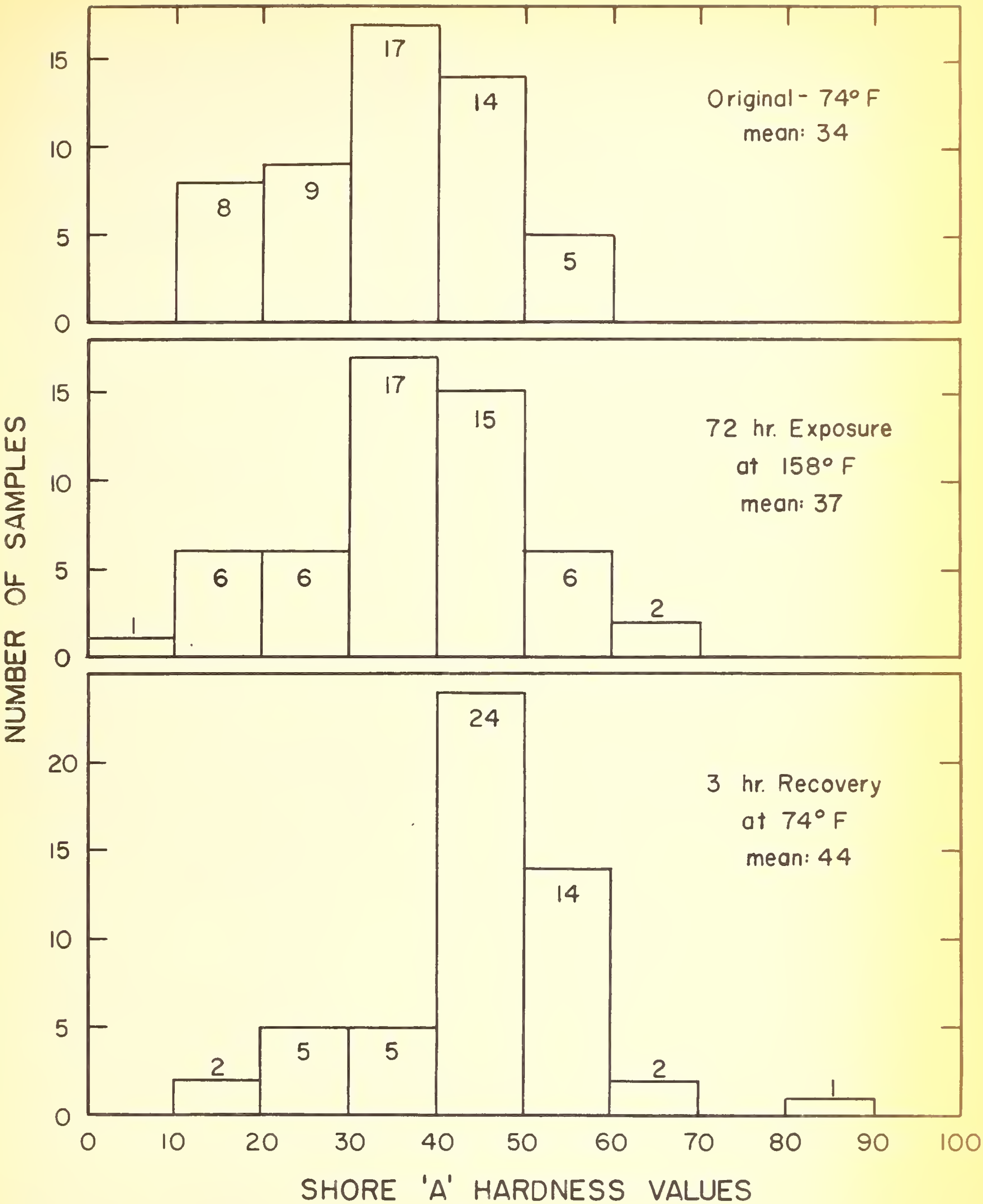


FIG. 6 DISTRIBUTION OF SHORE 'A' HARDNESS VALUES OBTAINED ON 53 SPECIMENS OF RUBBER BASE CALKINGS.

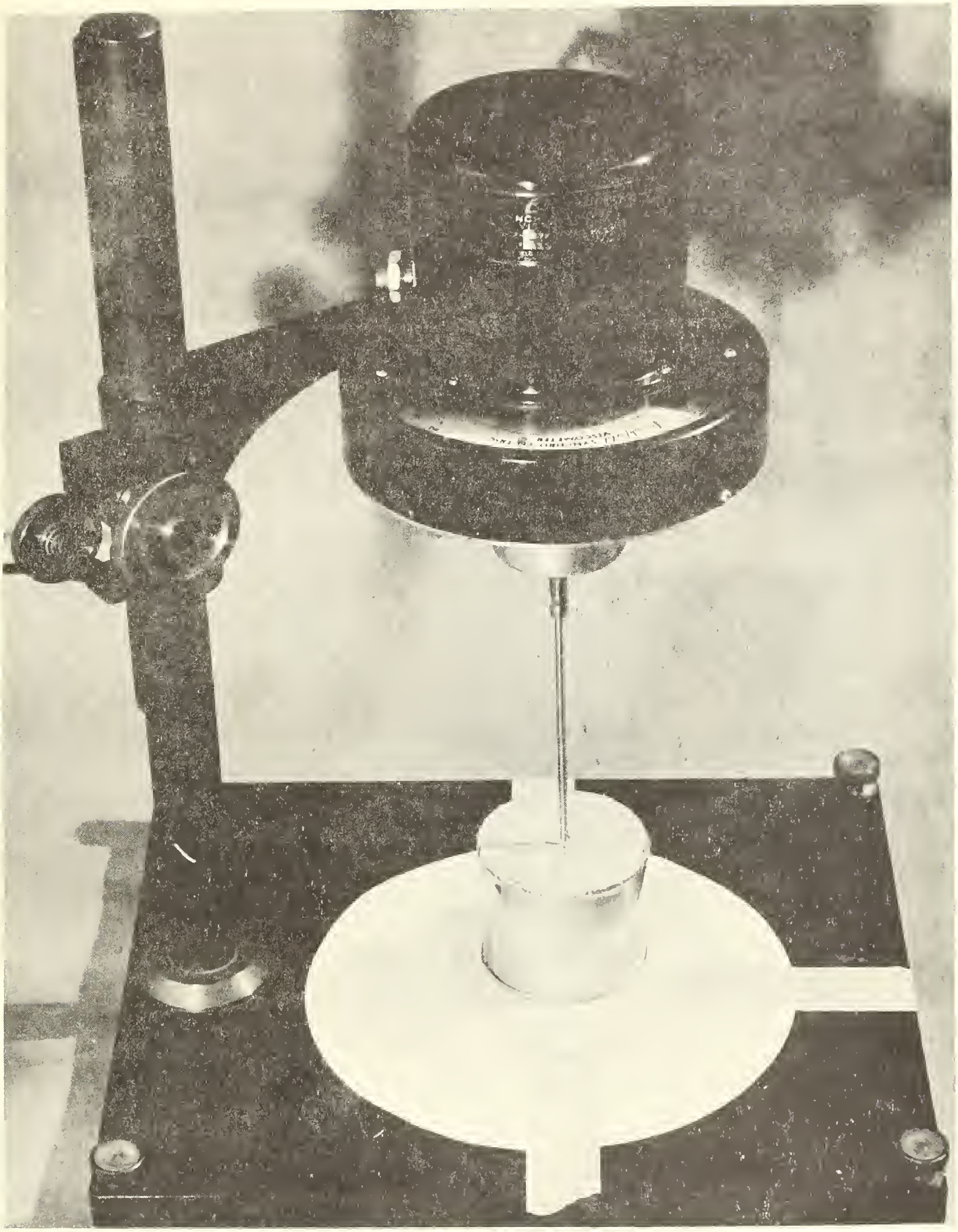


Fig. 7. Application or "pot" life of the calkings was measured with a Brookfield Viscometer fitted with no. 7 spindle rotating at 2 rpm. Readings were taken periodically and test was stopped when pointer reached the limit of the scale (100) or after 180 minutes- whichever came first.

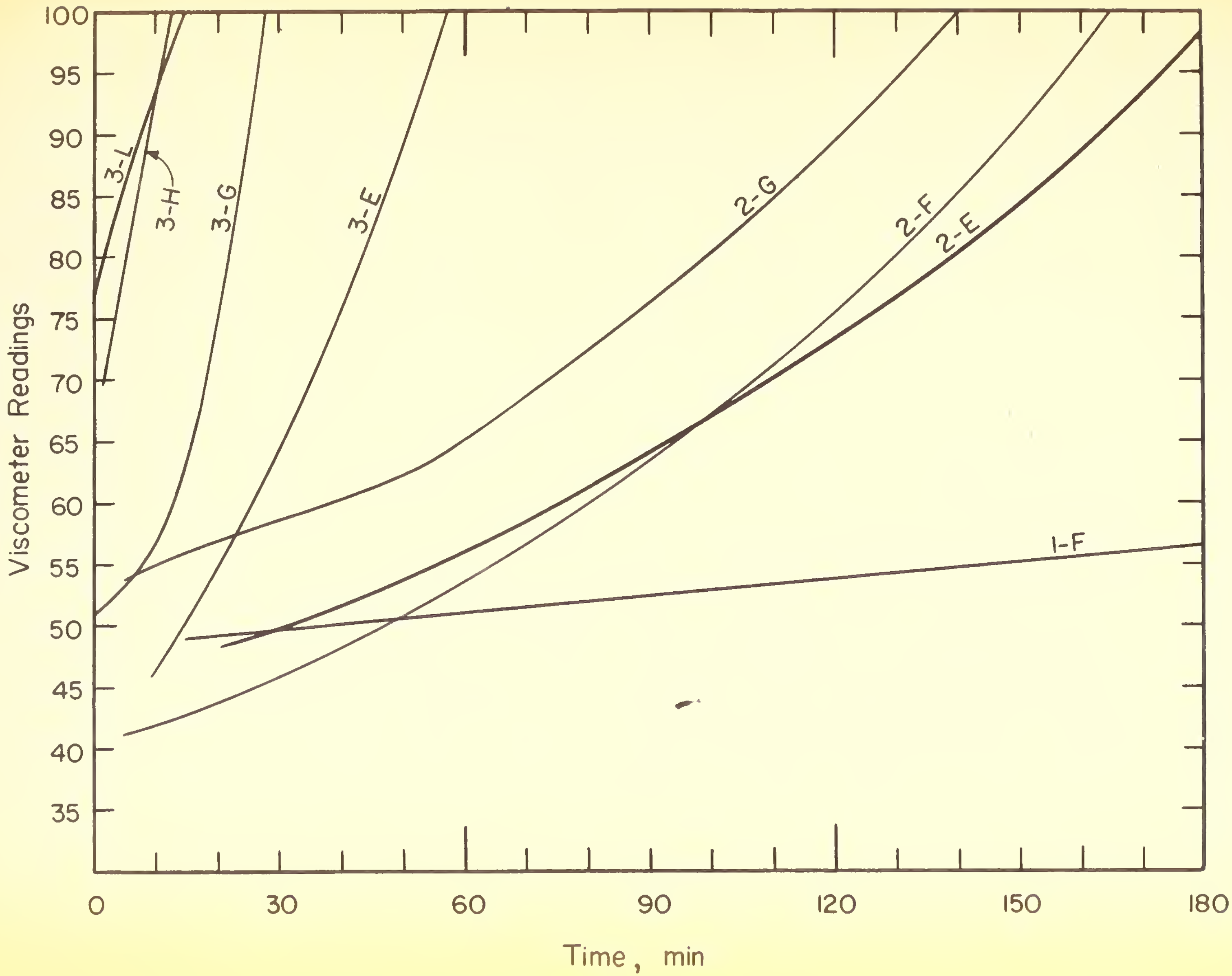


Fig. 8 Relationship of viscometer readings with elapsed time obtained in the determination of application life of calking samples.

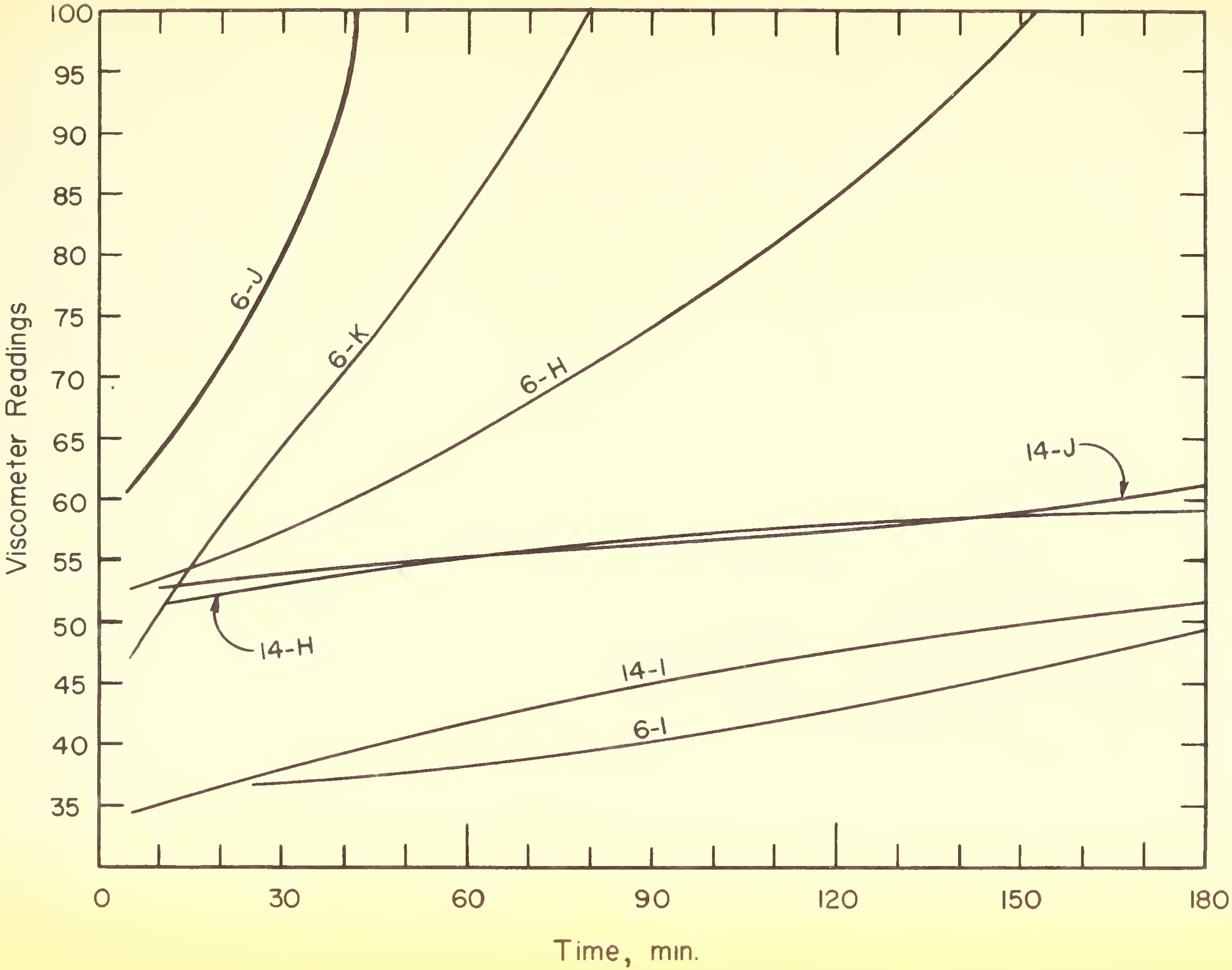


Fig. 9 Relationship of viscometer readings with elapsed time obtained in the determination of application life of calking samples.

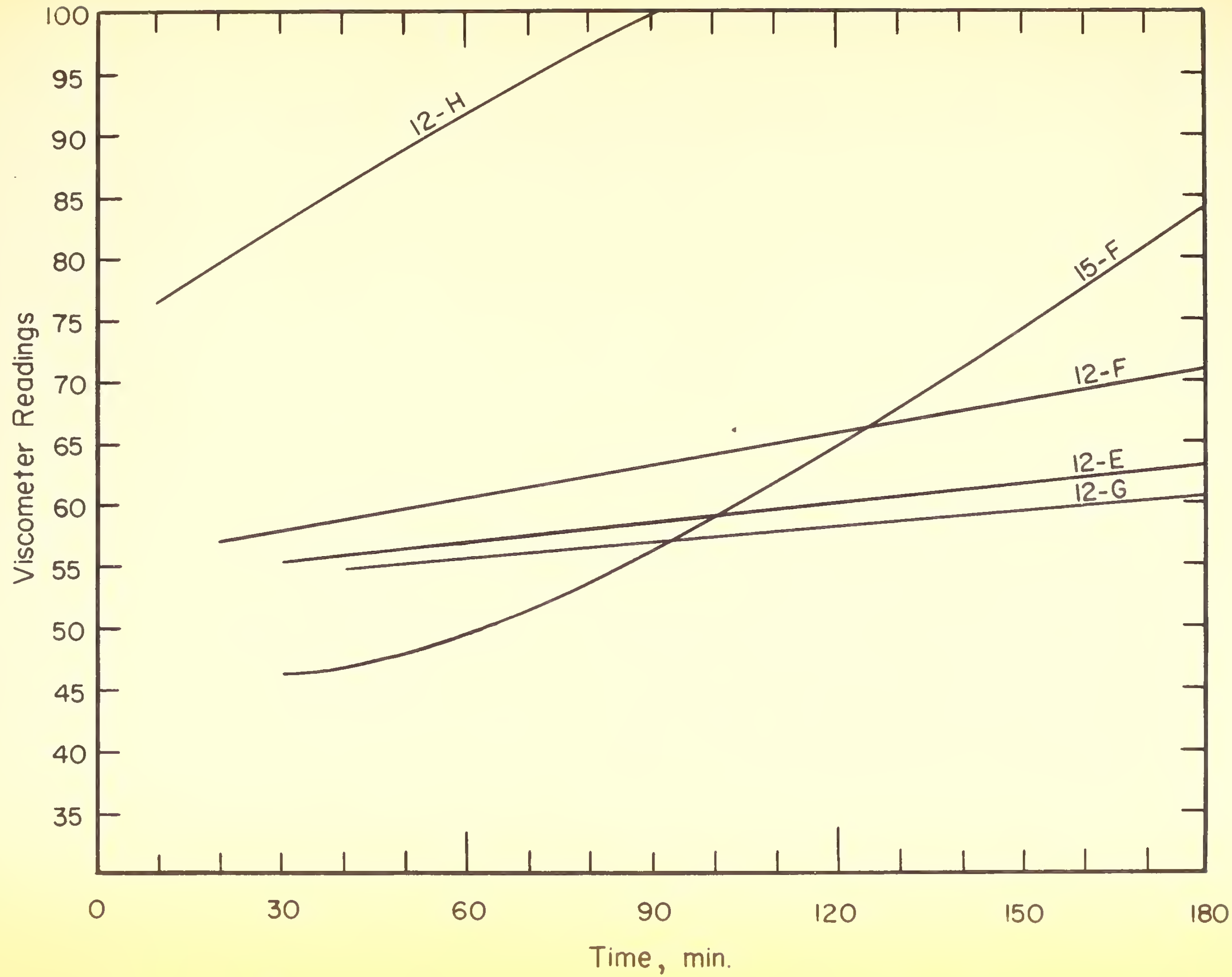


Fig. 10 Relationship of viscometer readings with elapsed time obtained in the determination of application life of calking samples.

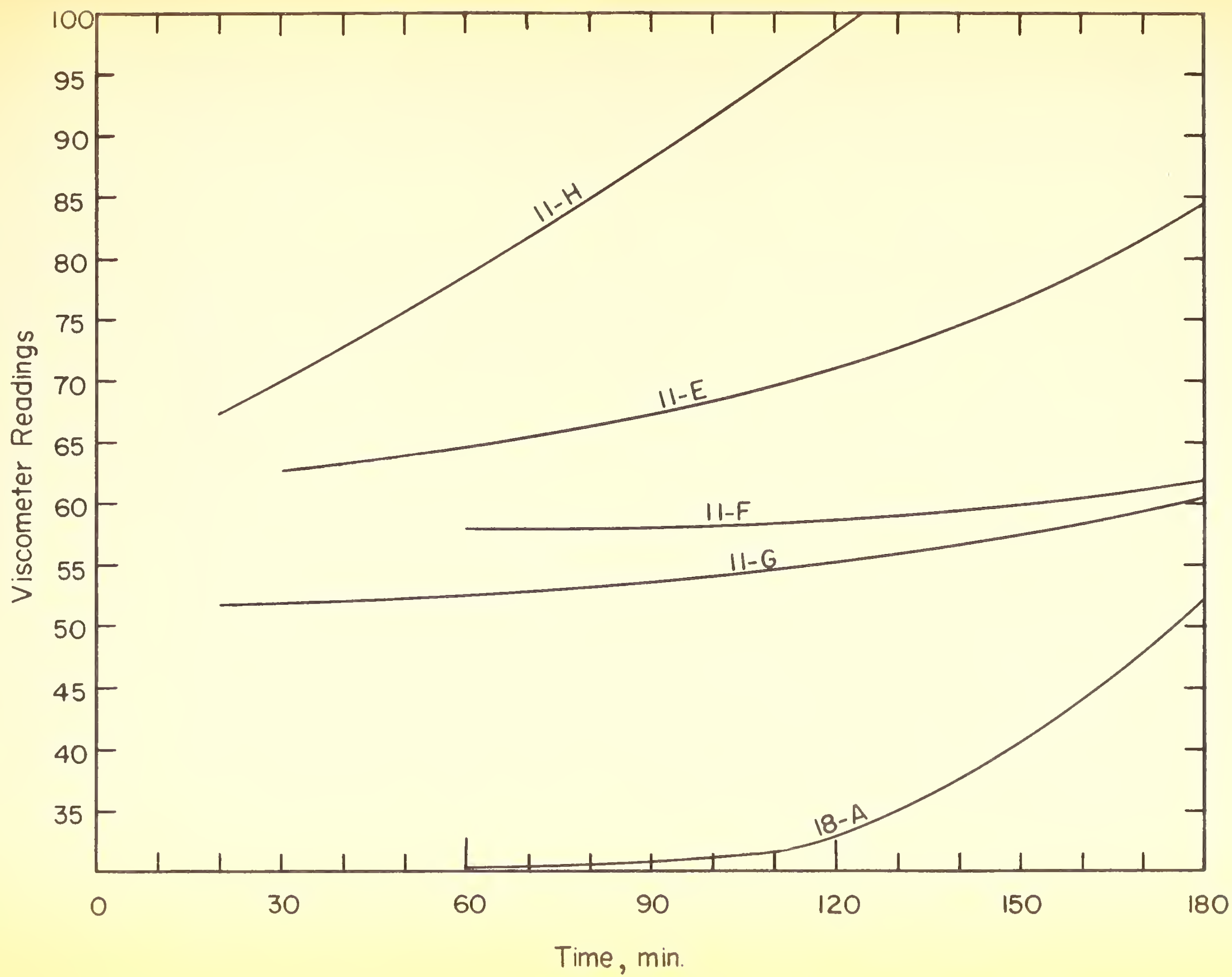


Fig. 11 Relationship of viscometer readings with elapsed time obtained in the determination of application life of calking samples.

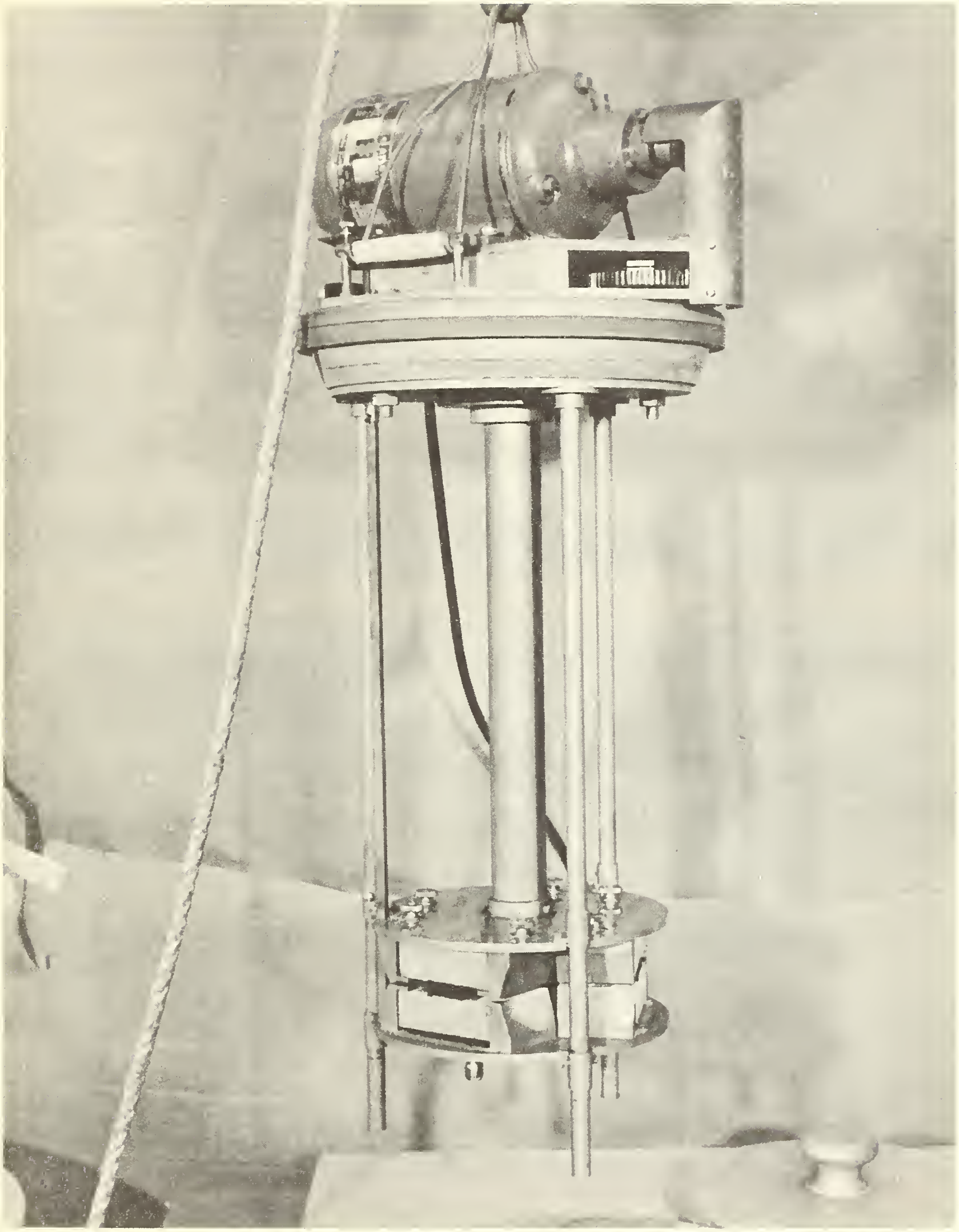


Fig. 12. Joint material extension machine used in the bond-ductility tests. Joint is stretched 50% of its original width at 0 deg F, at the rate of 1/8 in. per hour.

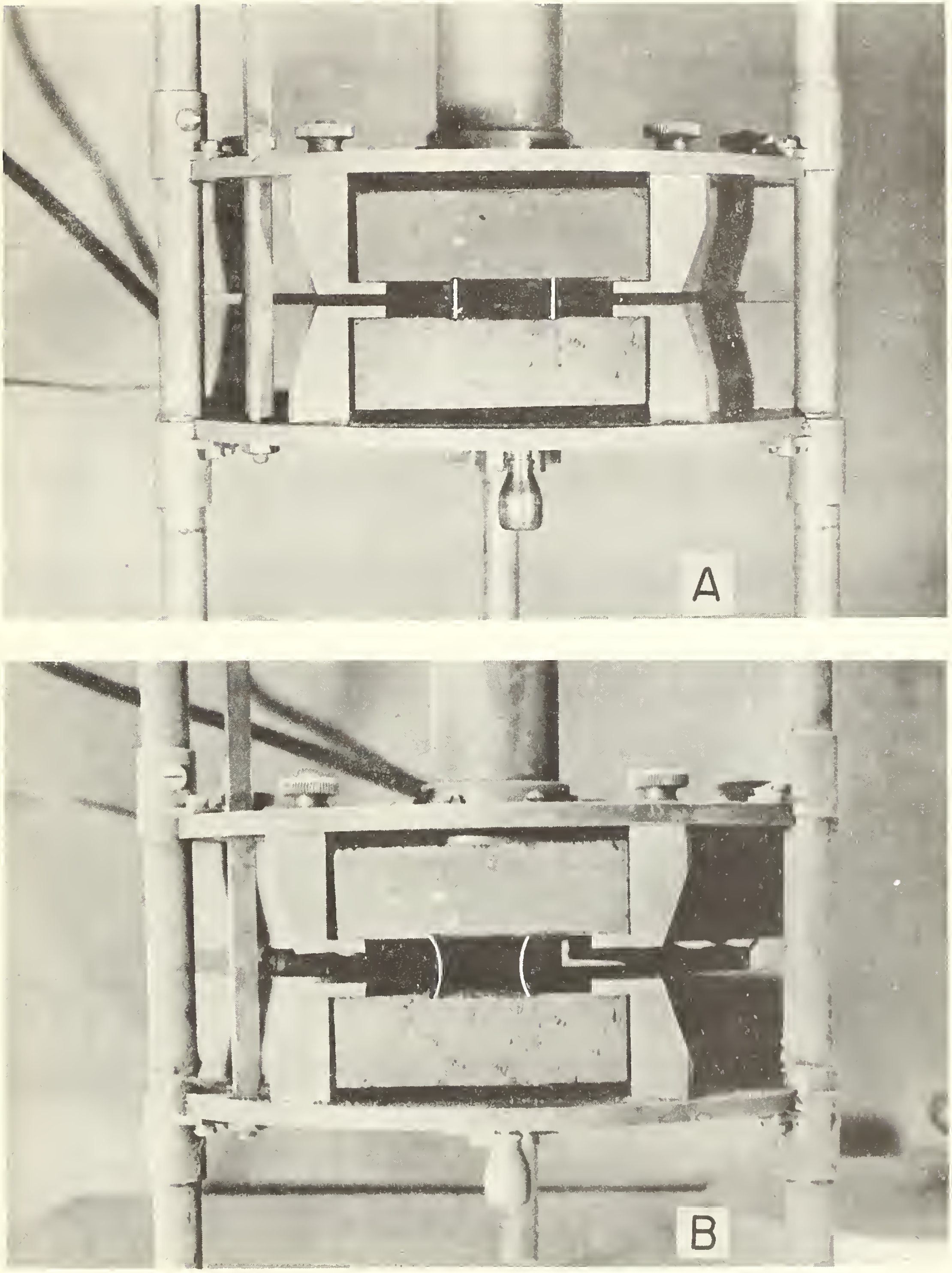


Fig. 13. Bond-ductility test specimens in joint material extension machine.
(A) Specimen with joint, 2 in. by 1 in. by 3/8 in. before extension.
(B) Specimen after extension of 50% of its original width.

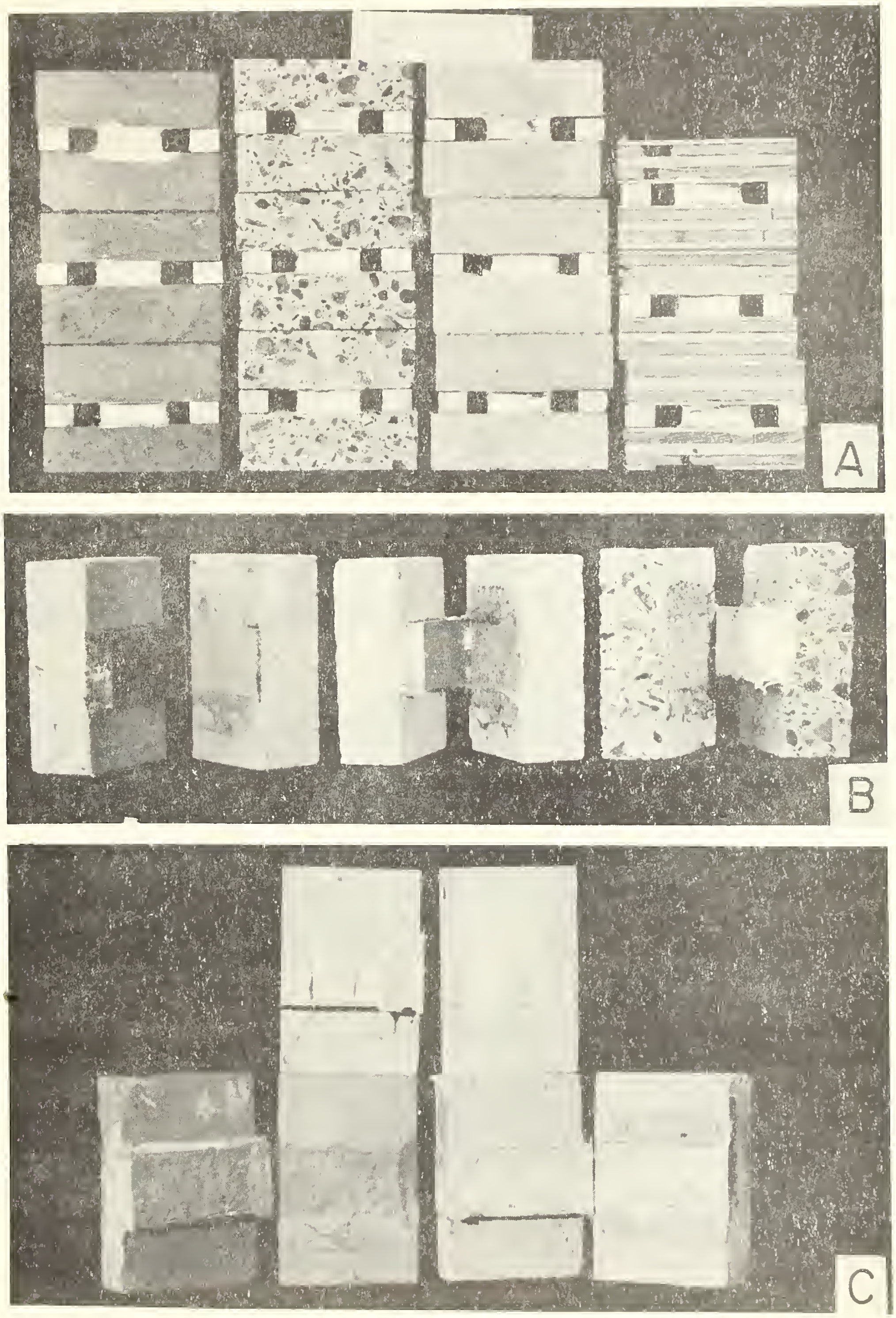


Fig. 14. Bond-ductility test specimens. (A) Typical specimens before test: brick, concrete block, concrete and wood. (B) Examples of complete bond failure after test. (C) Failure caused by combination of high hardness of calking and strong adhesion resulting in breakage of the accessory blocks

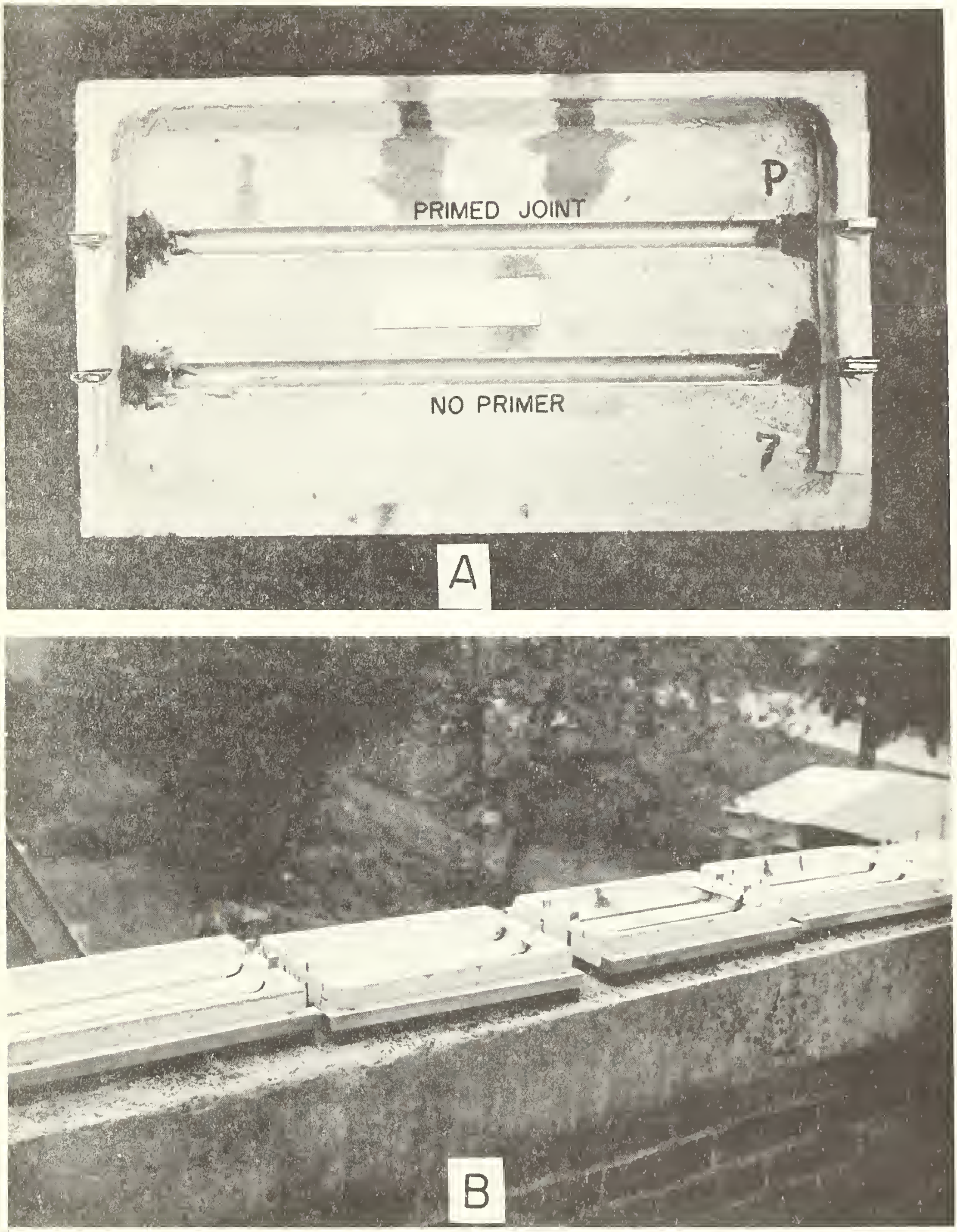
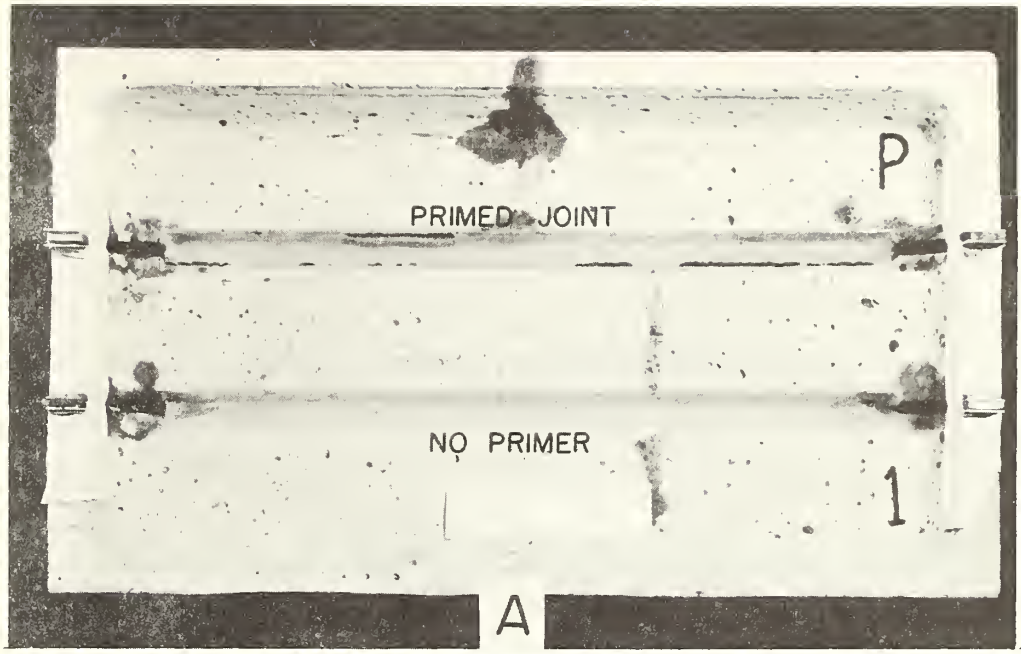


Fig. 15. Calked joints used in outdoor exposure studies.
(A) Typical concrete trough containing two joints 11 in. by 3/4 in. by 3/8 in., primed and unprimed, and filled with the calking under test.
(B) Troughs exposed to the weather on the coping of the Mineral Products building. Joints are stretched 1/32 in. every 3 months.



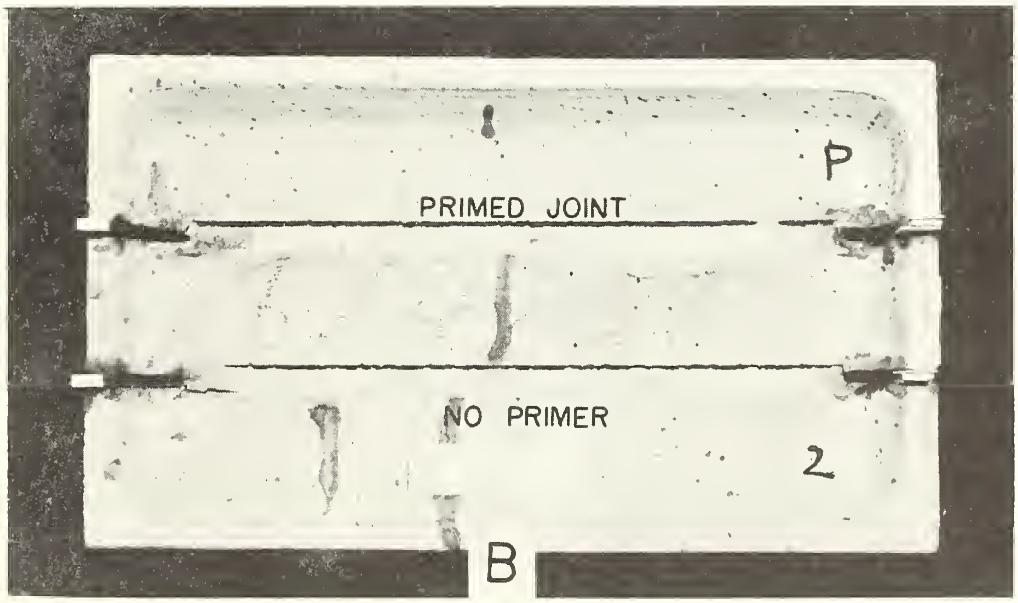
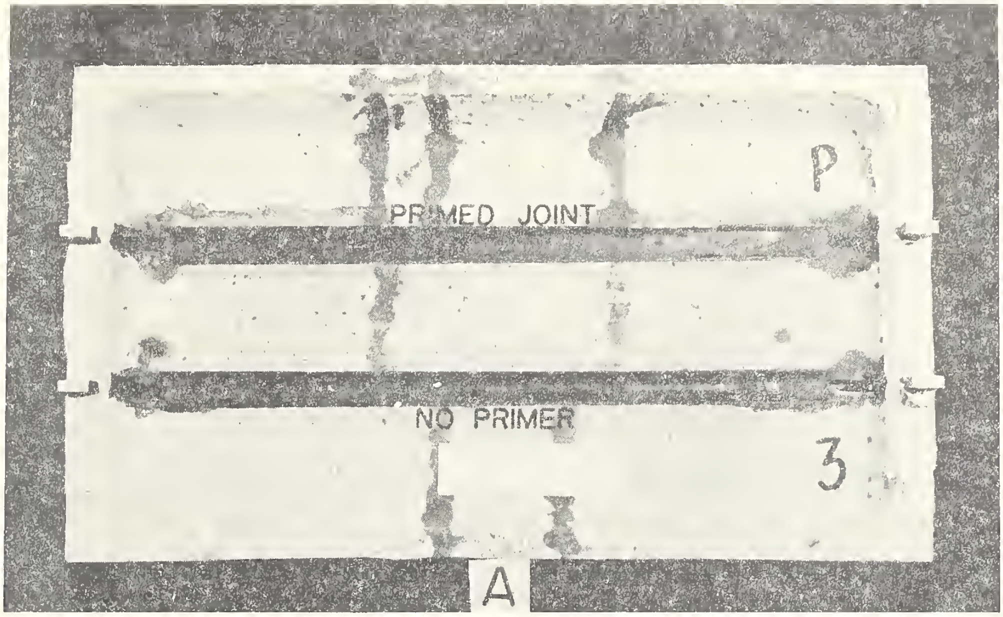


Fig. 16. Effect of six months exposure to the weather on calkings in concrete joints. Joints were stretched total of 3/32 in.
(A) Sample 9-B shows several bond breaks in primed joint and none in unprimed joint.
(B) Sample 14-A shows complete bond failure in both primed and unprimed joints.



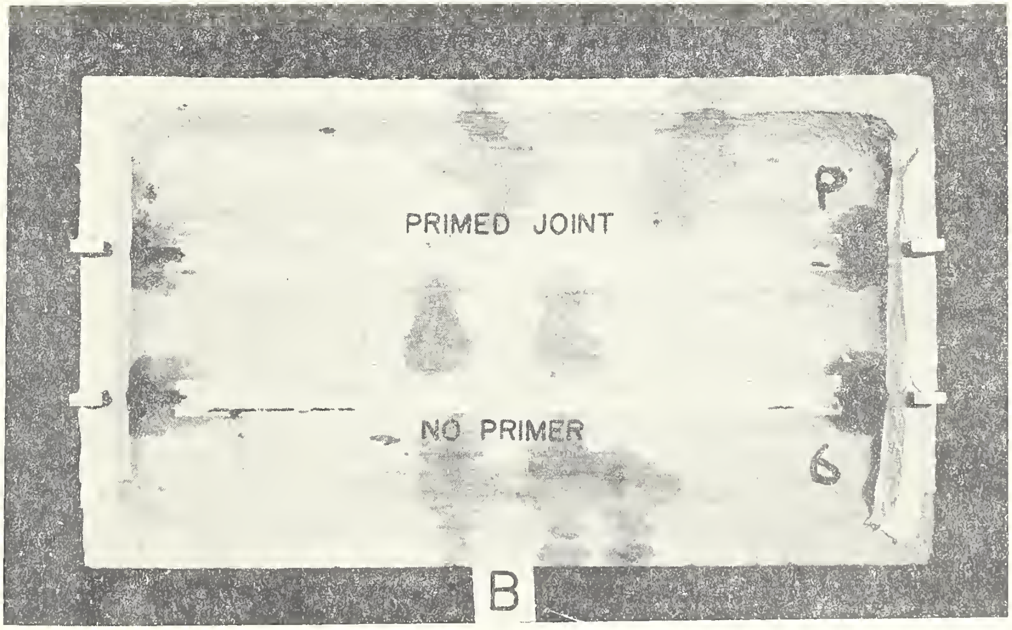


Fig. 17. Effect of six months exposure to the weather on two samples of calkings in concrete joints. Joints were stretched a total of 3/32 in.
(A) Sample 10-D shows complete bond failure in the primed joint and almost no failure in the unprimed joint.
(B) Sample 4-D shows about 3 in. of bond breaks in the unprimed joint and about 1/4 in break in the primed joint.

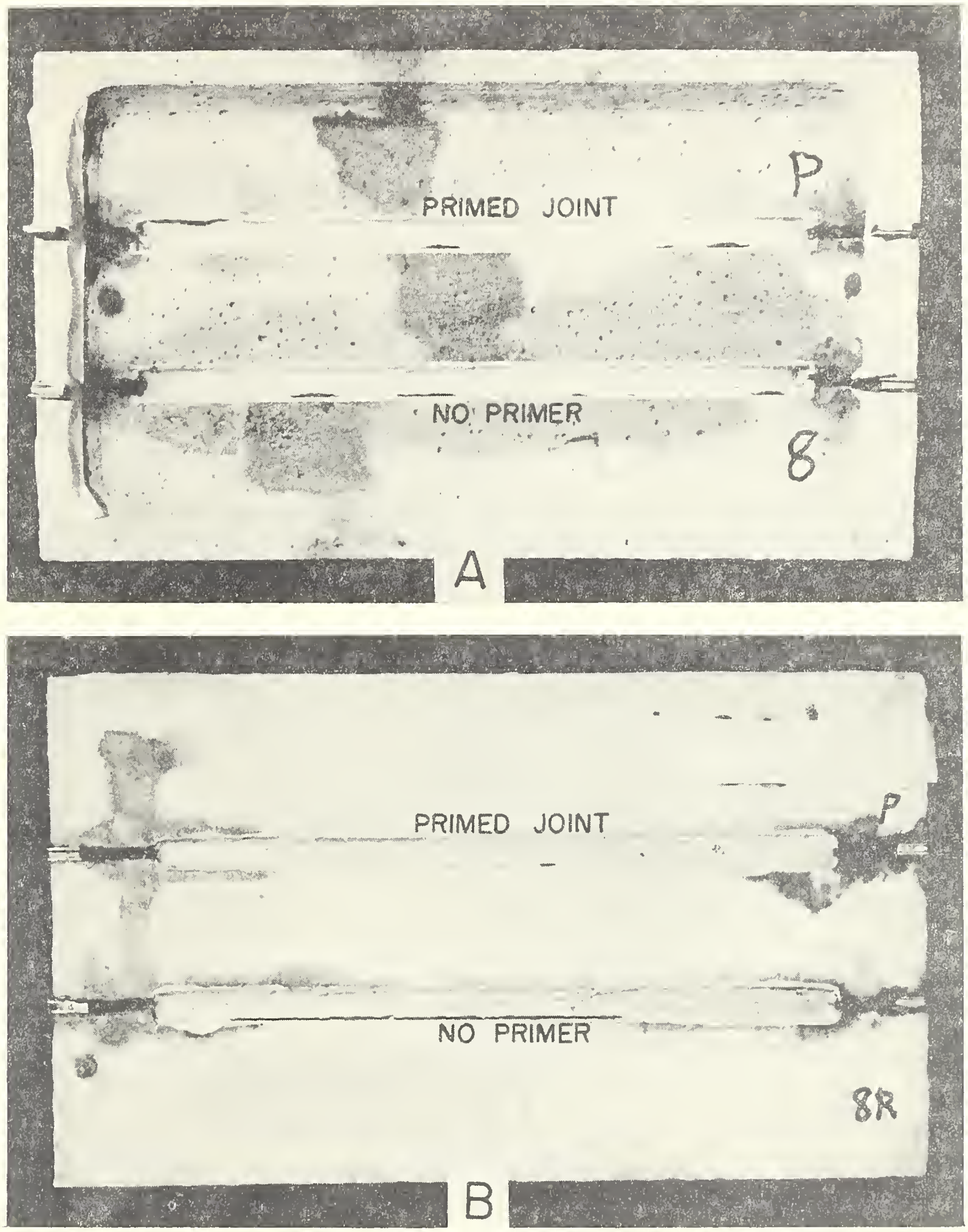


Fig. 18. Effect of six months exposure to the weather on a sample of calking in concrete joints (No. 15-B)
(A) Top surface of trough showing bond breaks in primed and unprimed joints.
(B) Under side of same trough showing more extensive bond breaks in unprimed joint and only about 1/4 in. break in primed joint.

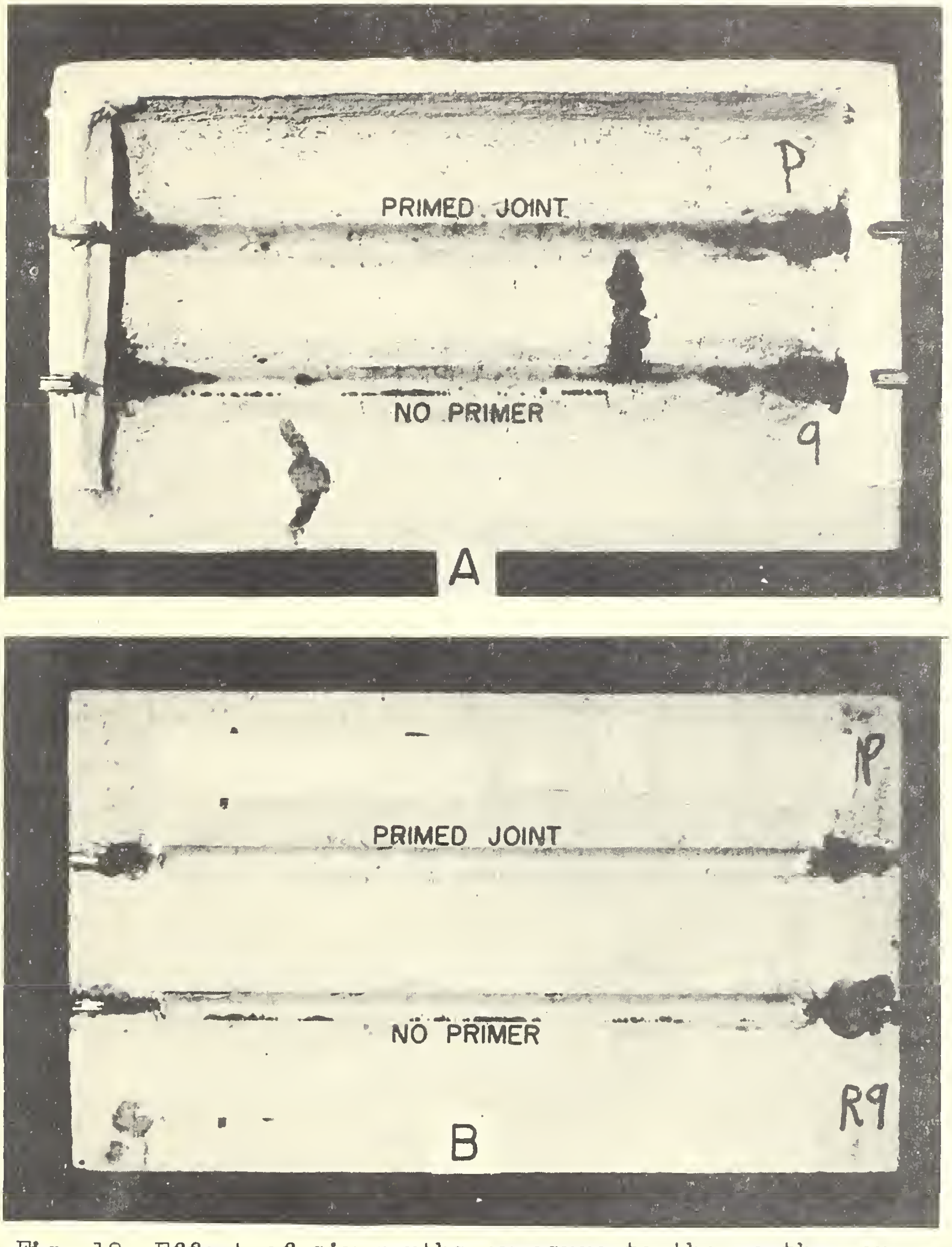


Fig. 19. Effect of six months exposure to the weather on a sample of calking in concrete joints (No. 6-B) Joints were stretched a total of 3/32 in.

(A) Top surface of trough showing about 4 1/2 in. of bond breaks in the unprimed joint and none in the primed joint.

(B) Under side of same trough with slightly more extensive breaks in the unprimed joint and none in the primed joint.

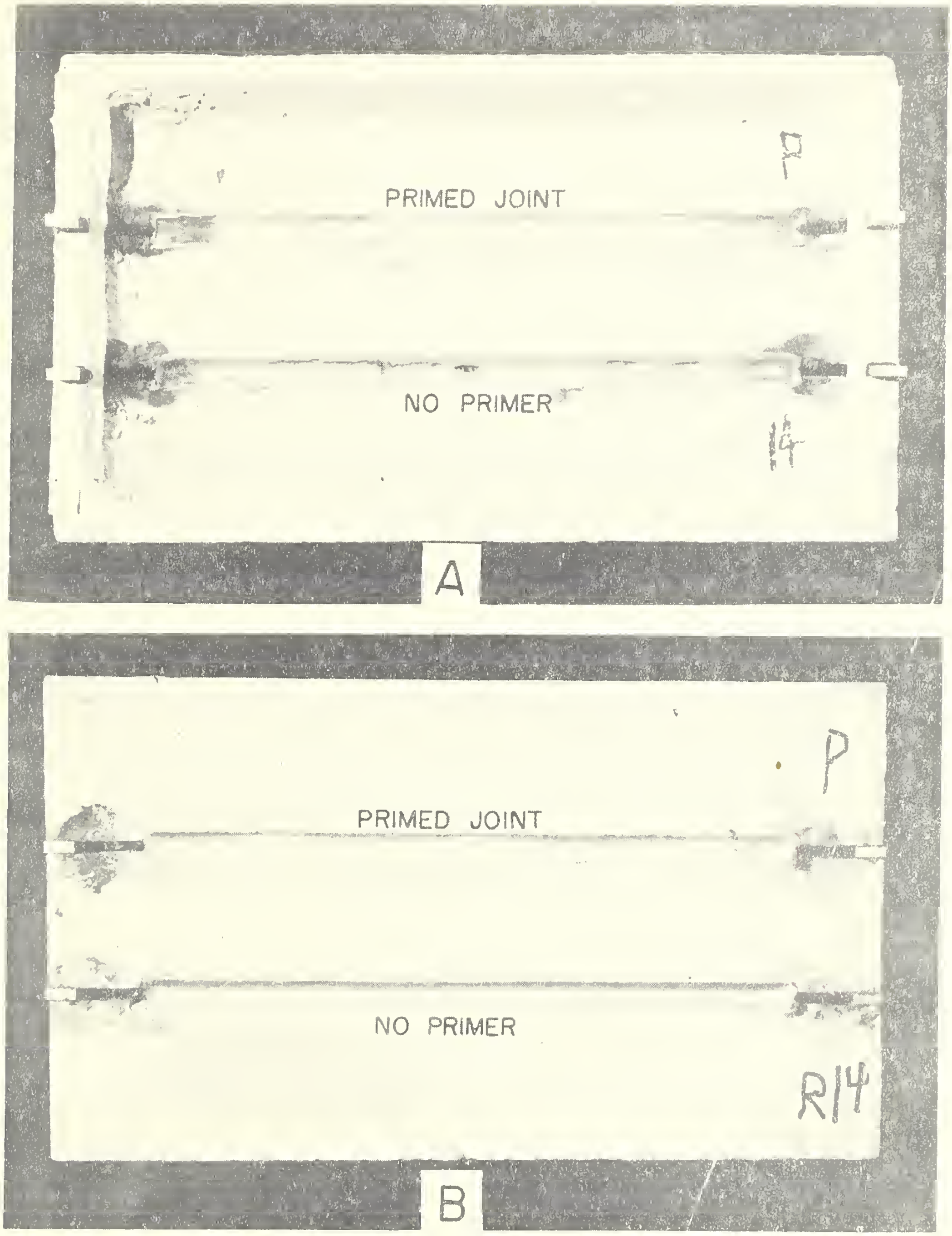


Fig. 20. Effect of six months exposure to the weather on calking sample No. 8-B. Joints were stretched a total of 3/32 in.

(A) Top surface of trough shows some ductility breaks in unprimed joint and none in primed joint.

(B) Bottom surface of trough shows no breaks in both joints.

U.S. DEPARTMENT OF COMMERCE
Frederick H. Mueller, Secretary

NATIONAL BUREAU OF STANDARDS
A. V. Astin, *Director*

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