NATIONAL BUREAU OF STANDARDS REPORT

6923

Study of the Properties of Rubber Base Calking Materials
(Progress Report No. 2)

by

Arthur Hockman

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

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

THE NATIONAL BUREAU OF STANDARDS

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U. S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

Study of the Properties of Rubber Base Calking Materials
(Progress Report No. 2)

Arthur Hockman

A study of the properties of polysulfide base joint sealers for use in masonry and other types of construction has been completed. For this purpose 157 samples were received from 19 manufacturers.

Standard laboratory test procedures have been developed for six physical properties: (1) staining effects and color changes, (2) slump, (3) tack-free time, (4) application life and ease of extrusion, (5) hardness, (6) durability.

Of 31 samples tested for staining, 25 produced stains on mortar exposed both in the laboratory at 74°F and in the Weatherometer for 500 hr. Intense red stains formed when the sample was protected from sunlight in the laboratory. Less intense stains of reddish brown colors occurred on samples in the Weatherometer or in sunlight. The stains tended to fade with time. White, gray, neutral, and tan compounds darkened to varying degrees after 500 hr in the Weatherometer.

Of 24 non-sag type compounds tested at 122°F in two types of slump troughs only one slumped excessively. Tack-free time tests made on 22 samples resulted in 3 samples remaining tacky for more than 72 hr.

Application life tests made with the Brookfield Viscometer indicated 41 percent of the 29 samples tested (non-sag type) as compounds with short application or pot life. Of eight flow type compounds tested, five also showed short pot life.

Shore "A" hardness values obtained on 100 samples from 17 producers ranged from 10 to 58 at 74°F with an average of 34. Average hardness increased by 56 percent when tested at 0°F but showed no change when tested at 158°F. There was no residual increase in hardness after the 0°F treatment but after cooling from 158°F, the residual increase was 24 percent. Seventeen samples exposed in air at 74°F for 7 to 9 months showed increases in hardness from 0 to 61 percent. Fourteen samples exposed outdoors for 16 months increased in hardness from 0 to 147 percent with an average of 31 percent.

The results of durability (bond-ductility) tests made on a total of 226 specimens with porous accessory materials (concrete, concrete block, brick and wood) indicated that 52 percent to 60 percent of the block, brick and wood specimens, and 39 percent of the concrete specimens rated "excellent." Twenty percent of each of the above porous groups rated "very poor." Similar tests made on 96 specimens with

non-porous accessory materials (porcelainized steel, porcelainized aluminum, glass, and aluminum) showed 72 percent of the porcelainized steel specimens as "excellent," and 18 percent "very poor." On the other hand, only 18 percent of the porcelainized aluminum specimens were "excellent" and 61 percent rated "very poor." Durability ratings for the glass and aluminum specimens averaged about midway between the porcelainized steel and porcelainized aluminum. Generally, priming of the porous joints did not improve durability.

Test data showed that durability was related to hardness. Results for 91 samples in concrete joints showed the lowest hardness group (10-21) averaged the best durability rating, and the highest hardness group (51-58), averaged the poorest durability. Bubbles formed in joints during the durability test in 26 of 93 samples tested in concrete joints. A relationship of bubble formation with low hardness was indicated.

In the study of durability, 54 percent of the 71 samples exposed outdoors for 21 months were rated "good"; 14 percent rated "fair"; and 32 percent "poor." Durability tests made on 23 samples by the standard laboratory method and by outdoor exposure showed good correlation for 17 of 23 samples tested.

1. INTRODUCTION

This is a second progress report on the investigation of the properties of rubber base calking materials for sealing joints in concrete, brick, stone and other types of masonry construction. The investigation also includes the study of the behavior of joint sealers in contact with non-porous building materials such as porcelainized steel, porcelainized aluminum, aluminum and glass.

This report gives the final results of the study of the polysulfide base joint sealers (Thiokols). Standard test procedures have been developed for six physical properties which will eventually be included in a purchase specification for polysulfide base sealers. Also included are laboratory test results, analysis of the data and results of outdoor exposure studies.

2. DESCRIPTION OF SAMPLES

All samples consisted of two separate components, base and accelerator, which were mixed immediately before use, in accordance with the manufacturer's directions.

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

The samples (in the cured state) comprised several colors, including white, neutral or natural, aluminum, gray, tan, brown, red, and 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. Identification of all samples are given in a separate list attached to this report.

3. PHYSICAL TESTS

3.1 Staining and color changes

3.1.1 Standard test procedure

The following test procedure (summarized version)[1/] was developed for predicting the staining and color change tendencies of polysulfide base joint sealers:

A mortar mixture consisting of 1 part white portland cement, 1/2 lime and 3 parts graded Ottawa sand (by volume) is placed over the entire surface of a thin aluminum plate having dimensions of 6- by 2 3/4 to a depth of 1/4 in. and struck off flat. This is accomplished with the aid of a rectangular brass frame, 1/4 in. thick with an opening having the same dimensions as the aluminum plate. After 4 hr curing of the mortar in air at 74 ± 2°F, 30 g of freshly mixed sealant (previously conditioned for 24 hr at 74 ± 2°F and 50 ± 10 percent humidity[2/] is spread over the mortar leaving a half inch margin of mortar uncovered by the sealant. Two such specimens are prepared.

After an exposure period in air at standard conditions, one specimen is exposed in an "Atlas Weatherometer"[3/] (single or double arc) for 200 hr and the other specimen is exposed in the laboratory at standard conditions for the same period. At the end of the exposure period, the specimens are examined for color changes and stains.

1/ The standard test procedures given in this report are condensed versions of the complete procedures and do not include some of the detailed requirements that will eventually be included in a purchase specification.

2/ Temperature of 74 ± 2°F and relative humidity of 50 ± 10 percent are referred to as "standard conditions" in this report.

3/ The Weatherometer was operated on 51-9 cycle, i.e. 51 min of light and 9 min of water and light.

3.1.2 Results

Table 1 summarizes the results obtained on 31 samples of calkings tested in accordance with the procedure described above. (The duration of exposure in the Weatherometer and laboratory was 500 hr instead of 200 hr. It was found later that the shorter exposure produced the same results, and was therefore included as such in the standard test).

Twenty five of the 31 samples exposed both at standard conditions and in the Weatherometer produced stains on the mortar backing. The results in table 1 indicate that 10 samples producing intense red stains after exposure in air at standard conditions caused reddish brown stains of slightly less intensity in the Weatherometer. The stains described in the table as slight were shades of yellow, tan or brown.

In regard to the color changes of the calkings after 500 hr in the Weatherometer, the whites, grays, neutrals and tans darkened to varying degrees. The aluminums and blacks were hardly affected. Changes in color after exposure in the laboratory at standard conditions were insignificant.

Figure 1 illustrates the types of staining obtained on 5 samples of joint sealers.

In actual installations polysulfide calkings with staining tendencies will produce stains of greater intensity (usually red) when exposed on the north side of a structure. When installed on the south side the stain usually is brownish red and the sunlight tends to lighten the stain as time passes. The Weatherometer test produces the effect of a southern exposure while the test made under standard conditions simulates conditions on the north side of a structure or an indoor exposure.

3.2 Slump or flow test

3.2.1 Standard test procedure

The following test procedure was developed for determining the slump tendencies of polysulfide base sealers, non-sag type:

The sample in the original container is first conditioned at standard temperature and humidity for 24 hr. At the end of this time a portion of the freshly mixed sample is placed in a clean aluminum trough 1/16 in. thick, 6 in. long, 3/4 in. wide, and 1/2 in. deep, with the back of the trough extending an additional 2 in. After the calking is struck flat on all exposed surfaces, the trough is immediately placed in a vertical position in an oven for 24 hr at 122 ± 2°F. At the end of this period the amount of slump is measured from the lower end of the trough to the lowest point assumed by the compound. Measurements are made to the nearest 1/16 in.

3.2.2 Results

Table 2 gives the results of the slump tests for each of the 24 samples tested in the standard trough (designated No. 2) and also in a trough 6 in. long, 3/8 in. wide and 3/4 in. deep (No. 1 trough).

Of the 24 samples tested only one slumped excessively (1/2 in. in both troughs). Twenty one samples showed no slump at all when tested in the No. 1 trough, and 17 samples did not slump when tested in the No. 2 trough.

Figure 2 illustrates the types of troughs used in the test as well as examples of slumping and non-slumping compounds.

As stated in the first progress report, slump in a polysulfide base sealer 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.

3.3 Tack-free time

3.3.1 Standard test procedure

The following standard test procedure has been established to determine the tack-free time of the polysulfide base calkings:

After conditioning the samples in the original container for 24 hr at standard temperature and humidity a portion of the mixed compound is spread on a thin aluminum plate over an area of approximately 5- by 1- by 1/4-in. thick. A template is used to aid in spreading the compound and also striking off flat the top surface. The specimen is then exposed in air at standard conditions for 72 hr.

At the end of the applicable tack-free time (72 hr) a 6- by 1-in. strip of polyethylene film, measuring 0.004 ± 0.002 in. in thickness is pressed on to the top of the calking for a period of 30 sec with a 30 g brass weight approximately 1.7- by 1- by 1/8-in. thick. The film is then progressively withdrawn at right angles to the compound. The sample is considered tack-free if the film pulls off without any calking adhering to it.

3.3.2 Results

The tack-free time test was applied to 22 polysulfide base compounds and the results are given in table 3. Of the 22 samples only 3 failed the test.

Compounds remaining tacky in installations for long periods of time tend to hold dirt and dust which results in an unsightly appearance.

3.4 Application life and ease of extrusion

3.4.1 Standard test procedure

3.4.1.1 Non-sag type

The following test procedure was developed to determine the application or pot life as well as the ease of extrusion of polysulfide base joint sealers:

A Brookfield Viscometer, Model RVF, fitted with a No. 7 spindle rotating at 2 r.p.m. is used to make the determinations. The sample in the original container is first conditioned for 24 hr at standard temperature and humidity. After mixing thoroughly about 300 g of base and accelerator, the compound is placed into a gill capacity can (with friction rim removed), slightly over filling the can. The calking is struck off flat with the edge of a spatula. The spindle is slowly brought down to the middle of the compound, and then lowered to the level of the standard immersion mark indicated on the spindle. Any voids around the spindle are filled in with the aid of a small spatula. The Viscometer motor is turned on and readings on the 0 to 100 scale are taken after 1 min (initial reading) and at periodic intervals thereafter. The motor is turned off after the initial reading, and turned on again one min before the following reading is taken. The test is stopped either at the end of 3 hr, or when the pointer reaches the end of the scale (100 mark), depending on which comes first.

3.4.1.2 Flow type

The test procedure described above for the non-sag type compounds is also used for the flow type except that the speed of the Viscometer motor is made to operate at 10 r.p.m. instead of 2 r.p.m. The size of the spindle remains unchanged. Application life tests were made on eight flow type compounds using the standard test procedure.

3.4.2 Results

The first progress report gave the results of application life tests of 29 samples of the non-sag type using the standard test procedure described above. Twelve of the 29 samples were relatively rapid curing compounds since the pointer reached the scale limit of 100 before 3 hr. For the remaining 17 samples the test ran the full 3 hr indicating relatively slow curing compounds.

The results of the tests for application life on eight flow type compounds are given in table 4. Five samples showed rapidly curing tendencies while the remaining three stayed soft for 3 hr.

A determination of the ease of extrusion of a compound can be made by placing a maximum limit on the initial scale reading in the standard application life test.

A series of tests were made to determine the feasibility of using an air calking gun (Semco brand) operated under a specific pressure, temperature and humidity for measuring application life. This was done by extruding 6 oz of a compound from a filled 12 oz cartridge at 50 lb pressure at standard conditions of temperature and humidity. After 3 hr, the remaining portion in the cartridge was extruded and the time required to empty it was noted.

The results of these tests indicated that the method was not as dependable as the Viscometer method. Several tests made on single samples gave variable results. Another disadvantage of the method was the relatively large amount of calking needed for each test.

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

3.5 Hardness

3.5.1 Standard test procedure

The instrument used for making the hardness determination is the Shore Durometer (Model A) described in Federal Test Method for Rubber No. 601 Method 3021 and also in ASTM Standard D 676-55T, 1958, part 9. All readings are taken by the instantaneous method.

The sample in the original container is first conditioned for 24 hr at standard conditions of temperature and humidity. After mixing thoroughly about 200 g of base and accelerator a portion of the mixture is placed into two polyethylene molds with inside dimensions of 4- by 1 1/2 by 1/4 in. The top surface of the compound is struck off with a spatula, and the two specimens are stored in the laboratory at standard conditions of temperature and humidity. When the compound has lost its tackiness (usually 2 days) it is removed from the mold, placed on a 3- by 6-in. thin aluminum plate and allowed to cure for an additional 12 days.

Immediately after the curing period hardness values are obtained on the two specimens at standard conditions. A total of five readings are taken on the two specimens and the average reading is regarded as the original value. One specimen is then exposed in a freezer at 0° ± 2°F for 72 hr and the other specimen in an oven at 158 ± 2°F for the same period. At the end of the cold and hot exposure periods hardness values are determined at the respective temperatures and also after a recovery period for 3 hr at standard temperature and humidity. The average of five readings on each specimen is the accepted value.

3.5.2 Results

Table 5 gives the hardness values obtained on 100 calking samples obtained at 74°F, 0°F, and 158°F. The recovery values after the cold and hot exposures are also given. The last column in the table lists "hardness factor" values which represent average values of the five corresponding values given in the first five columns of the table.

Figures 3 and 4 illustrate the distribution of hardness values for the three temperature conditions for all samples tested.

The data in figure 3 show that the average hardness value obtained at 0°F was 56 percent higher than the average at 74°F. On the other hand, the hot treatment (158°F) caused no immediate increase in hardness over the original value, but was followed by a residual increase of hardness, after recovery of 23.5 percent. (Fig. 4).

3.5.3 Effect of extended exposures on hardness

3.5.3.1 Exposure at 74°F

Seventeen samples of calkings were exposed in air at standard conditions for periods of 7, 8, and 9 months. Figure 5 shows the effects of the exposure on the hardness characteristics of the 17 samples.

Of the seven samples exposed for 9 months the increase in hardness ranged from 7 percent to a maximum of 61 percent. In the 8-month exposure group the increases ranged from 0 percent to 25 percent. In the 7-month exposure group three out of four samples from one producer showed no increase in hardness and the fourth increased 8 percent.

3.5.3.2 Combined exposure, 74 and 158°F

Five calking samples with original hardnesses ranging from 25 to 44 were exposed in air at standard conditions for 18 weeks, followed by exposure in an oven at 158°F for an additional 30 weeks. Figure 6 illustrates the effect of this treatment. The average increase in hardness for all specimens during the first 18 weeks was 36 percent. The subsequent 6 weeks exposure at 158°F raised the average increase in hardness to 94 percent. However, during the final 24 weeks the average hardness increased by only an additional 15 percent.

3.5.3.3 Effect of outdoor exposure on hardness

Fourteen calking samples were exposed on the roof of the Mineral Products Building for 16 months. Figure 7 shows the effect of the exposure on the hardness values. The hardness increases ranged from 0 percent to 147 percent with an average increase of 31 percent for all samples. Reference to figure 7 indicates that three samples (producer No. 6) showed no increase in hardness after exposure. It is worth noting that three other samples from the same producer likewise showed no increase in hardness after 7-months exposure at 74°F (see Fig. 5).

(For relationship between hardness and durability, see chapter on durability.)

3.6 Durability (Bond-Ductility)

Durability tests (referred to as bond-ductility in previous reports) were completed on the calking samples, using eight types of accessory materials as follows:

Concrete	-	93 samples tested
Concrete block	-	47 samples tested
Brick	-	53 samples tested
Wood	-	33 samples tested
Porcelainized steel	-	39 samples tested
Porcelainized aluminum	-	28 samples tested
Aluminum	-	14 samples tested
Glass	-	15 samples tested

3.6.1 Standard durability test procedure

The following test procedure was developed to predict the durability of polysulfide joint sealers in installations where joint movement take place:

A joint sealer stretching machine accommodating three specimens at one time is used to make the tests. The machine, with some changes in design, is similar to the one described in Fed. Spec. SS-R-406-C, Road and Paving Materials, Method 223.11. (See Progress Report No. 1 for Photo.)

The dimensions of the porous accessory blocks used in the testing of the joint sealers are 3- by 2- by 1-in. The dimensions of the non-porous materials are 3- by 2-in. with the thickness depending on the material available for the test.

The test joint is prepared by making a sandwich of two similar accessory blocks separated by two aluminum spacers and held together by a rubber band or clamp. After conditioning the sample for 24 hr at standard temperature and humidity, freshly mixed calking is placed between the spacers forming a filled joint 2- by 1- by 3/8-in. [4/]

Following a curing period of 14 days in air at standard conditions, the spacers are separated from the calking and the specimens are tested in accordance with the following cycle:

(a) Specimens are heated for 24 hr in an oven at 158 ± 2°F (with the aluminum spacers between the blocks).

(b) Specimens are allowed to cool to room temperature (74 ± 2°F).

(c) Specimens are immersed in distilled water for 7 hr.

(d) Specimens are surface dried and placed in a freezer for 8 to 16 hr.

(e) Specimens are placed (frozen) in the machine and extended at 0°F, 50 percent of the original width, at the rate of 1/8 in. per hr.

(f) The joint is held in the stretched position with 9/16 in. spacers for 3 hr at 0°F followed by 3 hr at 74°F.

4/ After making a considerable number of tests with other joint sizes, the 2- by 1- by 3/8 in. joint was found to be the most practical size for a standard test.

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

3.6.2 Criterion of failure in the durability test

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

Although most of the failures occurred as bond breaks, some failures were characterized by cohesion breaks in the calking, and by failure of the accessory material itself. (Fig. 8). A complete bond failure in a specimen was defined as a failure resulting in an 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 specimens showed complete bond or cohesion failure, the test was stopped regardless of the number of cycles completed. In all other cases the tests were continued through the five cycles.

The following formula for evaluating the durability of the calking samples was used:

Durability Factor, $D_1 = A\,(6-C)$

where A = bond or cohesion loss for each specimen in sq in.

C = number of cycles completed

For example, when the three specimens showed complete bond or cohesion failure after the first cycle, the Durability Factor was determined as follows:

$$D_1 = A\,(6-C) = 2\,(6-1) = 10$$

For 3 specimens D = 3 x 10 = 30

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

$$D_1 = A\,(6-C) = 2\,(6-4) = 4$$

For 3 specimens D = 4 x 3 = 12

5/ The test results described in this report are based on a five cycle treatment.

For report purposes performance ratings were assigned to the Durability Factors as follows:

0 to 5	=	"Excellent"
6 to 10	=	"Good"
11 to 15	=	"Fair"
16 to 20	=	"Poor"
21 to 30	=	"Very Poor"

3.6.3 Durability test results

3.6.3.1 Effect of accessory material on durability of calkings

Table 6 gives the averages of the durability factors obtained for all samples tested with eight types of accessory materials. Figures 9 and 10 show the distribution of the durability factors for the porous and non-porous materials, respectively.

In table 6 it is shown that the average durability factors for the porous accessory groups ranged from 11.2 for the concrete block to 12.8 for concrete, with the values for wood and brick in between. However, the results obtained on the non-porous materials showed wide differences in performance. Porcelainized steel gave the best performance and porcelainized aluminum, the poorest.

Reference to the data in figure 9 indicates that 39 percent of the concrete samples tested were rated "excellent," 20 percent "very poor," and the remaining samples distributed somewhat evenly from "good" to "poor."

The results in figure 10 indicate that the best performing material in the non-porous group was porcelainized steel with 72 percent of the samples rated "excellent" and 18 percent rated "very poor."

Figure 10 also indicates that the lowest durability rating of the non-porous group was obtained by the porcelainized aluminum samples with only 18 percent in the "excellent catagory" and 61 percent rated "very poor." The average value of glass and aluminum was approximately halfway between the porcelainized steel and the porcelainized aluminum.

3.6.3.2 Relationship between color and durability

In table 7 are listed the durability factors in relation to the various colors of the calkings. The results indicate that the best performing compounds were the tan, neutral and gray, while the black and the aluminum were the poorest.

3.6.3.3 Relationship between brand of calking and durability

In table 8 the durability factors are shown in relation to the producers of the various calking samples. The results indicate that producers 6 and 11 show the best performance ratings while producers 4, 13, and 23 show the poorest performance.

3.6.3.4 Relationship between hardness and durability

A considerable amount of experimental data has been accumulated during the investigation with the purpose of establishing a correlation between hardness of the polysulfide calkings and durability.

Figure 11 illustrates the correlation obtained between original hardness values and durability factors for 91 calking samples tested in concrete joints. In the sample group with the lowest hardness (10 to 21) only two of the 15 samples tested had "very poor" ratings. The group with highest hardness range consisted of six samples all with durability factors in the "very poor" catagory.

In figure 12, a correlation similar to that shown in figure 11 was obtained between hardness factor and durability. Hardness factor is defined as the arithmetical average of five hardness values, i.e. 74°F, 158°F, 0°F, recovery from 158°F and recovery from 0°F.

3.6.3.5 Bubble formation in durability specimens

It was noted that during the cycling of the specimens in the durability test there was a tendency for many calking samples to form bubbles in the joints. The bubbles formed mainly in the calkings in contact with porous accessory materials. In a typical case, the bubbles would first appear after the first or second heating in the cycle, increase in size during the water immersion, and become still larger during the subsequent cycles. Figure 13 illustrates typical bubble formations observed in polysulfide compounds in concrete joints.

In rating the performance of test specimens with bubble formation, the presence of the bubble was not taken into account if the latter was closed. As a result there was a tendency for specimens with closed bubbles to have high performance ratings since the presence of the bubble allowed the calking to be stretched with less than normal stress on the bond.

3.6.3.5.1 Bubble formation and hardness

Of the 93 samples tested for durability in concrete joints, 26 samples formed bubbles in the joints. A study of the hardness characteristics of the 26 samples revealed that the average original hardness value of the group was 25, or 26 percent less than the average for all specimens tested.

3.7 Outdoor exposure tests of calkings in concrete joints

During the course of the investigation of the polysulfide base joint sealers as many samples as possible were exposed to the weather for durability studies. To date 143 samples have been exposed up to periods of 21 months.

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. At first the calkings were placed in primed and unprimed joints. But after about a year's observations results indicated that slightly more failures occurred in primed than unprimed joints. It was then decided to expose new samples in unprimed joints only. Each joint was stretched 1/32 in. every 3 months during the exposure.

3.7.1 Results

Table 9 gives the results of observations and tests made on 71 polysulfide base calkings exposed to the weather for 21 months. The joints were rated "good," "fair," or poor." From the data presented in table 9 it was found that 51 percent of the samples in the primed joints and 54 percent of the samples in the unprimed joints received "good" ratings. Also indicated is the fact that 38 percent of the calkings in the primed joints and 32 percent in the unprimed joints were rated "poor." Twenty-one of the 143 samples exposed developed bubbles in the joints in varying degress. (Fig. 14). Original hardnesses which had been obtained on 12 of the 21 samples averaged 27. This supports the previous finding that bubbles tend to form in the compounds of low hardness.

3.7.2 Relationship between durability factor and outdoor exposure test results

An attempt was made to correlate durability factors (concrete joints) with ratings obtained in the exposure tests of 23 samples. Seventeen samples had been exposed for 21 months and 6 samples for one year.

The results of this comparison are given in table 10. Of the 23 samples tested, 17 samples showed "good" correlation, two showed "fair" and four, "poor" correlation.

Table 1. Staining and color change properties of 31 polysulfide base calkings.

Sample No.	Color (mixed compound)	Stain on Mortar After 500 hr in Lab. 74°F	Stain on Mortar After 500 hr in Weatherometer	Calking Color Change After 500 hr in Weatherometer
1-I	Tan	Red	Red-brown	Very light gray
3-M	Gray	None	None	Dark gray
4-G	Aluminum	Red	Red-brown	None
4-H	Gray	Red	Red-brown	Dark gray
4-J	White	None	None	Ivory
6-Q	White	None	None	Dark ivory
6-R	Aluminum	Slight yellow	Slight yellow	None
6-S	Neutral	Slight tan	Slight tan	Dark brown
6-T	Neutral	Slight yellow	Slight yellow	Dark brown
6-U	Black	Slight brown	Slight brown	None
6-V	Aluminum	Slight brown	Slight brown	None
6-W	Neutral	Slight yellow	Slight yellow	Dark brown
6-X	Neutral	Slight yellow	Slight brown	Dark brown
6-Y	Black	Slight brown	Slight brown	None
6-Z	Gray	None	Slight yellow	Almost black
7-B	Black	Red	Red-brown	None
7-C	Black	Red	Red-brown	None
8-F	Aluminum	Red	Red-brown	None
8-G	Gray	Red	Red-brown	Dark gray
8-H	Black	Red	Red-brown	None
8-I	Black	Slight yellow	Red-brown	None
8-J	Aluminum	Red	Red-brown	None
13-K	Tan	Red	Red-brown	Dark brown
14-K	Gray	None	None	Dark gray
22-B	Gray	Slight brown	Slight brown	Dark gray
22-C	Black	Slight brown	Slight brown	None
22-D	Tan	Slight brown	Slight brown	Brown
23-A	Neutral	Slight brown	Slight brown	Light brown
23-B	Aluminum	Slight brown	Slight brown	None
23-D	Neutral	Slight brown	Slight brown	Dark brown
23-F	Black	None	None	None

Table 2. Slump values of 24 samples of polysulfide base calkings.

Sample No.	Trough #1 3/8 in. wide, 3/4 in. deep	Trough #2 3/4 in. wide, 1/2 in. deep
	in.	in.
1-I	0	0
3-M	0	3/16
4-I	1/2	1/2
6-R	0	1/4
6-S	0	0
6-U	0	0
6-V	3/16	1/4
6-W	0	0
6-X	0	0
6-Y	0	0
6-Z	0	3/16
6-AA	0	0
7-B	1/16	1/16
8-F	0	0
8-G	0	0
8-H	0	0
8-I	0	1/16
8-J	0	0
14-D	0	0
14-E	0	0
14-F	0	0
14-J	0	0
14-K	0	0
14-L	0	0

Table 3. Results of Tack-Free time tests on 22 polysulfide base calkings.

Sample No.	Tack-Free, 72 hr test
4-D	Passed
4-I	Passed
6-D	Failed
6-Q	Failed
6-S	Passed
6-X	Passed
7-B	Passed
8-I	Passed
8-K	Passed
9-B	Passed
10-F	Passed
11-H	Passed
13-E	Failed
13-F	Passed
13-K	Passed
14-K	Passed
14-L	Passed
15-D	Passed
15-E	Passed
21-D	Passed
21-E	Passed
23-A	Passed

Table 4. Application life tests of polysulfide base calkings using the Brookfield Viscometer. (flow type compounds)

Sample No.	Viscometer Readings (0 - 100)		Time
	Minimum	Maximum	
6-G	6.0	100	50 min
6-T	6.0	100	90 min
9-F	27.5	100	10 min
12-D	26.5	62.8	3 hr
14-H	5.5	9.5	3 hr
14-I	6.5	13.3	3 hr
23-F	24.5	100	40 min
23-E	82.5	100	2 hr

Table 5. Shore "A" Hardness Values of 100 polysulfide base calkings.

Sample No.	Orig. value 74°F	0°F 72 hr	Recovery from 0°F	158°F 72 hr	Recovery from 158°F	Hardness factor *
1-F	32	51	40	32	40	39
1-G	32	52	36	32	44	39
1-H	34	59	36	34	42	41
1-I	33	51	29	20	37	34
2-D	38	55	40	40	48	44
2-E	42	55	41	42	47	45
2-F	38	50	40	32	39	40
2-G	34	51	35	38	43	40
3-E	32	49	32	24	41	36
3-F	33	53	34	35	40	39
3-G	46	56	41	44	43	46
3-H	33	51	34	41	46	41
3-I	30	61	33	38	48	42
3-J	38	56	36	39	46	43
3-K	31	56	34	36	46	41
3-L	25	48	26	29	42	34
3-M	18	46	19	11	14	22
4-G	39	64	46	40	51	48
4-H	22	56	35	39	46	40
4-I	44	60	43	49	51	49
4-J	32	63	29	28	44	39
6-A	15	40	16	7	16	20
6-H	20	37	20	15	24	23
6-J	16	34	22	12	16	20
6-K	16	43	19	12	20	22
6-L	16	53	18	15	22	25
6-M	35	58	34	27	37	38
6-N	14	41	16	12	18	20
6-O	21	41	21	15	24	24
6-P	12	34	12	6	14	16
6-Q	34	56	33	27	37	37
6-R	24	47	24	19	28	28
6-S	24	46	24	20	26	28

* Average of 5 hardness readings i.e., 74°F, 0°F, 158°F, recovery after 0°F, and recovery after 158°F.

Table 5 (Continued)

Sample No.	Orig. value 74°F	0°F 72 hr	Recovery from 0°F	158°F 72 hr	Recovery from 158°F	Hardness factor *
6-T	19	36	19	12	20	21
6-U	20	46	20	13	23	24
6-V	54	62	57	56	63	58
6-W	45	67	43	41	52	43
6-X	35	62	36	35	44	42
6-Y	38	54	36	39	45	42
6-Z	18	46	16	17	23	24
7-A	49	60	48	48	56	52
7-B	50	68	53	58	63	58
7-C	32	53	35	32	42	39
8-A	10	37	10	12	24	19
8-B	27	52	29	34	48	38
8-D	16	44	13	15	21	22
8-E	47	61	47	53	54	52
8-F	32	50	32	39	49	40
8-G	28	56	35	29	40	38
8-H	26	53	30	32	44	37
8-I	50	61	50	53	57	54
8-J	29	52	33	31	47	38
8-K	21	52	21	20	33	29
9-A	49	59	52	50	53	53
9-B	45	56	44	45	50	48
9-C	50	60	49	52	54	53
9-F	55	59	52	56	58	56
10-A	36	57	36	36	42	41
10-B	58	67	57	58	62	60
10-D	57	63	64	60	60	61
11-A	33	54	34	47	53	44
11-E	40	58	37	46	51	46
11-F	17	46	16	32	40	30
11-G	41	57	41	43	52	47
11-H	44	60	46	50	58	52

Table 5 (Continued)

Sample No.	Orig. value 74°F	0°F 72 hr	Recovery from 0°F	158°F 72 hr	Recovery from 158°F	Hardness factor *
12-A	42	62	44	46	54	50
12-B	43	57	41	43	48	46
12-C	42	62	41	44	52	48
12-E	41	54	42	44	50	46
12-F	39	56	37	40	47	44
12-G	28	52	32	35	46	39
12-H	45	60	46	48	57	51
13-F	36	54	36	39	45	42
13-H	24	54	26	30	43	35
13-I	21	42	20	28	36	29
13-J	24	44	26	33	43	34
14-A	36	56	36	31	41	40
14-D	23	56	26	16	29	30
14-E	34	65	32	26	47	41
14-F	29	61	30	21	38	36
14-G	17	26	19	25	24	22
14-J	32	63	29	28	44	39
14-K	24	56	22	20	24	29
14-L	28	44	29	26	28	31
15-B	46	57	46	48	51	50
15-E	41	54	52	46	40	47
15-F	16	42	15	26	36	27
21-A	23	51	27	38	33	49
21-B	36	54	36	43	52	44
21-C	34	61	35	35	52	43
22-A	33	56	31	26	41	37
22-B	26	51	21	26	34	32
22-C	47	66	49	55	60	55
22-D	30	46	30	23	33	32

Table 5 (Continued)

Sample No.	Orig. value 74°F	0°F 72 hr	Recovery from 0°F	158°F 72 hr	Recovery from 158°F	Hardness factor *
23-A	40	56	43	43	51	47
23-B	46	61	46	45	59	51
23-C	58	67	60	59	62	61
23-D	25	51	25	18	31	30
23-E	33	56	35	32	42	40
23-F	51	61	51	50	55	54

Table 6. Summary of Results of Durability Test on polysulfide base calkings.

Accessory material	No. of samples tested	Durability Factor (avg.)
Concrete*	93	12.8
Brick	53	12.0
Wood	33	12.4
Concrete block	47	11.2
Porcelainized steel	39	9.4
Porcelainized aluminum	28	22.1
Aluminum	14	19.6
Glass	15	14.4

* All accessory materials except wood were tested without a primer.

Table 7. Relationship of color to durability of calkings.

Color of calking	No. of samples tested	Durability factor (average)
Aluminum	21	14.7
Tan or neutral	22	8.5
Gray	18	9.2
Black	29	15.6
White	3	12.4

Table 8. Relationship of brand of calking to durability.

Producer No.	No. of samples tested *	Durability factor (average)
1	4	11.7
2	4	10.3
3	6	12.6
4	4	19.9
6	18	5.7
8	9	10.7
11	5	5.4
12	5	8.4
13	4	18.9
14	9	12.7
22	4	7.9
23	6	19.4

*Tested in concrete joints

Table 9. Results of 21 months exposure to the weather of 71 samples of polysulfide base calkings in concrete joints.[1/]

Sample No.	Color	Condition of Joints [2/]	
		Primed	Unprimed
1-A	Neutral	Good	Poor
1-B	Aluminum	Poor [3/]	Poor [3/]
1-C	Brown	Good	Good
1-D	Gray	Good	Good
1-E	Black	Good [4/]	Poor (leaked) [4/]
2-A	Aluminum	Good [4/]	Good [4/]
2-B	Gray	Fair	Good
2-C	Tan	Good	Good
2-D	Black	Poor	Good
3-A	Aluminum	Poor [3,4/]	Poor [3,4/]
3-B	Gray	Fair	Good
3-C	Tan	Good	Good
3-D	Black	Good [5/]	Good [5/]
4-A [7/]	Gray	Good	Good
4-B [8/]	Gray	Good	Good
4-C	Aluminum	Poor (leaked)[4/]	Fair [4/]
4-D	White	Poor	Poor (leaked)
4-E	Black	Good	Good
4-F	Red	Good [5/]	Good [5/]
5-A	Tan	Fair	Fair [3/]
5-B	Gray	Fair [5/]	Poor (leaked)
5-C	Aluminum	Poor	Fair
5-D	Black	Poor	Fair [3/]
6-A	Neutral	Good	Good
6-B [6/]	Neutral	Poor (leaked)	Good
6-C	Gray	Poor (leaked)	Fair
6-D [6/]	Gray	Fair [5/]	Good
6-E	Aluminum	Poor	Poor
6-F	Black	Poor	Good
6-G [6/]	Black	Good	Good
7-A	Black	Poor (leaked)	Poor (leaked)

Table 9 (Continued)

Sample No.	Color	Condition of Joints 2/	
		Primed	Unprimed
8-A 7/	Aluminum	Good	Good
8-B 8/	Aluminum	Poor 3,4/	Poor 3,4/
8-C 7/	Brown	Good	Good
8-D 7/	Black	Good 5/	Good 5/
8-E 8/	Black	Poor (leaked) 4/	Poor (leaked) 4/
9-A	Aluminum	Fair 4/	Fair 4/
9-B	Tan	Poor (leaked)	Poor (leaked)
9-C	Black	Poor (leaked)	Poor (leaked)
9-D 6/	Aluminum	Poor	Fair
9-E 6/	Tan	Poor	Poor (leaked)
9-F 6/	Black	Poor (leaked)	Poor (leaked)
10-A	Gray	Good	Poor
10-B	Gray	Poor (leaked)	Poor (leaked)
10-C	Aluminum	Good 4/	Good 4/
10-D	Black	Poor (leaked)	Poor (leaked)
10-E 6/	Black	Good	Good
10-F	Gray	Poor (leaked)	Good
11-A	Gray	Poor	Poor
11-B 6/	Gray	Good	Good
11-C	Tan	Good	Good
11-D	Black	Good	Good
12-A	Gray	Good	Good
12-B	Tan	Good	Good
12-C	Black	Good	Good
12-D 6/	Black	Poor (leaked)	Fair
13-A	Tan	Poor (leaked)	Fair
13-B	Gray	Fair	Poor (leaked)
13-C	White	Good	Poor
13-D	Aluminum	Good	Good
13-E	Tan	Good 5/	Good
13-F	Black	Good	Fair

Table 9 (Continued)

Sample No.	Color	Condition of Joints 2/	
		Primed	Unprimed
14-A	White	Poor (leaked)	Poor (leaked)
14-B	Gray	Good 5/	Good 5/
14-C	Black	Fair	Poor]
15-A	Aluminum	Good	Good
15-B	Aluminum	Poor (leaked)	Poor (leaked)
15-C	Black	Good	Good
15-D	Black	Good	Good
16-A	Aluminum	Good	Good
16-B	Black	Good	Good

1/ Calkings are non-sag type, except where specified in a footnote.

2/ Joints were stretched 1/32 in. at periodic intervals to a total of 7/32 in. or 58%. Condition of joints were rated as Good, Fair or Poor. Good: no bond or cohesion breaks present, or contains shallow break no greater than 1/2 in. Fair: shallow bond or cohesion breaks totaling no more than 1 in. in length. Poor: bond or cohesion breaks totaling more than 1 in. in length.

3/ Cohesion breaks.

4/ Crazing of surface.

5/ Air bubbles in compound.

6/ Flow type compound.

7/ Compound classed as soft by producer.

8/ Compound classed as hard by producer.

Table 10. Relationship between durability factors and exposure test results for 23 samples. 1/

Sample No.	Durability factor	Outdoor Exposure: Bond or cohesion breaks	Correlation
2-C	20	Absent	Poor
3-I	24	Present	Good
3-K	12	Absent	Good
3-L	4.8	Absent	Good
6-M	30	Present	Good
6-N	0.3	Absent	Good
6-O	0.2	Absent	Good
7-A	30	Present	Good
8-A	8.0	Absent	Good
8-B	13.1	Present	Fair
8-D	0	Absent	Good
8-E	30	Present	Good
9-A	30	Present	Good
9-B	30	Present	Good
9-E	16.9	Present	Fair
9-F	27.5	Present	Good
10-A	2.6	Present	Poor
10-B	30	Present	Good
10-D	25	Present	Good
11-A	3.6	Present	Poor
12-A	4.6	Absent	Good
13-F	26	Absent	Poor
15-B	30	Present	Good

1/ Durability Factors were obtained with a sandwich of two concrete accessory blocks containing a 2- by 1- by 3/8-in. joint, subjected to heating and cooling cycles and stretched 50 percent at 0°F. The Exposure Tests were made with concrete troughs containing 11- by 3/4- by 3/8-in. joints exposed to the weather and stretched 1/32 in. every 3 months.

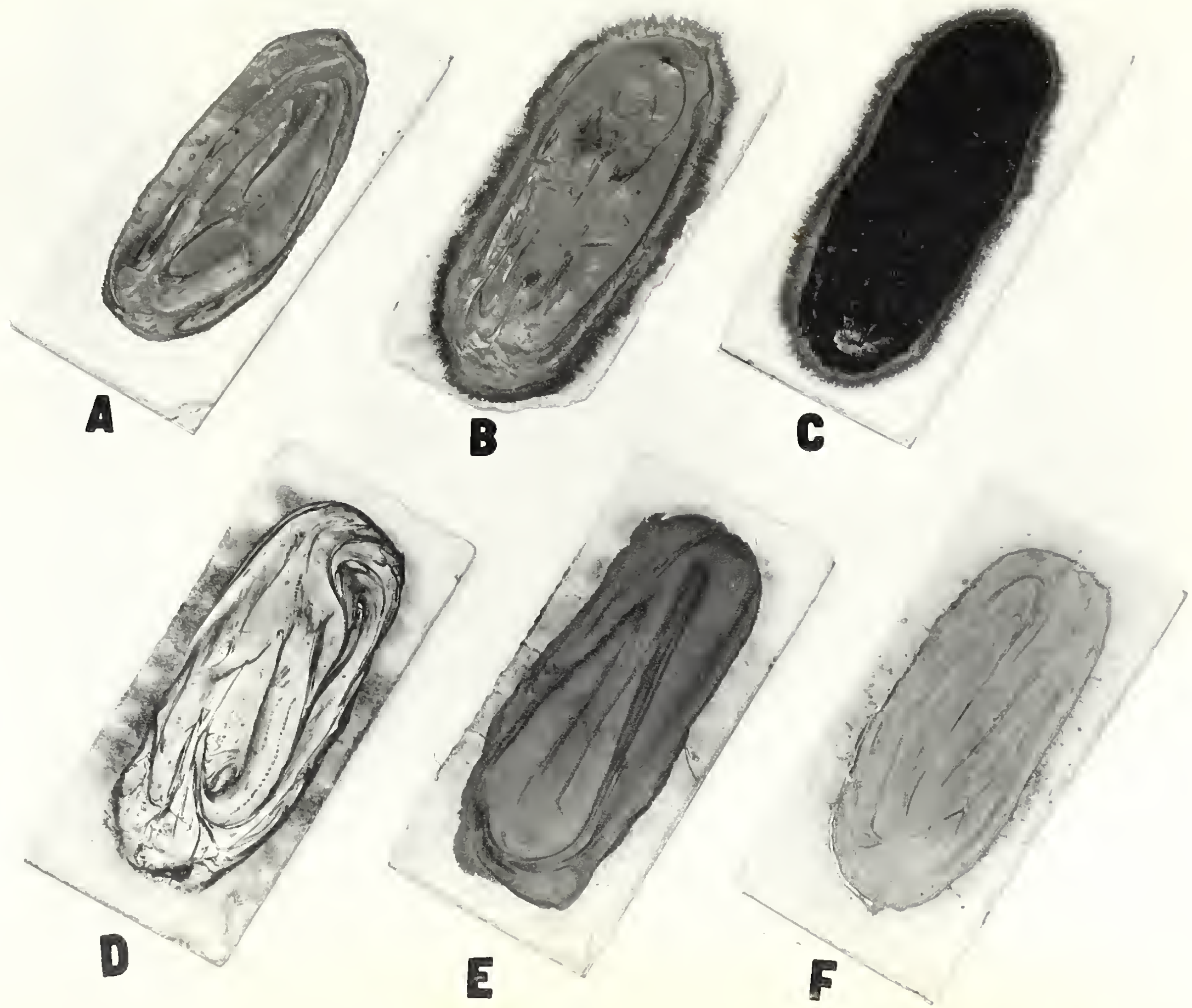

Fig. 1 - Staining tendencies of some polysulfide joint sealers. (A), Non-staining type - no stain appeared after 500 hr in Weatherometer. (B, C), Specimens with deep red stains after 500 hr in lab, 74°F. (D, E, F), Various degrees of reddish brown staining after 500 hr in Weatherometer.

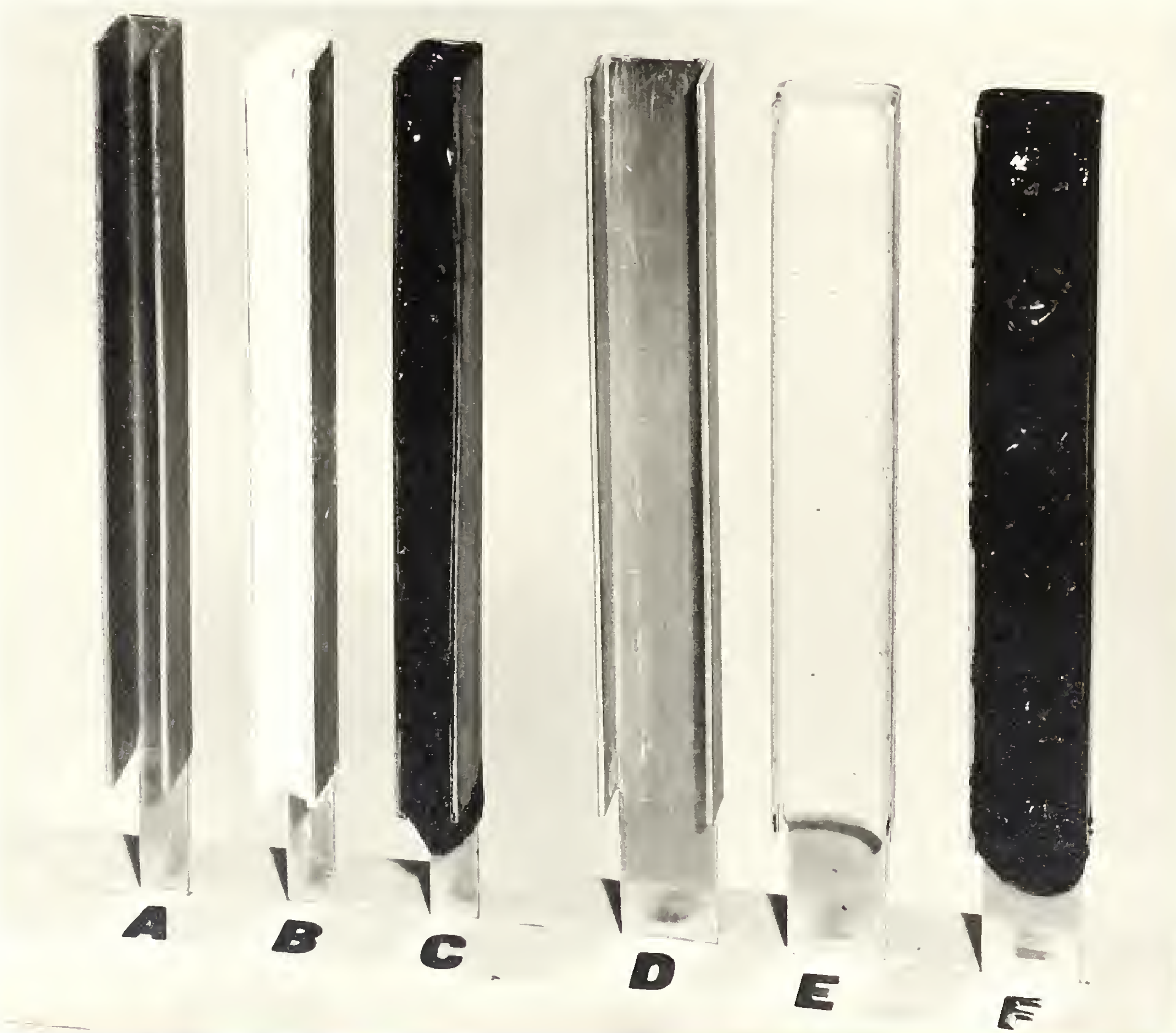

Fig. 2 - Two types of troughs used to test for slump of polysulfide calkings. (A, D), Troughs before filling. (B, E), Non-slumping compounds. (C, F), Samples showing 1/4 in. slump.

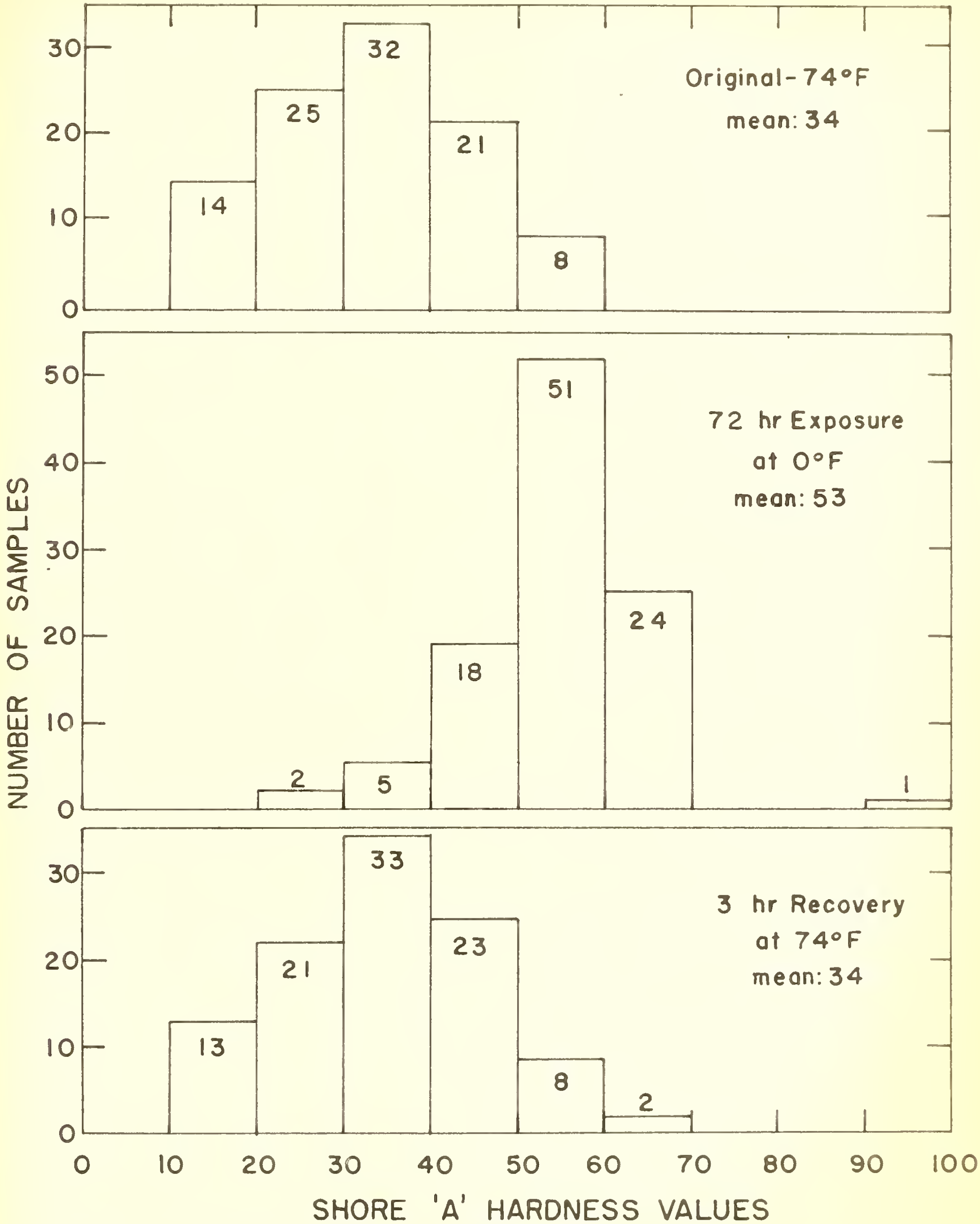

FIG. 3 DISTRIBUTION OF SHORE 'A' HARDNESS VALUES OBTAINED ON 100 SPECIMENS OF RUBBER BASE CALKINGS

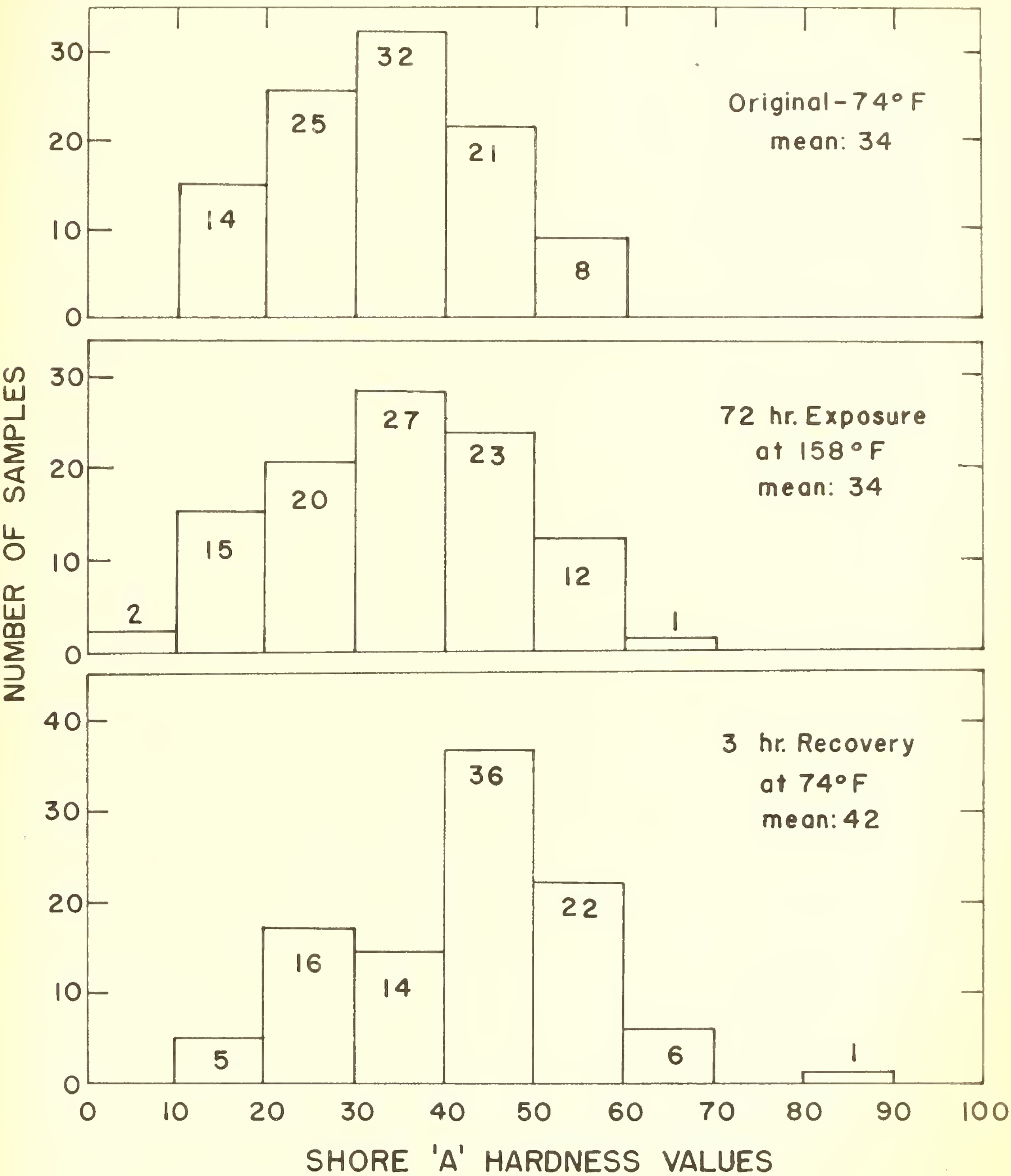

FIG. 4 DISTRIBUTION OF SHORE 'A' HARDNESS VALUES OBTAINED ON 100 SPECIMENS OF RUBBER BASE CALKINGS.

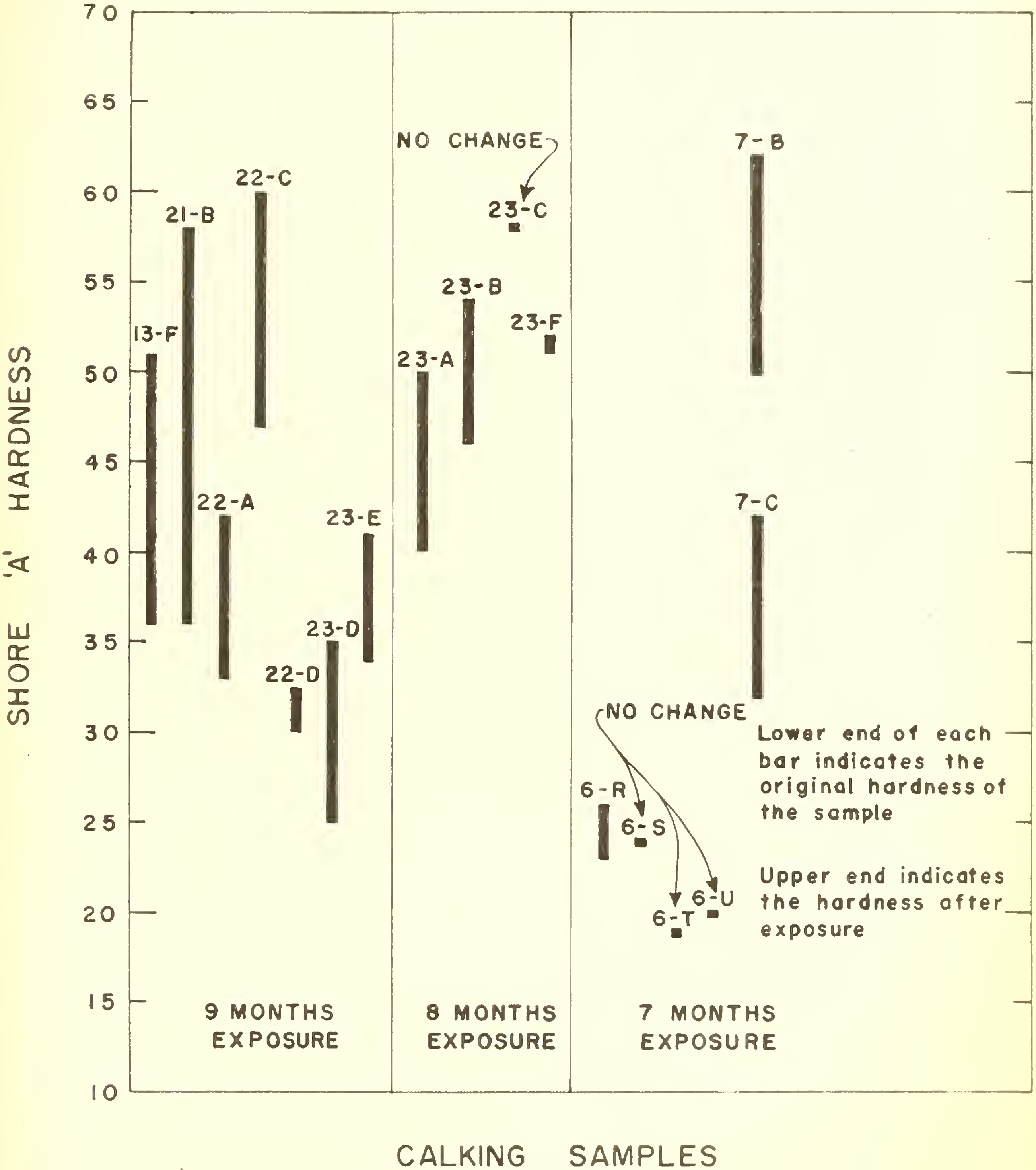

FIG. 5 EFFECT OF LABORATORY EXPOSURE AT CONSTANT TEMPERATURE (74 ± 2 °F) ON THE HARDNESS VALUES OF 17 CALKING SAMPLES

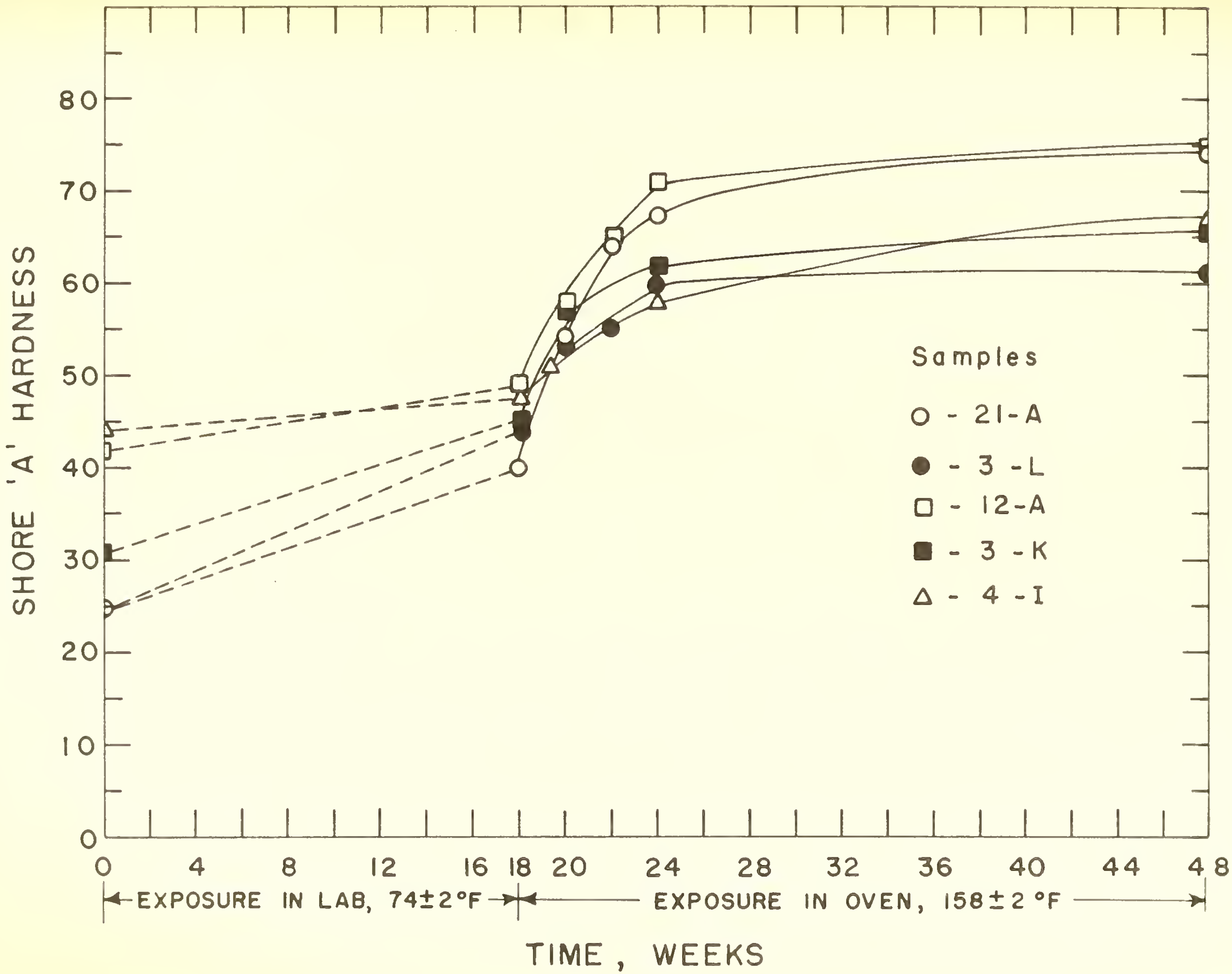

FIG. 6 EFFECT OF EXPOSURE AT 74 °F AND SUBSEQUENT EXPOSURE AT 158 °F ON THE SHORE HARDNESS VALUES OF 5 CALKING SAMPLES

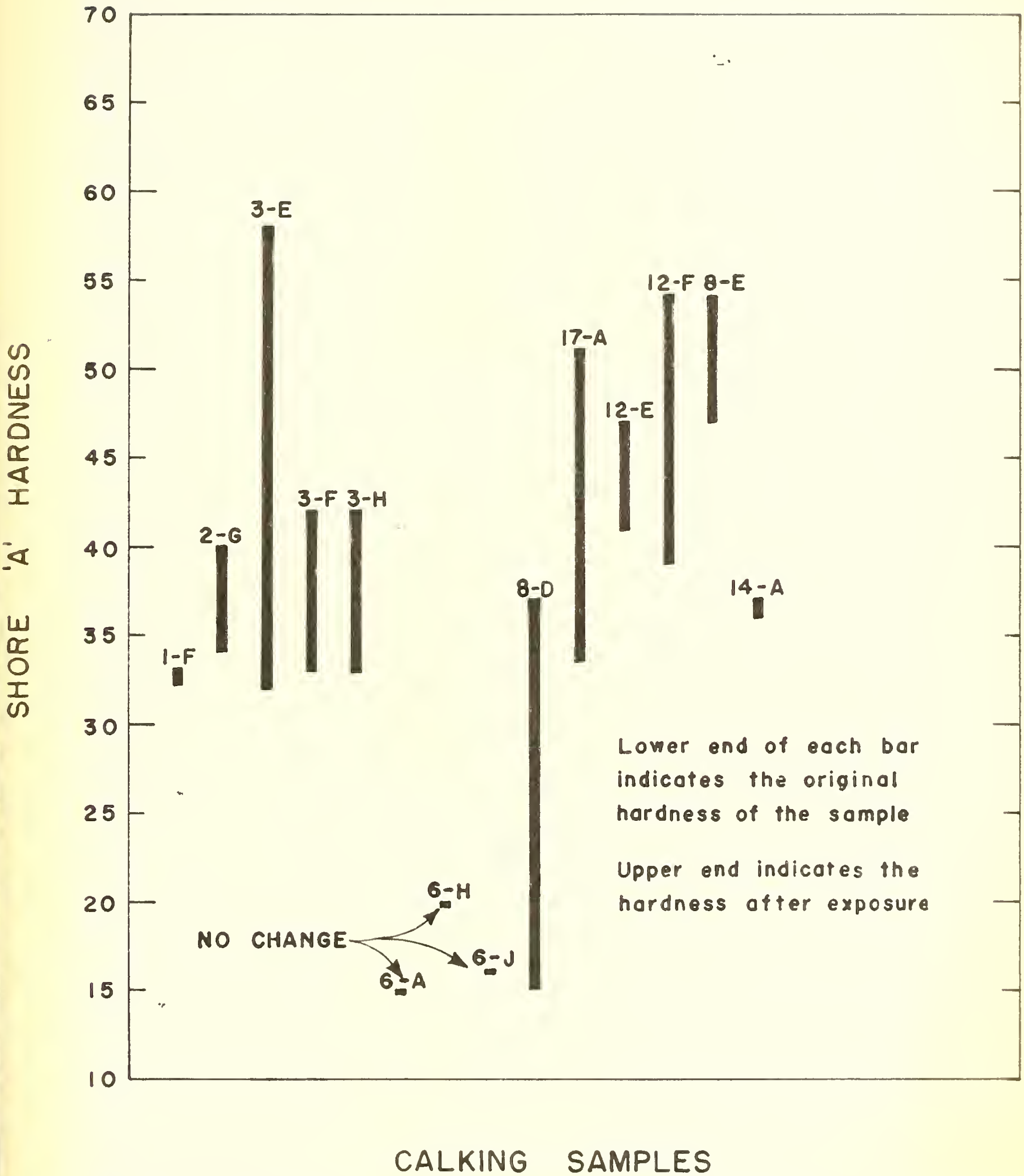

FIG. 7 EFFECT OF 16 MONTHS OF OUTDOOR EXPOSURE ON THE SHORE HARDNESS VALUES OF 14 CALKING SAMPLES

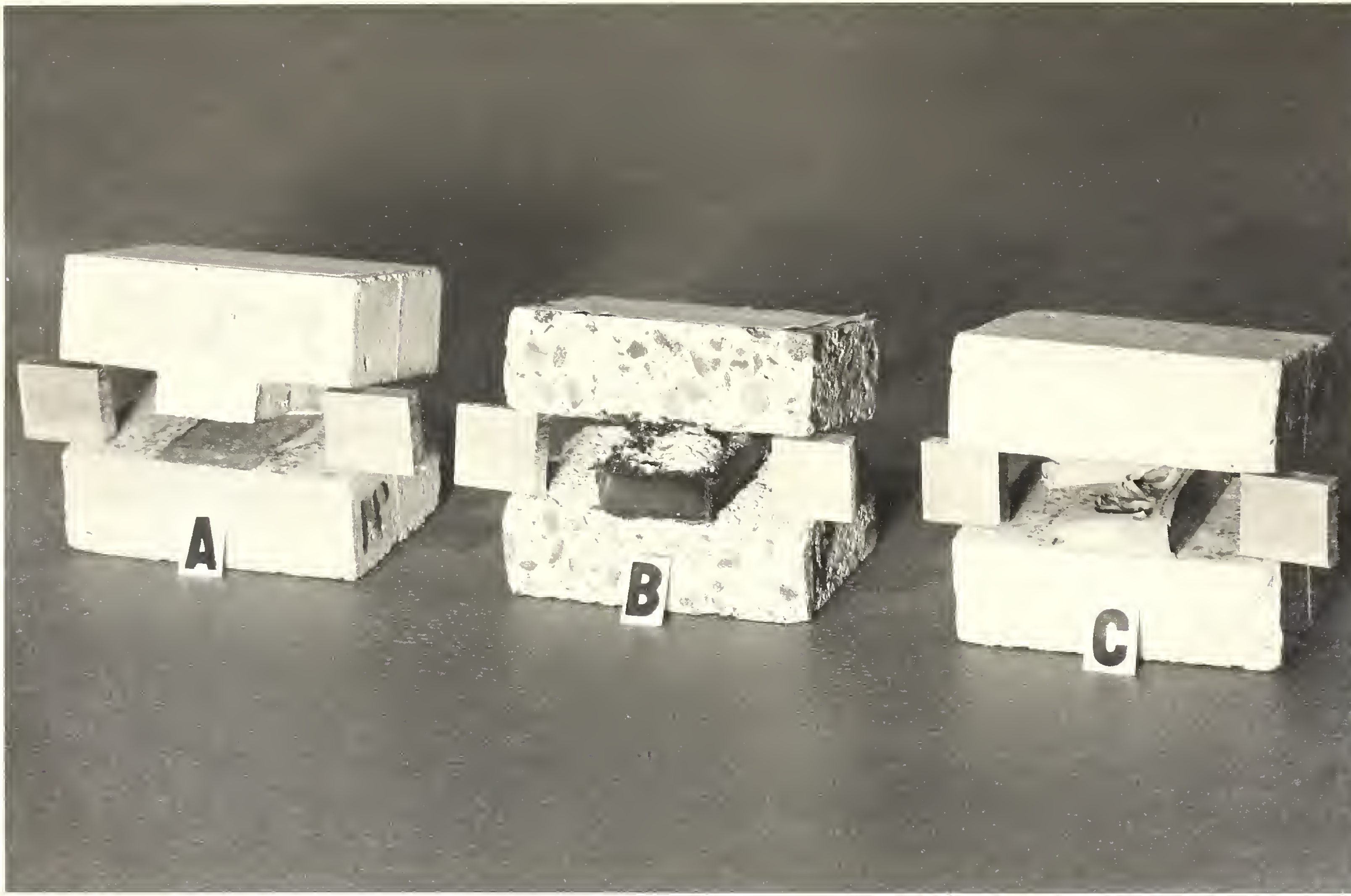

Fig. 8 - Types of failures obtained in the durability test. (A), Complete bond break between accessory material and calking. (B), Failure resulting in breakage of the accessory block due to high hardness and strong adhesion of calking. (C) Cohesion failure of calking.

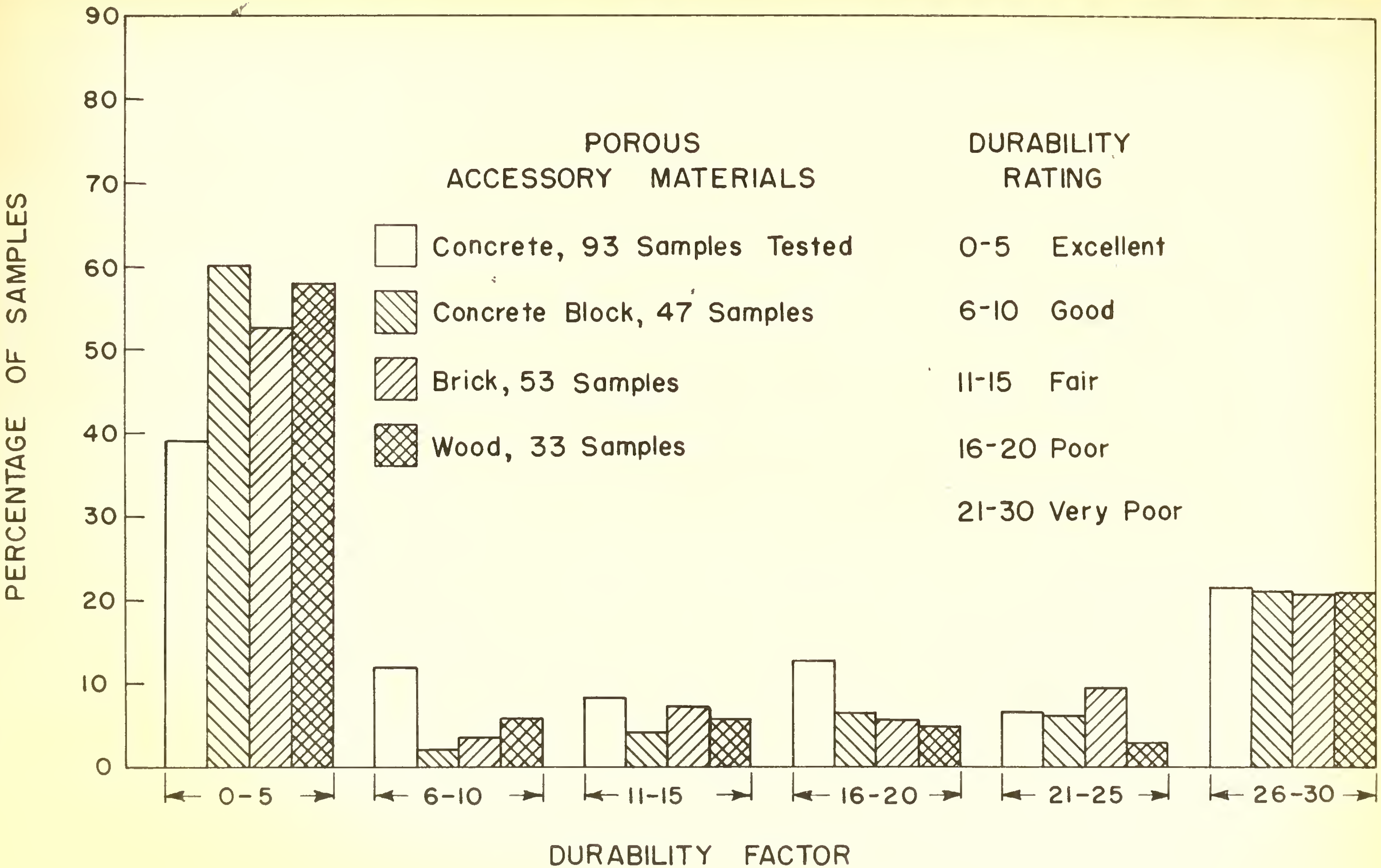

FIG. 9 Distribution of durability factor values obtained on calking samples tested with porous accessory materials

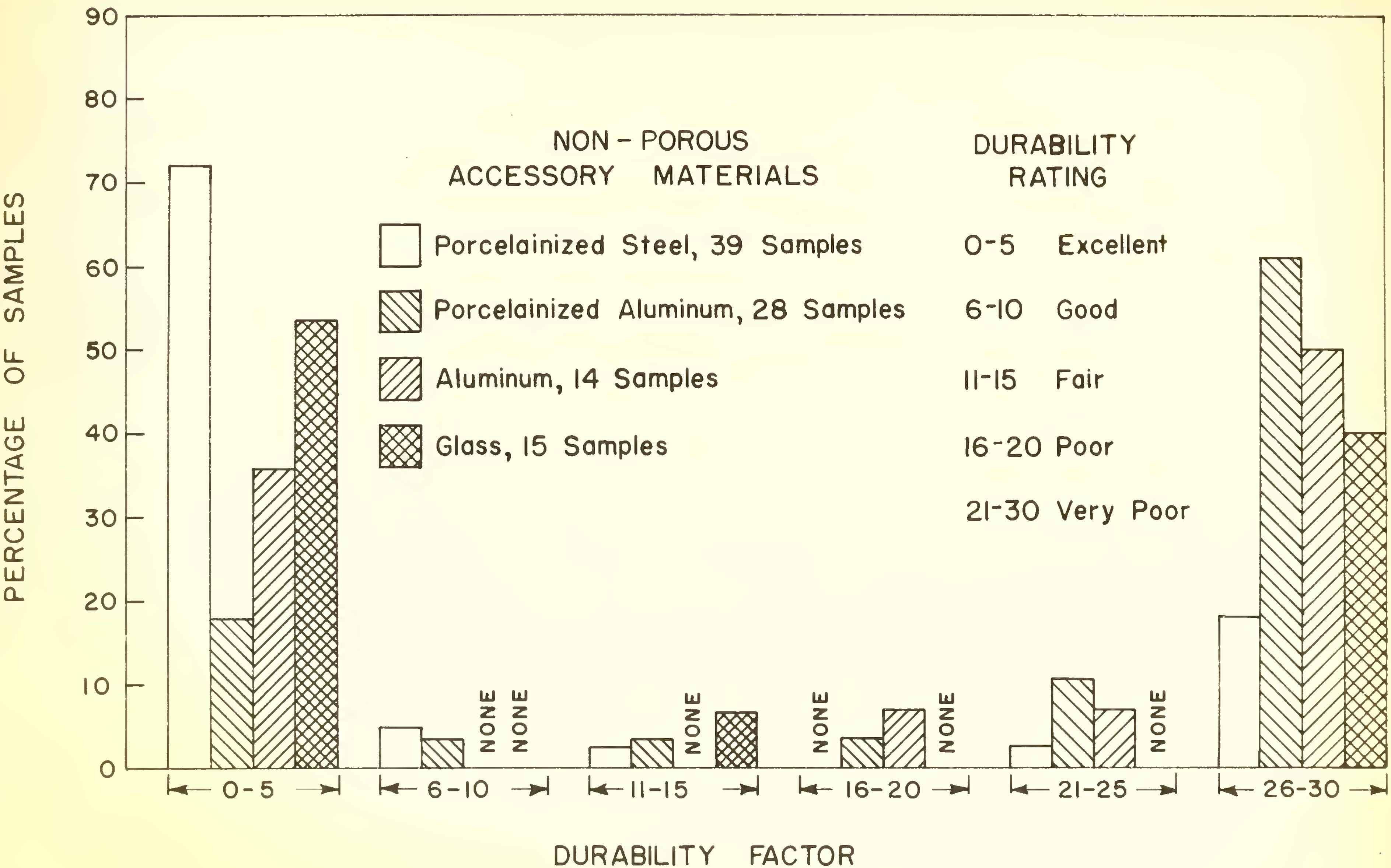

FIG. 10 Distribution of durability factor values obtained on calking samples tested with non-porous accessory materials

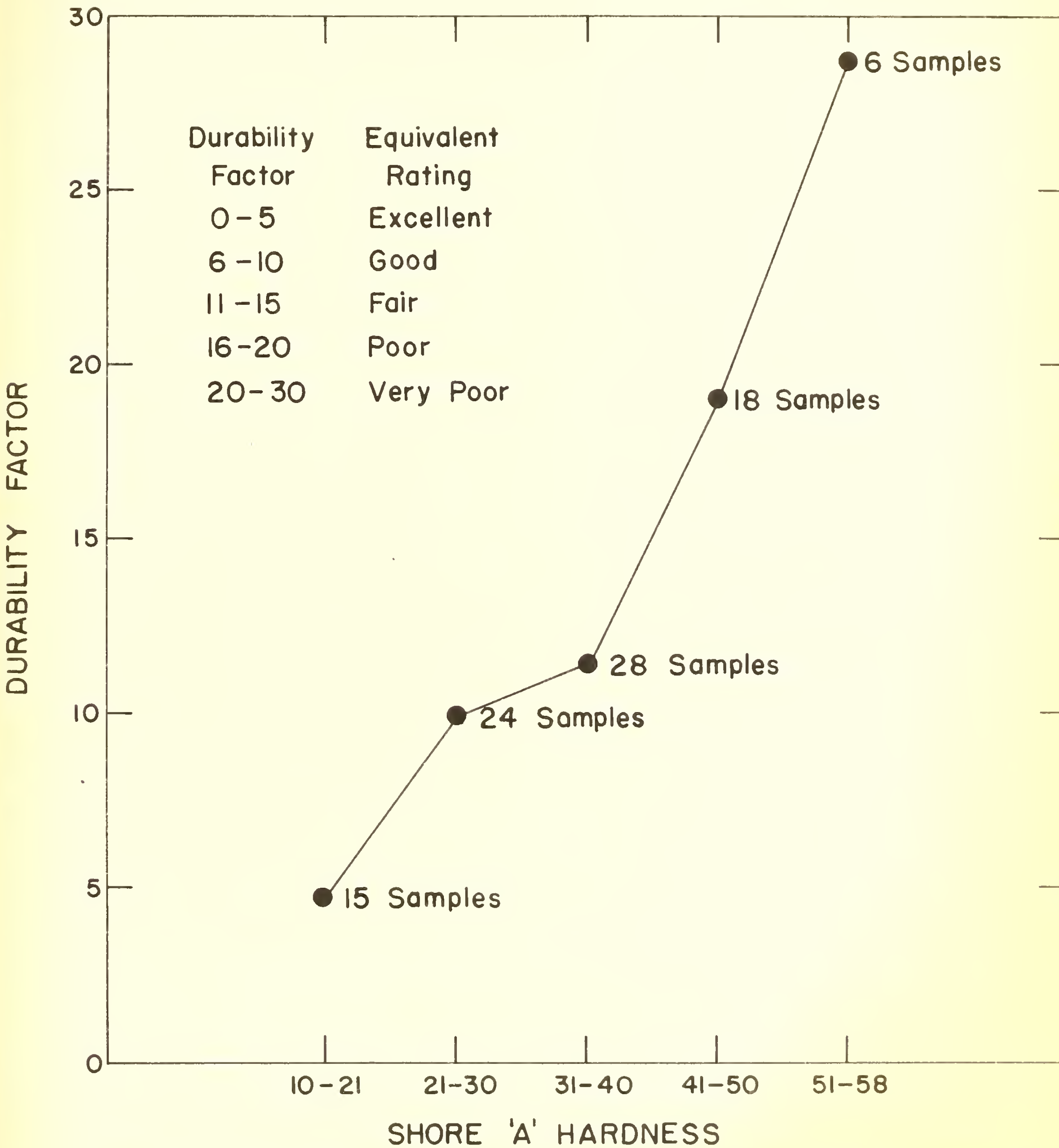

FIG. 11 Relationship between original shore hardness values and durability factor for 91 samples of polysulfide joint sealers tested in concrete joints

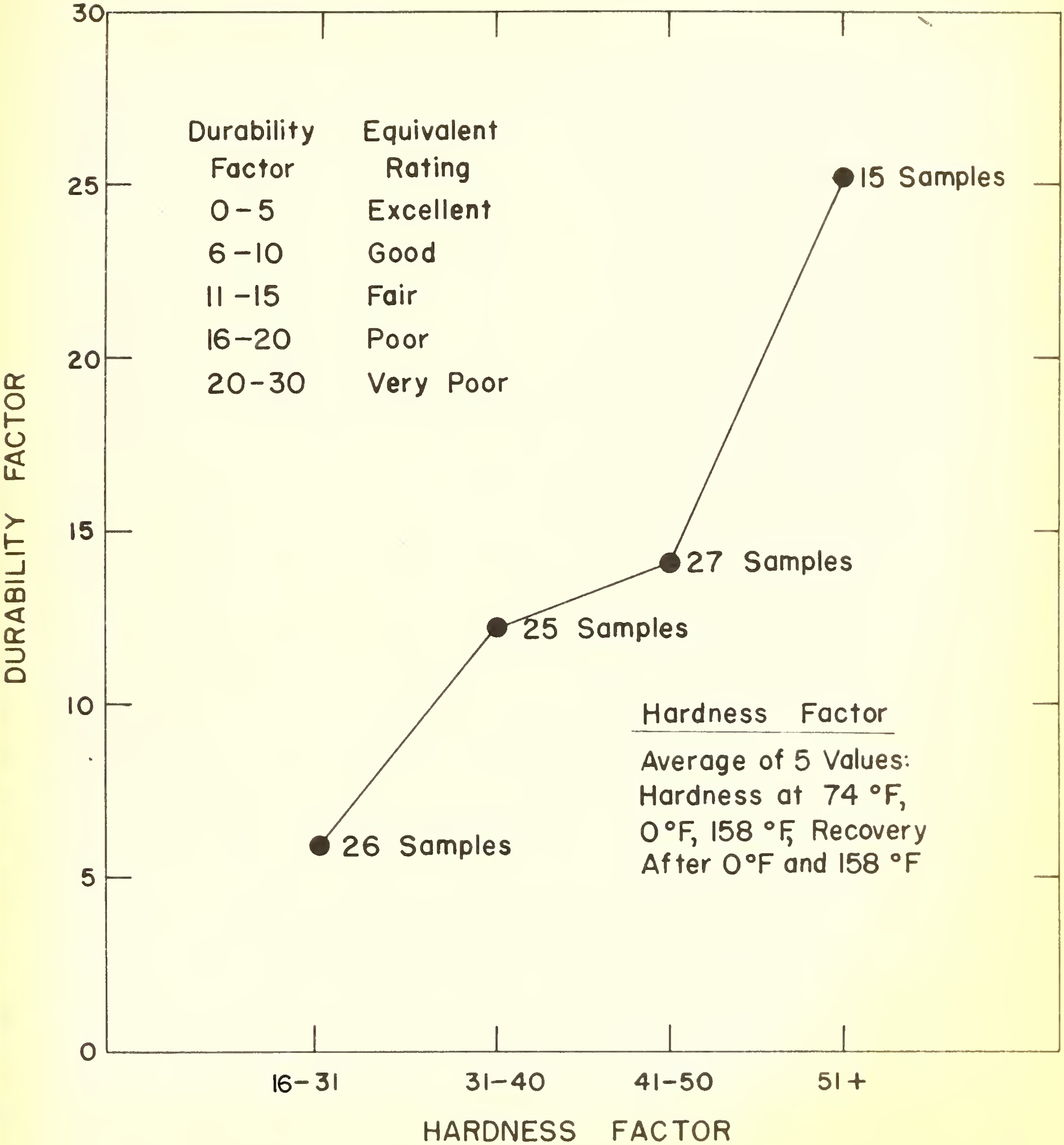

FIG. 12 Relationship between hardness factor and durability factor for 91 samples of polysulfide joint sealers tested in concrete joints

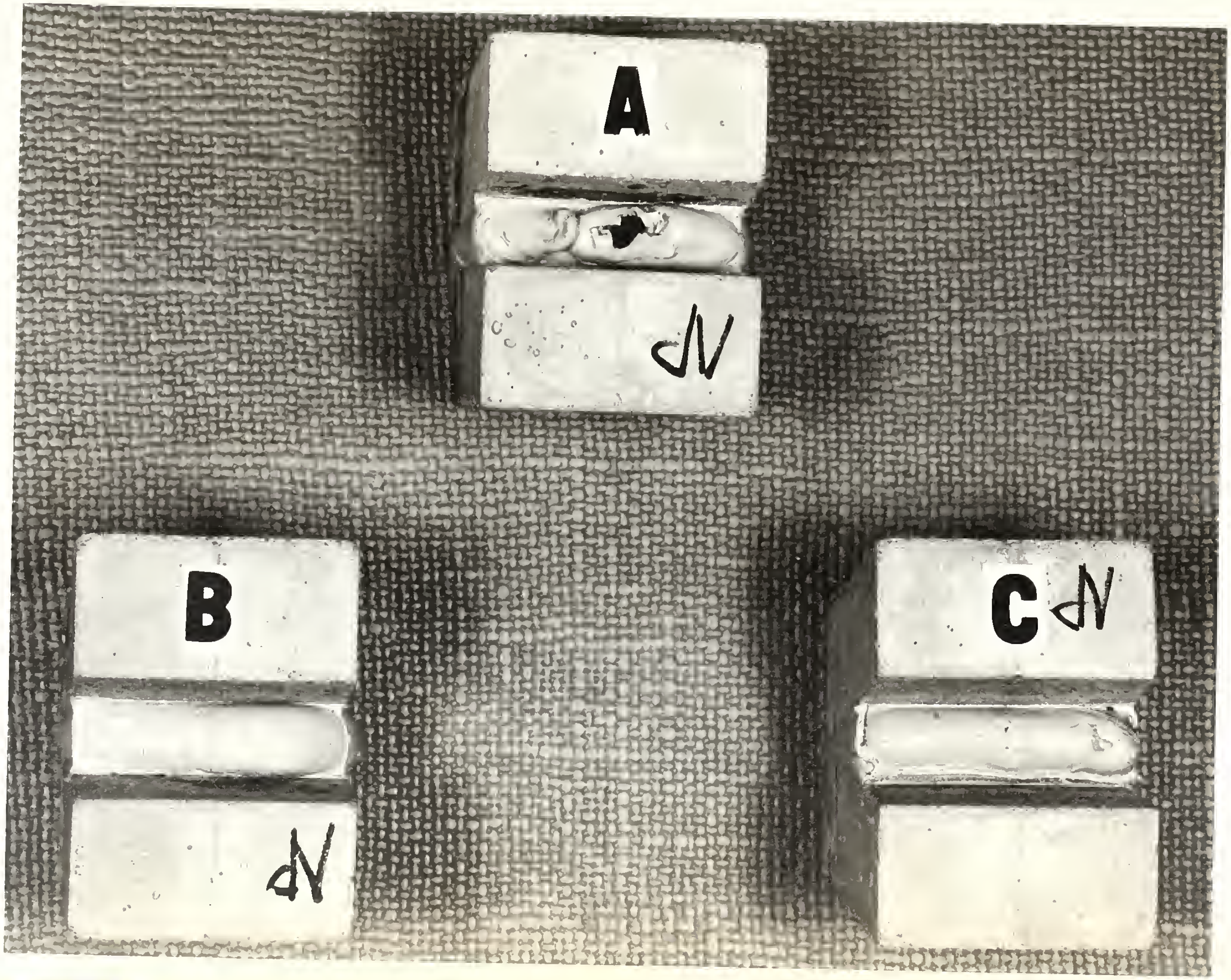

Fig. 13 - Bubbles formed in joint sealers in durability test. (A), Joint with open bubble. (B, C), Joints with closed bubbles.

Fig. 14 - Bubbles formed in joint sealers after exposure to the weather.
Left, 6 months exposure, open bubbles.
Middle, 21 months exposure, open and closed bubbles.
Right, 6 months exposure, closed bubbles.

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