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BUILDING MATERIALS and STRUCTURES

REPORT BMS33

Plastic Calking Materials

by J. J. TREGONING, K. A. MILLIKEN, A. HOCKMAN, W. H. SLIGH, and D. W. KESSLER



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Foreword

The penetration of moisture through masonry walls is usually confined to joints which were improperly filled or to joints which opened subsequent to construction on account of structural movements. Joints which are particularly apt to open from structural movements are those between the masonry and frames of doors or windows. Plastic calking materials have come into extensive use for filling certain joints in masonry as well as in some other types of construction. They are intended to remain sufficiently pliable to yield and maintain a seal in joints that vary in width to such an extent as to render the use of mortar or other rigid joint filler impracticable. Although the cost of the calking is relatively small, the failure to properly maintain a seal may result in costly repairs.

Considerable difficulty has been experienced in obtaining entirely satisfactory service from the plastic calking compounds, and hence a study has been made to devise suitable tests for determining the quality of such materials. Tests for rate of hardening, bond, tenacity, and shrinkage have been devised, and such determinations are of value in showing the probable behavior in service. It has been found that their performance is influenced, to a large extent, by the nature of porous materials with which the compounds are placed in contact. There is a marked variation in uniformity of all brands studied, which indicates that more rigid control is necessary in the manufacture of such products.

LYMAN J. BRIGGS, Director.

Plastic Calking Compounds

by J. J. TREGONING, K. A. MILLIKEN, A. HOCKMAN, W. H. SLIGH, and D. W. KESSLER

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ABSTRACT

One phase of this study was concerned with the development of test procedures and the accumulation of data to form the basis of a specification. By inspection of several installations and numerous specimen joints after exposure to the weather for several years, it was decided that tests for shrinkage, rate of hardening, bond, tenacity, staining, and slumping tendency afford a good basis for predicting the performance. Since calking compounds are commonly used in contact with porous materials whose absorptive properties materially affect the performance, it is necessary to employ an accessory porous material in the tests. For this purpose a certain limestone has been selected because of its uniformity. The test procedures have been so devised that three specimens are prepared for each sample and on these eight properties are determined.

A study of the composition of proprietary compounds has shown that a large variety of formulations are used. Oils most commonly used are fish, soybean, linseed, and tung. Cottonseed and rapeseed oils were employed in some mixtures whereas there were a few brands in which the vehicle consisted of rosin, tallow, or synthetic resin. In a considerable number of the proprietary compounds, the volatile content was too high for good performance. A study of compositions in relation to performance has indicated that pretreat-

ment of the oil is of first importance, and that considerable care must be exercised in selecting fillers as well as keeping the volatile content very low.

X. Summary_____

Certain factors which may affect the performance of plastic calking materials in service have been studied. Compounds that give good service in one type of masonry do not always give similar results on all other types. Often very porous masonry absorbs less of the vehicle than much denser materials do. Capillary forces of the pores seem to be of considerable importance. A compound may be satisfactory in wide joints but fail in narrow joints. Primers have been found to have some value in reducing shrinkage of mixtures of pronounced staining characteristics, but they may affect the bond adversely. Good mixtures do not require primers.

I. INTRODUCTION

The purposes of this investigation of calking compounds were (1) to develop simple test procedures and obtain data for use in specifications, (2) to study compositions and physical properties in relation to performance, and (3)to study the installation and service conditions which have a bearing on performance and durability.

A large number of commercial samples were collected and numerous experimental compounds were prepared in the laboratory. Chemical analyses were made of the commercial compounds. Physical tests were made on both commercial and experimental compounds. The tests now used in certain specifications were employed as a guide in determining the specific properties which give suitable indications of quality. Laboratory studies have been supplemented, as far as possible, with exposure tests and inspections of actual installations.

II. SAMPLES

Forty producers cooperated by supplying samples of their products. In several cases, two or more samples of the same brands were obtained at different times in order to determine the causes of certain undesirable properties. One hundred and ninety experimental compounds were prepared for a supplementary study of compositions.

A large number of samples submitted for test in connection with various Government construction projects were utilized for comparing the results of current tests with those of certain new tests which will be described later. Exposure tests, started at intervals during the past 8 years, were examined for information on quality. The specimens consisted of grooves in limestone blocks filled with the compounds. About 50 brands of calking are represented in these tests, and there are more than 100 specimens of some brands.

III. COMPOSITION STUDIES

1. Methods

The methods used in the chemical examination of commercial calking compounds are briefly outlined as follows:

(a) The volatile matter was determined by spreading 5 to 10 g of the calking compound in a thin layer in a tin dish about $2\frac{1}{2}$ in. in diameter, which had previously been weighed, weighing, heating the dish and contents for 3 hr at 105° C, and reweighing. The loss in weight at 105° C was regarded as "volatile," and from this figure the percentage of volatile was computed.

(b) The percentage of pigment was determined by weighing accurately about 15 g of the compound in a centrifuge tube. Fifty milliliters of extraction mixture (10 volumes of ethyl ether, 6 of benzol, 4 of methyl alcohol. 1 of acetone) was added and the sample thoroughly disintegrated in the solvent. The tube and contents were centrifuged rapidly for 5 min. The clear supernatant liquid was decanted into a casserole and heated to remove volatile constituents from the nonvolatile vehicle. The extraction was repeated two more times. The tube and pigment were then heated at 100° C for 3 hr, cooled, weighed, and the percentage of pigment calculated. The pigment was examined petrographically to identify its ingredients.¹

(c) The percentage of nonvolatile vehicle was determined by difference.

The identification of the type of saponifiable oil in the vehicle and its method of bodying, as given in table 1, were based on suitable chemical and physical methods The viscosities of the vehicles were estimated by comparison with Gardner-Holdt standards, after the extracting liquid had been removed from the vehicle.

2. Analytical Results

Table 1 contains the results of the chemical analyses of 72 commercial calking compounds. Most of them consisted of finely ground and fibrous materials (the pigments) mixed with a drying or semidrying oil and some volatile thinner (the vehicle). A great variety of materials were identified in the finely ground portion of the pigments, calcite being the most common, but quartz, lithopone, zinc sulfide, dolomite, and diatomaceous silica were also used. The fibrous material was usually asbestos in the medium-short fiber grades of the crysotile or tremolite varieties. The amount of pigment varied from 28 to 88 percent.

The vehicles in a few compounds were mineral oil, sulfurized oil, tallow, rosin, or cottonseed pitch. Mineral spirits was the most commonly used thinner, but kerosene was used in two of the samples. The amount of volatile matter varied from 1 to 37 percent.

 $^{^1}$ The authors are indebted to H. F. McMurdie for the petrographic examinations.

The treated oils used in the commercial calking compounds could not be identified as accurately as untreated oils. Because of the changes in the physical and chemical characteristics of oils which occur in the different methods of bodying, the identification of the bodied oils was sometimes uncertain. The oils in seven commercial compounds could not be identified, but the characteristics of others classified them into definite groups.

TABLE 1.—Composition an	d physical	studies on 72	2 commercial	samples o	f plastic calking
-------------------------	------------	---------------	--------------	-----------	-------------------

Pigment			Vehicle			lime-	Shrii	nkage Stain on-#			Condition of pats on line-		
Q		- Burent		Nonvolatile		5° C	vs on	at 15	days (Courr	011	stone h	
Sample designa- tion ^s	Amount	Nature •	Amount	Major constituent	Approximate viscosity °	Volatile d at 10	Strain at 15 day stone	Limestone	Glass	Limestone	Marble	Skin	Hardness
		COMPOS	SITI	ONS CONTAINING LI	NSEE	D 0	IL				_		
1 A-k	% 69	Calcite and chrysotile	% 30	Apparently blown lin-	V_1	$^{\%}_{1}$	<i>in</i> . 0.00	$\frac{\%}{6.9}$	$\frac{\%}{2.5}$	88	S	B, T, W	1
1 B-k 2 A-g 2 B-g 3 A-g 3 B-k		Calcite and chrysotile Lithopone, calcite, and chrysotile Calcite and lithopone Calcite, amphibole, and chrysotile Calcite, amphibole, lithopone, and abrueetile	$34 \\ 40 \\ 25 \\ 27 \\ 24$		$V_1 \\ V_2 \\ V_2 \\ V_2 \\ V_2 \\ V_2 \\ V_2$	$ \begin{array}{c} 1 \\ 6 \\ 6 \\ 3 \end{array} $. 03 . 00 . 00 . 21 . 16	4. 2 14. 7 6. 4	$\begin{array}{c} 3.2 \\ 4.6 \\ 5.2 \\ 4.5 \\ 2.4 \end{array}$	0K 0K 0K 0K	S S OK SS	T- WTh - WTh - WTh - WSm - Th	$ \begin{array}{r} 1-2 \\ 2 \\ 3 \\ 2-3 \\ 2-3 \end{array} $
4 A-g 5 A-g 6 A-g 6 B-k 7 A-g 7 B-k 6 C-g 8 A-k		Chrysotile and amphihole Calcite, amphibole, and chrysotile Tremolite and zinc oxide Calcite. Tremolite, caleite, and feldspar Calcite Zinc oxide, chrysotile, and tremolite Calcite	33 45 36 14 29 21 31 11	do do do do do Apparently polymerized linseed oil. do	$V_2 V_2 V_2 V_3 V_3 V_3 V_2 V_2 V_2 V_2$	$ \begin{array}{r} 7 \\ 5 \\ 10 \\ 2 \\ 12 \\ 1 \\ 9 \\ 1 \end{array} $	$ \begin{array}{r} 28 \\ .18 \\ .07 \\ .01 \\ .16 \\ .08 \\ .00 \\ .08 \\ .00 \\ .08 \\ .$	5.56.410.65.510.85.615.4 6.4	2.5 3.2 8.3 2.3 7.0 2.0 5.9 2.3 2.3	OK OK OK OK OK	OK OK SS OK SS OK	$\begin{array}{c} Th-W\\ T-W\\ Th-W\\ Th-W\\ Th-W\\ Th-W\\ Th-W\\ T-W\\ B-Th-W\\ \end{array}$	2-3 1 2-3 2-3 3 2-3 1 1 2
8 B-g	87	Calcite	12	do	V 2	1	. 02	2.9	1.6	OK	OK	B-Th-W	2
		COMPOS	SITI	ONS CONTAINING SO	YBE	IN C	DIL	_					
9 A-g 10 A-g	53 57	Amphibole and lithopone	35 30	Apparently blown soy- hean oil.	V_1 V_1	12 13	0. 05 . 12	12.3 18.4	7.4 5.8	0K 0K	s	Th-W Th-W	23 23
10 B-k 11 A-g 11 B-k 12 A-g 13 B-k 13 B-k 14 A-g 14 B-g 14 B-g 14 C-k	$ \begin{array}{r} 60 \\ 60 \\ 68 \\ 48 \\ 47 \\ 79 \\ 47 \\ 49 \\ 55 \\ 10 \\ \end{array} $	amphibole. Lithopone, amphihole, and chrysotile Quartz, chrysotile, and amphihole Amphibole, quartz, and chrysotile Calcite, tremolite, and chrysotile Calcite, chrysotile, and opaque material Calcite, chrysotile, amphibole, and opaque material. Calcite, chrysotile, and opaque material	27 30 23 38 43 20 42 42 42 42 36 36	do	$V_1 V_1 V_1 V_2 V_2 V_2 V_2 V_2 V_2 V_2 V_2 V_2 V_2$	$ \begin{array}{r} 13 \\ 10 \\ 9 \\ 14 \\ 10 \\ 1 \\ 11 \\ 9 \\ 9 \\ 9 \\ 9 \\ 14 \\ 10 \\ 11 \\ 9 \\ 9 \\ 14 \\ 10 \\ 11 \\ 9 \\ 9 \\ 14 \\ 10 \\ 11 \\ 9 \\ 9 \\ 14 \\ 10 \\ 11 \\ 9 \\ 9 \\ 14 \\ 10 \\ 11 \\ 9 \\ 9 \\ 14 \\ 10 \\ 11 \\ 9 \\ 9 \\ 14 \\ 10 \\ 11 \\ 9 \\ 11 \\ 9 \\ 9 \\ 11 \\ 9 \\ 11 \\ 9 \\ 11 \\ 9 \\ 11 \\ 9 \\ 11 \\ 9 \\ 11 \\ 9 \\ 11 \\ 9 \\ 11 \\ 9 \\ 11 \\ 9 \\ 11 \\ 10 \\ 11 \\ 11 \\ 10 \\ 11 \\ 11 \\ 9 \\ 10 \\ 11 \\ 11 \\ 9 \\ 11 \\ 10 \\ 11 \\ 11 \\ 9 \\ 11 \\$	$\begin{array}{c} .18\\ .00\\ .00\\ .04\\ .07\\ .01\\ .05\\ .00\\ .00\\ .00\\ \end{array}$	$ \begin{array}{c} 11.5\\20.3\\17.7\\17.7\\12.3\\2.2\\9.6\\9.8\\11.8\\\end{array} $	$\begin{array}{c} 6.4\\ 6.3\\ 6.1\\ 8.7\\ 6.4\\ 1.7\\ 4.9\\ 6.0\\ 5.0\\ 5.0\\ \end{array}$	OK OK OK OK OK OK	S OK OK OK OK OK	$\begin{array}{c} Th-W\\ T-W\\ Th-W\\ Th-W\\ Th-H\\ Th-W\\ \hline \\ Th-W\\ Th-W\\ \end{array}$	3 1 2-3 2 (i) 2-3 2-3 2-3 (i) (i)
14 D-g 14 E-g 13 C-k	48 43 57	Calcite, chrysolite, and amphibole Zine oxide and chrysotile. Calcite, chrysolite, and amphibole	38 45 41	do do do	$V_2 \ V_2 \ V_3$	$\begin{array}{c}14\\12\\2\end{array}$	0.02 .23 .39	$24.\ 0\\16.\ 1\\3.\ 6$	9.7 9.5 3.6	${ OK \atop OK \\ OK \\ OK }$	SS SS OK	Th-W Sm-Th Th	2 2 2-3
		COMI	POSI	TIONS CONTAINING	FISH	on	1						
13 D-g	56	Dolomite, calcite, chrysotile, and amphi- bole.	27	Apparently hlown fish oil.	V_1	17	0. 07	18.4	11. 8	OK	s		(i)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{r} 49\\ 53\\ 71\\ 61\\ 34\\ 54\\ 56\\ 56\\ 47\\ 78\\ 57\\ 54\\ 43\\ \end{array}$	Calcite and chrysotile. Calcite, chrysotile, and amphibole. Calcite and chrysotile, and tremolite. Calcite, chrysotile, and tremolite. Calcite, cotton fibers, and chrysotile. Calcite. Cotton fibers, calcite, and chrysotile. Chrysotile, calcite, and dolomite. Calcite and chrysotile. Calcite, tremolite, and chrysotile. Calcite, amphibole, and chrysotile. Calcite, amphibole, zine oxide, and chrysotile.	$\begin{array}{c} 26\\ 29\\ 21\\ 24\\ 59\\ 39\\ 33\\ 30\\ 19\\ 33\\ 45\\ 46 \end{array}$	do do do do do do do do do do	$\begin{array}{c} V_{1} \\ V_{2} \\ V_{2} \\ V_{2} \\ V_{2} \\ V_{2} \\ V_{3} \end{array}$	$25 \\ 18 \\ 8 \\ 15 \\ 7 \\ 7 \\ 11 \\ 11 \\ 23 \\ 3 \\ 10 \\ 1 \\ 11$	$\begin{array}{c} . \ 02 \\ . \ 09 \\ . \ 00 \\ . \ 21 \\ . \ 13 \\ . \ 11 \\ . \ 17 \\ . \ 06 \\ . \ 11 \\ . \ 12 \\ . \ 24 \\ . \ 15 \end{array}$	$\begin{array}{c} 24.7\\ 22.7\\ 18.2\\ 17.0\\ 8.8\\ 9.8\\ 13.5\\ 14.6\\ 19.5\\ 12.0\\ 8.9\\ 6.3\\ 14.6\end{array}$	$\begin{array}{c} 12.1\\ 11.9\\ 6.0\\ 10.1\\ 4.5\\ 4.1\\ 9.6\\ 9.8\\ 11.7\\ 2.2\\ 7.5\\ 3.7\\ 9.2 \end{array}$	OK SS OK OK OK OK OK OK OK	S S OK OK OK OK S OK OK	Sm-Th Sm-Th Th-W Th-W	
17 B-k 19 A-g 20 A-g 20 B-g 16 C-k 21 A-g 22 A-k	79 50 60 59 60 49 76	Amphihole and calcite Calcite and chrysotile Calcite Chrysotile and calcite Calcite, chrysotile, and amphihole Amphihole and chrysotile Calcite, amphihole, and lithopone	$20 \\ 37 \\ 32 \\ 33 \\ 35 \\ 38 \\ 16$	do	$V_3 V_3 V_3 V_3 V_3 (e) (e) (e) (e)$	$ \begin{array}{c} 1 \\ 13 \\ 8 \\ 5 \\ 13 \\ 8 \end{array} $	$ \begin{array}{r} 21 \\ 13 \\ 10 \\ 19 \\ 09 \\ 13 \\ 00 \end{array} $	$\begin{array}{c} 2.9\\ 14.2\\ 9.9\\ 10.9\\ 6.4\\ 12.1\\ 10.0 \end{array}$	$\begin{array}{c} 1.\ 0\\ 9.\ 4\\ 5.\ 4\\ 7.\ 0\\ 4.\ 0\\ 5.\ 0\\ 5.\ 3\end{array}$	OK OK OK OK OK	OK OK OK OK S S	Sm-T Th-W Sm-Th Th-W	(i) (i) (i) (i)

See footnotes at end of table.

[3]

TABLE	1.—Compos	ition and	physical	studies a	on 72	commercial	samples	of	plastic	calking—	-Continued	ł
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		Dismont		Vehicle	lime-	Shrinkage		Stein	0.0	Condition	of		
		Likment		Nonvolatile		5° C		at 15	at 15 days		01 -	stone	
Sample designa- tion	Amount	Nature	Amount	Major constituent	Approximate viscosity	Volatile at 10	Strain at 15 da ston	Limestone	Glass	Limestone	Marble	Skin	Hardness
		COMPO	0817	TIONS CONTAINING T	rung	011	L						
18 C-g	% 60	Calcite and chrysotile	$\frac{\%}{29}$	Apparently polymerized tung oil.	V_2	% 11	in. 0.25	% 6.3	% 4. 1	OK	OK	B-T-W	1
18 D-k 18 E-k 23 A-g 24 A-g		Calcite	$14 \\ 12 \\ 28 \\ 30$	do	$V_2 \\ V_2 \\ V_2 \\ V_2 \\ V_2 \\ V_2$. 01 . 37 . 31 . 13	4.0 3.2 8.7 16.7	$ \begin{array}{r} 1.7 \\ 1.7 \\ 4.6 \\ 9.5 \\ \end{array} $	OK OK OK OK	OK OK SS OK	$\begin{array}{c} \text{do.}\\ Th-W\\ Th-W\\ B-Th-W\\ \end{array}$	$ \begin{array}{r} 1-2 \\ 1-2 \\ 1-2 \\ 2 \end{array} $
	I	COMPOSITION	s C (ONTAINING MISCELL	ANE	ous	VEHI	CLES					
25 A-k 25 B-g 26 A-g	$\begin{array}{c} 64\\ 63\\ 47\end{array}$	Chrysotile and lithopone Chrysotile and lithopone Tremolite, feldspar, and lithopone	19 21 47	Apparently rapeseed oil do Apparently blown cot-	$V_1 \ V_1 \ V_1 \ V_3$	$\begin{array}{c} 17\\16\\6\end{array}$	0. 01 . 01 . 37	$13. \ 6 \\ 16. \ 0 \\ 9. \ 4$	$6.0 \\ 7.0 \\ 6.8$	OK OK OK	S S OK	Tb-W	(i) (i) 2
27 A-g 27 B-g 27 C-g 16 E-g	$30 \\ 50 \\ 69 \\ 56$	Opaque material Opaque material Opaque material Amphibole, calcite, chrysotile and	$33 \\ 24 \\ 21 \\ 40$	Cottonseed pitch	$\stackrel{({\bf e})}{\stackrel{({\bf e})}{\overset{({\bf o})}{V_3}}}$	$37 \\ 26 \\ 10 \\ 4$. 10 . 02 . 01 . 02	$36.2 \\ 34.2 \\ 7.3 \\ 11.3$	$20.\ 6\\15.\ 7\\2.\ 2\\2.\ 5$	OK OK OK SS	8 8 88 88	<i>T-W</i>	${(i)} {(i)} {(i)} {(i)} {1}$
16 F-k 28 A-g 29 A-k 30 A-g 30 B-g 30 C-k 30 D-g 30 E-g	$70 \\ 28 \\ 63 \\ 47 \\ 57 \\ 81 \\ 48 \\ 47$	Amphibole and chrysotile Lithopone and chrysotile Talc, chrysotile, calcite, and amphibole Calcite, amphibole, and chrysotile Calcite and chrysotile Calcite. Amphibole, calcite, and chrysotile Calcite, chrysotile, amphibole, and zinc oxide	$29 \\ 71 \\ 34 \\ 35 \\ 29 \\ 18 \\ 38 \\ 37$	do Apparently rosin Apparently tallow Unknown do do do do do	$\begin{array}{c} V_{3} \\ (\bullet) \\ (\bullet) \\ V_{2}^{2} \\ V_{3}^{2} \\ V_{3}^{3} \\ V_{3}^{3} \end{array}$	$ \begin{array}{c} 1 \\ 1 \\ 3 \\ 18 \\ 14 \\ 1 \\ 14 \\ 16 \\ 16 \\ \end{array} $.02 .29 .38 .18 .00 .00 .17 .00	$\begin{array}{r} 4.2 \\ 16.2 \\ 18.5 \\ 8.2 \\ 13.6 \\ 21.5 \end{array}$	$2.1 \\ 8.1 \\ 7.5 \\ 1.7 \\ 5.6 \\ 9.6$	OK OK OK OK OK OK	OK SS OK SS OK S OK OK	Sm-Th B-Th B-Sm-T B-W Th-W Th-W	${\begin{array}{*{20}c}1\\1\\2\\2\\(^{i})\\2\\1-2\end{array}}$
31 A-g 32 A-g	$\begin{array}{c} 52 \\ 66 \end{array}$	Magnesite and chrysotile Calcite	33 19	do	$V_3 \ V_3$	$\begin{array}{c} 15\\ 15\end{array}$. 02 . 00	$13.8 \\ 6.4$		OK OK	OK OK	Th B-Sm-T	2-3 1

Numbers designate particular manufacturers; letters A, B, C, etc., different samples or brands; g and k stand for gun grade and knife grade respectively; e.g. manufacturer 2 submitted 2 gun-grade samples, designated 2 A-g and 2 B-g.
 Calcite includes the various forms of whiting such as chalk whiting, precipitated whiting and ground limestone. Chrysotile is a variety of asbestos while the minerals identified as amphibole and tremolite are mainly mixtures of fibrous (absestos) and nonfibrous particles.
 Viscosity values approximated on extracted oils {V_i, in range A to T (Gardner-Holdt standards).
 V_i, in range C to Z (Gardner-Holdt standards).
 V_i, in range J to Z (Gardner-Holdt standards).

d Volatile by method described in section 111, 1-Tbis ingredient was apparently mineral spirits except in samples 25A and 26B.

Not determined.
 Shrinkage of ¼-in. layer of plastic on limestone; ½-in. layer on glass.

limestone is used for this purpose.

 $^{\circ}$ OK=satisfactory; SS=slight stain; S=stain. ^b These observations were made at end of first year, on specimens stored in the laboratory. B=brittle, Sm=smooth, T=thin, Th=thick, W=wrinkled. Numerals signify hardness, namely, 1=satisfactory, 2=hard, 3=very hard. Two figures, 1-2, signify intermediate hardness. ⁱ Specimen hard throughout.

IV. PHYSICAL DETERMINATIONS

The tests used heretofore in connection with specifications have been of value in determining the early behavior of materials under conditions similar to those of service, but some of them are cumbersome and do not afford the desired information as to the cause of poor performance. Hence a series of simpler tests has been devised which it is thought will determine the quality of calking compounds more easily and definitely. A satisfactory means of testing these products without an accessory porous material has not been found, and hence a particular

1. CURRENT TESTS

Tests² used during the past 8 years consist of (1) a strain test (see fig. 1) made on a joint between two slabs of a designated limestone;³ (2) a stain test on limestone (see fig. 2, D and F) and marble; (3) a slump test (see fig. 2, E); and (4) a copper corrosion test. The requirements are that a ¼-in. joint in the strain test can be extended (stretched) 0.05 in. without breaking the seal, that the plastic in a groove 3% in. wide

² A test procedure for plastic calking materials, Proc. Am. Soc. Testing Materials 35, pt. 2, 581 (1935).

³ This limestone is produced by the Rockwood Alabama Stone Co., Russellville, Ala., and is designated as "veinless gray" from the Aday quarry. Before use in any of the tests the stone is dried for 24 hr at 105° C.

and $\frac{3}{4}$ in. deep in a block of limestone shall not slump or flow when the filled groove is placed in a vertical position for 24 hr at room temperature followed by 24 hr at 50° C (122° F), that oil shall not separate from the plastic and stain the limestone block used in the slump test or spread around a pat placed on white marble,

(a) Shrinkage

This property is determined by means of a brass ring 2% in. in diameter and $\frac{1}{2}$ in. high (volume, V, known to nearest 0.01 ml), a ground-glass cover plate and a slab of limestone $3\frac{1}{2}$ by $3\frac{1}{2}$ by $\frac{3}{4}$ in. (see fig. 2, C). The combined



FIGURE 1.-Strain-testing devices.

A, horizontal type; E, vertical type, showing strain gages C and F with specimens in position for testing. D is a specimen joint used in type A, and G shows the accessory materials for making a joint for type E.

and that the plastic shall not discolor a bright copper plate when a pat is left on the surface for 5 days.

2. Proposed Tests

Physical properties which are believed to have a bearing on performance are (a) shrinkage, (b) bond, (c) tenacity, (d) rate of hardening, (e) oil retention, and (f) consistency. Density and volatile loss of the plastics are obtained in connection with the shrinkage determination. Since these procedures have not been published heretofore, they are described here in detail. weight (W_1) of the ring, slab, and cover plate is determined (all weights to 0.01 g); then the ring is centered on the slab, and a $\frac{1}{2}$ -in. layer ⁴ of the plastic is spread inside the ring to form contact with the stone and ring, and the weight (W_2) determined (with cover plate). The portion of the ring above the plastic is then filled

⁴ A tool used for spreading and gaging the thickness of the layer is made from a piece of brass 4 hy $\frac{3}{4}$ hy $\frac{1}{2}$ in. At $\frac{1}{2}$ in. from one end a slot $\frac{1}{2}$ in, wide and $\frac{5}{4}$ in, deep is cut perpendicular to one edge. From the slot to the nearer end the edge of the metal is cut back for $\frac{1}{2}$ in. The sharp corner hetween the notch and the new edge is rounded and beveled slightly for the purpose of producing a fillet with the calking where it joins the ring. The tool is held with the notch over the edge of the ring and the short end inside the ring. The edge outside the ring is held firmly against the stone slah as the tool is moved around to level the calking sample.

with distilled water, so there are no air bubbles when the cover plate is in place and this weight (W_3) is determined. Since the volume of 1 g of water is approximately 1 ml, the volume of the plastic $V_c = V - (W_3 + W_2)$. The water is then poured off and the sample exposed to the air for 15 days, after which the weights W_2' split, leaving a soft layer in contact with the stone.

This property may be also determined by spreading a %-in. layer of the plastic on a slab of limestone and after submerging the specimen in water for 2 days. If the bond is unsatisfactory, blisters will form in the plastic.



FIGURE 2.—Apparatus and specimens.

A, penetration specimen; B, penetration apparatus; C, shrinkage specimen and equipment. D, E, and F, are performance tests in which D shows no slump or stain, E shows slumping, and F shows staining.

and $W_{3'}$, corresponding to W_2 and W_3 , are determined. The volume shrinkage is $(W_{3'} - W_{2'}) - (W_3 - W_2)$ and this times 100 divided by V_e is the percentage of shrinkage.

(b) Bond

The bonding value of the plastic is estimated from the shrinkage specimen after removing the ring. One-fourth of the plastic is loosened from the stone with a spatula and pulled upward. If the plastic peels off, the bonding property is not satisfactory. A satisfactory sample will

(c) Tenacity

This may be determined on the sample used for shrinkage. The portion loosened for the bond test is folded over on the remaining part and creased three times (bent 180°) along the same line. Embrittlement, or lack of tenacity, is indicated if the specimen breaks or cracks.

(d) Rate of Hardening

This is determined with a penetrometer (see fig. 2, B) on a joint between limestone slabs spaced $\frac{3}{8}$ in. filled with the plastic to a depth

of 1¼ in. and length of $3\frac{1}{2}$ in.⁵ The apparatus is the same as that required for determining the penetration of bituminous materials (ASTM standard D 5–25, penetration of bituminous materials),⁶ except that the weight on the needle point is reduced to 12.5 g. The depth to which the needle penetrates the plastic in 5 sec is recorded for three trials made near the middle of the joint. After curing the joint for 15 days, the skin is removed and three more penetration readings made. The hardening is computed from

$$h = \frac{(P_1 - P_2)100}{P_1},$$

in which h is the rate of hardening, P_1 and P_2 are respectively the averages of the original and final penetrations.

(e) Oil Retention

This is rated from the specimen used to determine rate of hardening by measuring the distance on either side of the calking that the limestone is discolored.

(f) Consistency

Since the principal concern of the buyer in regard to consistency is whether the material will flow from the joints or sag, this test is made to simulate service conditions. A joint specimen is made between the same two slabs of limestone used for the rate of hardening test (see fig. 2, A). The space filled is $\frac{3}{6}$ in. wide, $1\frac{14}{4}$ in. deep, and $3\frac{12}{2}$ in. long. The joint is placed in a vertical position with no support below the plastic. The amount of sag is measured from the lower face of the stone to the lowest point assumed by the plastic after storing 1 day at room temperature and 1 day at 50° C (122° F).

(g) Density

Density is computed from the data obtained in the shrinkage test as follows:

$$D = \frac{W_2 - W_1}{V_c}$$

(h) Volatile Loss

The loss in weight of the specimens used for the shrinkage test is considered as the volatile loss. This is reported as a percentage by weight of the sample, and is computed from $v=(W_2-W_2')100/(W_2-W_1)$.⁷

3. Comparison of Results of Current and Proposed Tests

Table 2 gives results of physical tests on 25 samples of trade brands and also on 25 experimental compounds made in the laboratory. The trade samples were submitted for various Government projects between April 1938 and February 1939. Laboratory mixtures show typical results for those made in this investigation. The data indicate that the shrinkage and rate of hardening values were lower for experimental than for commercial compounds, indicating a probability of better performance in service.

⁵ Three penetration readings are made along the center line of the joint, as soon as the specimen has been prepared, by determining the depth of penetration in 5 see. The position of the points for test are selected so that the first is about 36 in. from one end of the joint, and the other two are spaced at intervals of about 34 in. The specimen is then stood on edge with the joint horizontal and aged for 15 days. After removing the skin from the ealking material, three penetration readings are made at points 34 in, apart, the first being 38 in. from the end opposite to that used for the first penetration.

 $^{^{6}}$ Am. Soc. Testing Materials standards, nonmetallie materials, pt. 2, p. 1080 (1936).

⁷ The percentage of volatile determined in this way is less than that determined by heating at 105° C (the basis for "volatile" in table 1).

TABLE !	2.—Results	of	physical	tests ¹	on	commercial	and	experimental	compound	ls
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		Stair	1 on 3			Volatile		T	Penet	ration	Rate of
Sample designation ²	Strain	Lime- stone	Lime- stone Marble Shump ³ Shrinkage loss (by weight) Bond ³ ity ³		ity ³	Original	Final	barden- ing			
C1 C2 C3 C4 C5	<i>in</i> . 0.40 .30 .24 .11 .14	OK OK OK OK	OK SS OK OK OK	OK OK OK OK		% 0.1 4.0 2.2 1.7 6.1	OK OK OK OK	OK OK OK OK	$\begin{array}{c} 0.01\ cm \\ 58 \\ 243 \\ 150 \\ 170 \\ 212 \end{array}$	$\begin{array}{c} 0.01\ cm \\ 41 \\ 134 \\ 76 \\ 92 \\ 106 \end{array}$	% 29 45 49 46 50
C6 C7 C8 C9 C10	.11 .12 .16 .31 .14	OK OK OK OK	S OK OK OK	0K 0K 0K 0K	$17.5 \\ 8.9 \\ 19.8 \\ 6.2 \\ 13.5$	$\begin{array}{c} 6.4\\ 2.2\\ 6.7\\ 0.1\\ 9.5\end{array}$	OK OK OK OK	OK OK OK OK	224 83 282 138 238	$108 \\ 38 \\ 134 \\ 67 \\ 103$	52 54 52 51 51 57
C11 C12 C13 C14 C14 C15	20 31 04 04 90	OK OK OK OK	0K 0K 0K 0K	OK OK OK OK	$10.3 \\ 9.4 \\ 25.1 \\ 24.0 \\ 27.0$	$ \begin{array}{r} 1.6 \\ 1.6 \\ 12.4 \\ 9.4 \\ 13.7 \\ \end{array} $	OK OK F OK OK	OK OK F F OK	251 224 273 275 210	$129 \\ 120 \\ 67 \\ 93 \\ 115$	$49 \\ 46 \\ 76 \\ 66 \\ 41$
C16 C17 C18 C19 C29 C20	. 04 . 02 . 00 . 01 . 03	OK SS OK OK OK	S SS OK S OK	OK SS OK OK	$\begin{array}{c} 23.\ 0\\ 7.\ 2\\ 26.\ 2\\ 16.\ 7\\ 22.\ 3\end{array}$	$\begin{array}{c} 8.5 \\ 0.2 \\ 10.8 \\ 6.2 \\ 10.5 \end{array}$	OK F OK OK OK	OK F OK F OK	$242 \\ 95 \\ 160 \\ 97 \\ 250$	95 19 60 38 87	61 80 61 61 65
C21 C22 C23 C24 C24 C25	. 00 . 04 . 03 . 00 . 00	OK OK OK OK	OK OK OK S	OK OK OK OK	$\begin{array}{c} 20.\ 2\\ 22.\ 4\\ 6.\ 0\\ 7.\ 2\\ 4.\ 2\end{array}$	$\begin{array}{c} 6.7 \\ 5.2 \\ 0.2 \\ 0.0 \\ 0.6 \end{array}$	F F OK F F	OK OK F F F	$232 \\ 163 \\ 135 \\ 258 \\ 163$	$74 \\ 68 \\ 46 \\ 45 \\ 24$	68 58 66 83
E103 E166 E167 E168 E170	$ \begin{array}{r} 20 \\ 24 \\ 10 \\ 17 \\ 17 \end{array} $	0K 0K 0K 0K	VS OK VS OK VS	OK OK OK OK	$7.1 \\ 4.8 \\ 12.7 \\ 11.6 \\ 13.0$	$\begin{array}{c} 0.0\\ 0.0\\ 1.9\\ 1.3\\ 1.7\end{array}$	OK OK OK OK	OK OK OK OK	160 225 278 255 159	138 130 137 106 70	14 42 51 58 56
E171 E172 E173 E173 E174 E175	.12 .26 .27 .27 .40	0K 0K 0K 0K 0K	VS OK OK OK	OK OK OK OK	$14.7 \\ 5.0 \\ 7.6 \\ 11.5 \\ 13.0$	$ \begin{array}{r} 1.9 \\ 0.0 \\ 0.7 \\ 1.5 \\ 1.9 \\ \end{array} $	0K 0K 0K 0K	0K 0K 0K 0K	$2500 \\ 200 \\ 273 \\ 265 \\ 223 $	82 230 170 169 146	41 22 38 30 31
E176 E177 E178 E179 E180	.36 .03 .00 .18 .10	OK SS S OK SS	OK SS S VS S	OK OK OK OK	$\begin{array}{c} 16.\ 0\\ 25.\ 2\\ 22.\ 0\\ 12.\ 8\\ 12.\ 3\end{array}$	$ \begin{array}{r} 1.5 \\ 3.2 \\ 0.0 \\ 3.3 \\ 0.6 \\ \end{array} $	OK OK F OK OK	OK OK F OK OK	$\begin{array}{c} 214 \\ 205 \\ > 290 \\ > 290 \\ 286 \end{array}$	130 130 88 222 242	39 31 70 22 15
E181	. 22 . 22 . 24 . 26 . 25	0K 0K 0K 0K	OK OK OK OK	OK OK OK OK	$13. \ 4 \\ 11. \ 3 \\ 13. \ 1 \\ 10. \ 6 \\ 10. \ 4$	3.1 1.9 4.4 2.4 2.3	OK OK OK OK	OK OK OK OK	$\begin{array}{c} 290 \\ > 290 \\ 283 \\ 225 \\ 202 \end{array}$	225 244 213 187 126	22 16 24 17 38
E186 E187 E188 E189 E190	$. 15 \\ . 26 \\ . 25 \\ . 33 \\ . 45 $	OK OK OK OK	OK OK OK OK	OK OK OK OK	$ \begin{array}{c} 16.9\\ 11.5\\ 13.2\\ 8.8\\ 12.5 \end{array} $	$\begin{array}{c} 4.5 \\ 2.8 \\ 3.1 \\ 1.6 \\ 3.8 \end{array}$	OK OK OK OK	OK OK OK OK	259 249 261 245 202	188 152 134 144 113	27 39 49 41 44

[All gun-grade samples]

¹ Strain, staining, and slump tests were made by the methods described in section IV, 1; the other tests were made by the methods described in section IV, 2. ² Commercial samples designated by C; experimental, by E, ³ OK=satisfactory; VS=very slight; F=failure; S=stain; SS=slight stain.

The average and range values for commercial and experimental compounds are given in table 3.

TABLE 3.—Average and range values for commercial and experimental compounds

Compounds	Shrin	ikage	Rat hard	e of ening	Volatile loss		
	Average	Range	Average	Range	Average	Range	
Commercial Experimental	Percent 15 12	<i>Percent</i> 4 to 27 5 to 25	Percent 60 36	Percent 29 to 85 17 to 70	Percent 5	Percent 0 to 14 0 to 4	

V. RELATION OF TYPE OF BUILDING MATERIAL AND WIDTH OF JOINTS TO PERFORMANCE

The performance of calking compounds on different types of building materials is influenced by the porosity and the capillary forces in the pores. Shrinkage and hardening of the mastics, are to a large extent, caused by the loss of some of the vehicle. Porous materials in contact with calking compounds may absorb

a sufficient amount of the oil to cause excessive shrinkage and hardening, whereas the same plastic in contact with less porous materials may give satisfactory results (see figs. 3 and 4). On the other hand, the mastics adhere more tenaciously to porous materials. When a joint is made between briekwork and a metal and exeessive shrinkage occurs, the mastie will usually separate from the metal. Furthermore, the ability of the masties to remain in a vertical joint without slumping is affected by porosity and capillary forces. Workmen prefer a ealking material that ean be easily applied with a gun. Producers naturally cater to their customers and make the mixtures as soft as possible. This sometimes leads to trouble, because a mixture that will remain in joints between absorptive briek may flow out of joints between marble or other less absorptive materials.

Figure 5 illustrates the manner in which a calking compound may flow from vertical joints between dense brieks, whereas the same plastic remains in place between porous bricks. Figure 6 shows similar results when a ealking compound was placed in vertical joints ranging from nonporous on the left to very porous materials on the right. Figure 7 shows the progressive shrinkage of three commercial calking compounds on glass and on limestone over a period of 30 days, and figure 8 shows the shrinkage of six samples on limestone for 10 months. The shrinkage on glass is assumed to be caused entirely by evaporation of volatile thinner, whereas that on limestone is the result of volatilization and absorption of vehicle. For samples referred to in figure 7, the loss of volatile caused more than half of the shrinkage. Mixtures of low volatile eontent shrink mainly from loss of oil to adjoining materials, and if in contact with nonporous materials or porous materials of low eapillarity, such mixtures show little shrinkage.

Calking materials give better results in wide joints between porous materials than in narrow joints. Figure 9 shows one calking mixture in joints between limestone slabs where the joint widths vary from $\frac{1}{8}$ in to $\frac{1}{2}$ in. The narrow



FIGURE 3.—Staining of various materials by one calking mixture.

The irregular stains on briek (8), limestone (6), concrete (4), and marble (1) probably indicate variations in the eapillary properties of these materials. Craeks formed in the joints between the materials which absorbed eonsiderable amounts of oil.

joints show excessive shrinkage and cracking. The actual amount of oil absorbed from the calking was about the same for all joints, but the total amount of oil in the plastic required to fill a narrow joint is less than that for a wide one. For example, if a $\frac{1}{2}$ -in. joint and a $\frac{1}{2}$ -in. joint are filled with the same mixture, there will be only one-fourth as much in the



FIGURE 4.—One calking compound between dense bricks (light colored) and more porous bricks. Cracks formed between most of the porous bricks within a few days, whereas the calking compound was in good condition between the dense bricks.

first as in the second. Usually plastic calking in joints less than ¼ in. wide is not very satisfactory. When it is necessary to calk very narrow joints or cracks, one should use a plastic containing very little volatile and an oil that will not stain. A somewhat stiffer mixture is required for wide joints in order to prevent sagging.

VI. RELATION OF COMPOSITION TO PERFORMANCE

Table 1 gives the results of composition studies and physical tests on 72 commercial compounds. The data on volatile and shrinkage were determined by methods which were changed later to simplify the tests, and for this reason these data are not strictly comparable with those presented in tables 2, 4, and 6.

There are so many variables involved in the manufacture of commercial compounds that the results in table 1 do not furnish a very satisfactory basis for evaluating the effects of various ingredients on performance. For this reason the results on trial mixtures, some of which are described in table 2, are considered to give a more adequate basis for estimating the effect of certain ingredients.



FIGURE 5.—Slumping of a calking material from joints between dense bricks (3 and 4) while the same plastic remains in place between porous bricks (1 and 2).

1. Vehicle

(a) Linseed Oil

Twelve commercial compounds contained blown linseed oil. Four of these gave satisfactory results in the strain and staining tests, but only one remained as soft as desired in a pat on limestone for 1 year. Samples which contained polymerized linseed oil were less satisfactory in some of the tests, but their hardening, as indicated in the last column of table 1, seemed to be less than for compounds prepared from blown oil. Six experimental mixtures were made from blown linseed oil of viscosities from Z1 to Z4, and four mixtures were made with polymerized linseed oils of Z3 to Z6 viscosities. All of these compounds gave satisfactory test results, but those with polymerized oils hardened less rapidly on limestone than the others. One of the three mixtures made with heavy-bodied polymerized linseed oil, thinned to lower viscosities with raw soybean oil, was unsatisfactory. Data on one trial mixture from each group is included in table 4.



FIGURE 6.—Amount of slumping in joints of various materials.

The compound slumped when placed between steel (1 and 1), steel and marble (1 and 2), marble (2 and 2), and marble and wood (2 and 3). The same compound did not slump when placed between wood (3 and 3), concrete (4 and 4), limestone (5 and 6), and brick (7 and 7). Considerable shrinkage and cracking occurred between the more absorptive materials.

(b) Soybean Oil

The commercial compounds in which treated soybean oil was the vehicle gave lower strain values than those containing bodied linseed oil. The correlation between the viscosities of the extracted oils and the tendency of the compounds to stain marble was better with this group of samples than with other oils. With one exception, pats of these compounds on limestone hardened upon standing in the laboratory.

Six experimental compounds were prepared from a commercially blown soybean oil of Z6viscosity. These were entirely unsatisfactory. Eighteen more were made with blown soybean oil of Z4 viscosity, and these gave satisfactory test results. With one exception, they remained plastic after 6 months in pats on limestone and stayed soft in the container. Penetration tests on two of these compounds showed an average hardening of 38 percent in 15 days. Experimental mixtures which used tung oil and soybean oil, polymerized to viscosities Y, Z2, Z3, and Z4, were satisfactory except when the Y viscosity oil was used. These compounds formed wrinkled skins, but retained their plasticity in the containers. The penetration test on these compounds showed an average hardening of 21 percent in 15 days. Data on three experimental mixtures with soybean oil as a vehicle are included in table 4.



FIGURE 7.—Shrinkage of three commercial samples of calking on glass and limestone for 30 days. Sample numbers correspond to serial numbers in table 1.

(c) Fish Oil

Blown fish oils were used as a vehicle in 22 of the commercial compounds. Only four of these failed to give strain-test readings above 0.05 in., but, like most of the compounds, hardened excessively when pats were left standing on limestone for 1 year.

Sixteen experimental compounds were made from commercially blown fish oils of V, Y, Z, and Z6 viscosities, but all were unsatisfactory. The viscosity of the lighter oils was increased by further blowing, and 36 experimental mixtures made from them. These were satisfactory in the physical tests and had a very slow rate of hardening, for only two became hard after 1 year on limestone. The viscosity of the Z6 fish oil was lowered by the addition of other oils, and 24 experimental compounds prepared. All were satisfactory in the physical tests and were still plastic after 9 months on limestone. Data on six of the experimental mixtures with fish oil as a vehicle are given in table 4.



FIGURE 8.—Progressive shrinkage of six commercial samples of calking on limestone for 10 months.





Panel of limestone with joints varying from $\frac{1}{2}$ in. to $\frac{1}{2}$ in. calked with one plastic mixture. Excessive shrinkage and cracking occurred in all joints less than $\frac{3}{2}$ in.



Sample numbers correspond to serial numbers in table 1.

	Pigment			Ve	hicle				Phy	zsical to	ests	Observations			
	Composition	-		Nonvolatile			Volatile	e		Stain	on "			of me-	
No.	Material	Amount	Amount	Kind	Oil vis- cosity	Amount	Kind	Amount	Strain	L i m e - stone	Marble	Shrinkage	Skin at 48 hours	Hardness pats on li stone ^b	
				MIXTURES	CON	TA	INING FI	зн (HL						
<u>E</u> 63	Precipitated whiting Talc Lithopone Asbestos, 7M	% 35 30 30 5	% }68	Fish oil blown with small amount of commercial drier.	Z3	% 32	None	%	in. 0. 35	OK	0K	Medium	Smooth	1 (13)	
E 159	Tale Lithopone Asbestos, 7M Asbestos, Fl	$ \begin{array}{r} 50 \\ 25 \\ 10 \\ 10 \\ 5 \end{array} $	60	$\begin{cases} Fish & oil & blown & at\\ 130^\circ & C. & 0.05\% & co-\\ balt & added & as & drier.\\ Varnish & 2\%. \end{cases}$	Z_4	37	{Mineral spirits.	} 3	. 20	OK	0K	do	do	1 (9)	
E18 8	Precipitated whiting Asbestine Titantox Asbestos, 7D	50 30 15 5	}62	$\begin{cases} Fish & oil & blown & at \\ 140^\circ C. & 0.1\% & cobalt \\ and & 0.2\% & lead & added \\ as & drier. \end{cases}$	Z_5	33	do	5	. 25	OK	OK	do	do	1 (7)	
E68	Ground limestone Tale Asbestos, 7M (Chalk whiting	$50 \\ 40 \\ 10 \\ 40$	63	Blown fish oil (com- mercial) and boiled linseed oil (9:1). (Blown fish oil (com-	Z_1	37	None		. 26	OK	ss	do	$ \{ \begin{array}{l} S \ l \ i \ g \ h \ t \ l \ y \\ wrinkled. \end{array} $	} 1 (13)	
E128	Talc Zinc sulfide Asbestos, Fl Precipitated whiting	30 15 15 40	}67	mercial) and raw lin- seed oil (24:1). 0.2% Mn added as drier. Blown fish oil (com-	} Z5	29	{Mineral spirits.	} 4	. 35	OK	OK	do	do	2 (10)	
E142	Lithopone Asbestos, Fl Asbestos, 7M	25 15 15 5	}54	seed oil (24:1). 0.05% cobalt added as drier Varnish 2%.	7.5	41	do	5	. 63	OK	OK	Slight	Smooth	2 (10)	
				MIXTURES C	ONT	AIN	ING SOYB	EA	z oir						
E 165	Precipitated whiting Tale Lithopone Asbestos, 7D	$50 \\ 25 \\ 15 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10 \\ 1$	58	Soybean oil blown at 120°C. 0.01% cobalt added as drier. Var- nish 2%.	Z_4	38	${ {{\rm Mineral}\atop {{\rm spirits}} } }$	} 4	0. 20	OK	OK	Medium	Slightly wrinkled_	} 1 (9)	
E173	Precipitated whiting Talc Lithopone Asbestos, 7H (Precipitated whiting	$50 \\ 25 \\ 15 \\ 10 \\ 50$	57	Soybean oil blown at 140° C., varnish, 2%. 0.08% cobalt added as drier.	$\left\{ Z_{2}^{2}\right\}$	43	None		. 27	OK	OK	Low	Wrinkled	1 (7)	
E 181	Talc Lithopone Asbestine Asbestos, 7M	10 15 15 10	63	Mixture of soybean and tung oils (4:1) heated to 285° C. 0.05% co- balt as drier.	Z_4	32	{Mineral spirits.	} 5	. 22	OK	OK	Medium .	Smooth	1 (6)	
				MIXTURES C	ONT.	AIN	ING PERI		OIL						
E59	Ground limestone Lithopone Asbestos, 7M		72	{Blown perilla oil (com- mercial).	26	22	{Mineral spirits.	6	0.18	0K	OK	Medium .	Smooth	3 (13)	
E 92	I ithopone Talc Asbestos, 7M	35 30 30 5	}70	{Mixture of blown pe- rilla oil (commercial) and raw tung oil.	$\left. \right\} Z 5$	25	do	5	. 25	0K	OK	do	$\left\{ egin{array}{c} { m Slightly} \ { m wrinkled}. \end{array} ight.$	2 to 3 (11)	
				MIXTURES	CON	TAI	NING LIN	ISEI	ed oi	L		,			
E 137	Precipitated whiting Talc Lithopone Asbestos, <i>Fl</i> Asbestos, <i>7M</i>	$40 \\ 25 \\ 15 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10 \\ 10 \\ 1$	53	Mixture of polymer- ized linseed and raw soybean oils (3:1).	z_4	42	{Mineral spirits.	} 5	0. 30	0K	0K	Medium .	Smooth	1 (10)	
E_{155}	Precipitated whiting_ Talc Lithopone Asbestos, Fl	$ \begin{array}{r} 40 \\ 25 \\ 15 \\ 10 \\ 10 \\ 10 \end{array} $	48	Polymerized linseed oil. 0.05% cobalt added as drier. Var- nish 2%.	$\left. \right\} Z5$	46	do	6	. 38	OK	OK	do	$\begin{cases} S \ light ly \\ wrinkled. \end{cases}$	} 1 (9)	
E 183	Precipitated whiting	50 25 15 10	59	$ \begin{cases} \mbox{Linseed oil blown at} \\ 120^\circ \ C. \ 0.24\% \ lead \\ \mbox{and} \ 0.03\% \ manganese \ added \ as \ driers. \end{cases} $	$\left\{ Z_{4}\right\}$	36	do	5	. 24	OK	OK	do	do	1 (7)	

TABLE 4.-Composition and physical studies on 14 experimental mixtures

 $^{\circ}$ OK=satisfactory; SS=slight strain. $^{\circ}$ Numerals signify hardness, namely, 1=satisfactory, 2=hard, 3=very hard. Two figures, 1-2, signify intermediate hardness. Numbers in parentheses indicate the time (in months) the pats have been on linestone.

(d) Tung Oil

The five commercial compounds which contained tung oil also contained resins and may be classed as varnish base compounds. These had good adhesion, remained plastic, and had excellent workability. A small amount of tung oil was bodied in the laboratory and used for preparing four experimental compounds. These were inferior to the commercial compounds, formed wrinkled skins, and were unsatisfactory in the physical tests.

(e) Miscellaneous

All of the compounds grouped in table 1 under "Miscellaneous" were unsatisfactory except 28 A-g, which contained rosin.

Seventeen experimental mixtures were made from commercially blown perilla oil of Z6 viscosity, and seven mixtures were made with a commercially blown rapeseed oil of the same viscosity. Although some of these preparations were adhesive enough to pass the strain test, all but one of them became hard upon standing. When the viscosity of these two oils was lowered by the addition of other oils and the resulting mixtures used to prepare experimental compounds, the preparations made from blown perilla oil were slightly better than the six made from blown rapeseed oil, but all of them hardened too rapidly.

2. Pigments

As shown in table 1, the pigments consisted of a number of materials, some of which were finely ground, the others being fibrous. The fine-ground portion included calcium carbonate in one of its several forms, talc, quartz, and diatomaceous silica; to these lithopone and zinc oxide were sometimes added to produce lightcolored compounds. The fibrous portion was generally short fiber asbestos, but cotton fibers were found in two compounds.

Of 11 commercial samples which contained calcite alone as the pigment, 7 were knife-grade

and 4 were gun-grade. All of the knife-grade and two gun-grade samples showed poor bonding properties. In the experimental mixtures it was found that precipitated whiting as the entire pigment produced compounds that hardened rapidly and had poor bonding properties, as in the case of the commercial samples. On the other hand, compounds made with other types of calcium carbonate as the entire pigment, such as chalk whiting or ground limestone, showed fairly good plastic and adhesive properties when mixed with the proper oils. However, one outstanding property common to all calking mixtures containing only calcium carbonate as the pigment was their tendency to slump. The addition of about 10 percent of any one of the medium-fine grades of asbestos (7D, 7K, or 7M) overcame the slumping, but an equivalent amount of the very fine grades of fiber did not.

A pigment which gave good results in several experimental mixtures was composed of 50 percent of precipitated whiting, 30 percent of tale, 10 percent of lithopone, titanox, or zinc sulfide, and 10 percent of asbestos.

3. Thinners

The amount of volatile matter in commercial compounds varied within wide limits. Eleven samples contained 1 percent or less, one compound 37 percent, and the average of the entire group was 9.4 percent. Mineral spirits was the most commonly used thinner, although $25 \ A-k$ and $25 \ B-g$ contained large amounts of kerosene.

The amount of volatile used in experimental mixtures depended largely on the viscosity of the oil. With viscosities of Z4, Z5, and Z6, the volatile varied between 5 and 7 percent. Oils of lower viscosities required less volatile and, in many cases, thinner was unnecessary. When higher percentages of volatile were used, the mixtures showed little or no adhesion, high shrinkage, and they hardened rapidly.

Five mixtures ⁸ were made similar in all respects with the exception that the volatile varied from zero to 16 percent in equal steps. The various mixtures were placed in ¼-in. joints between slabs of limestone. Cracks formed in all mixtures with more than 4 percent of volatile. Strain tests and penetration tests were also made on these mixtures at the end of 15 days. The results are shown in table 5.

TABLE 5.—Strain and penetration tests

Sample	Volatile	Strain	Penetra- tion					
	Percent	Inches	0.01 cm					
1	0	0, 26	>155					
2	4	, 23	110					
3	8	. 03	74					
4	12	. 02	0					
5	16	. 02	õ					

Of the commercial compounds in table 1 containing more than 15 percent of volatile, only two remained satisfactorily soft in pats on limestone for 1 year.

VII. RELATION OF PHYSICAL PROP-ERTIES TO PERFORMANCE

The period of time during which a ealking compound will serve its purpose depends on the ability of the compound to retain certain physical properties such as adhesiveness, cohesiveness, and plastieity. Adhesiveness supplies the necessary bonding property, whereas cohesiveness enables the compound to resist rupture within itself. The plastic condition depends on the fluid ingredients in the mixture. Volatile constituents may soon evaporate, thus eausing shrinkage and stiffening of the compound. Porous materials in contact with the ealking compound may extract some of the nonvolatile fluid, causing further hardening and shrinkage.

Changes in eonsistency can be measured as described in section IV, 2 (d) of this report. Such determinations on commercial materials have shown that a large change in consistency occurs within a period of 2 weeks. Observa-

⁸ Base composition as follows:

Oil: Three parts blown fish and 1 part raw tung heated to 270° C. (viscosity=Z4).

Pigment:	Precipitated whiting	60%
	Talc	25%
	Zinc sulfide	10%
	Asbestos 7K	5%

tions on exposure specimens indicate that this is a continuous process which may render the rapidly hardening compounds useless in less than a year, whereas some remain sufficiently soft to function for several years.

Shrinkage indicates that the compound has lost a portion of the vehicle. The first manifestation of shrinkage is the formation of eoneave joints or eraeks. Some compounds also form voids within the mass, which may be the result of shrinkage. Regardless of whether shrinkage is accompanied by hardening, it is undesirable, because it places the joint filler in tension even when there are no structural movements. Figure 11 shows several rigid joints in which shrinkage has caused eraeks. These usually occur at the sides of joints, but sometimes they form within the compound.

When a joint is in tension, brought about by shrinkage or a structural movement, the ealking should stretch without breaking the bond. The forces are transmitted to the joint filler by means of its bond; hence the adhesive property is very important. The most common type of failure observed in test joints and in buildings was that of ruptured bonds. A satisfactory means of determining the actual bonding strength of plastic substances has not been found. The test for bond outlined in section IV, 2 (b) is not quantitative, but enables one to distinguish between satisfactory and unsatisfactory bonding properties.

The formation of cracks within the plastic indicates that the eohesive strength is less than the bonding strength. Mixtures of high-volatile content are apt to become somewhat brittle or deficient in cohesive strength after a short time. The test for tenacity described in section IV, 2 (c) affords a means of determining this property.

The density of proprietary compounds varies from about 1.3 to 2.5 (10.8 to 20.8 lb to the gal). Since the density of the materials composing various products does not vary greatly, it seems logical to believe that those of low weight contain more entrapped air than the heavy compositions. If this is true, a lowdensity product may be expected to harden more rapidly than one of high density, provided the oil contents of the two are identical, because the entrapped air will cause some oxidation of the oils within the mass. A high-density compound is more apt to flow or slump in the joints than a low-density material of the same consistency. Therefore, the high-density material must be made somewhat stiffer to overcome slumping, and this may add materially to the cost of application. Since the data obtained in the shrinkage test enables one to compute the density, this should be done for additional information.

VIII. PRIMERS AND THEIR EFFEC-TIVENESS

Various types of primers are used with plastic calking materials. They are intended to seal the pores of the masonry or other building material with which the calking compound is to be placed in contact. Shellac is one of the common primers; but thinned oils, thinned varnish, aluminum soap solutions, and waterglass are also used. It seems to be a common practice to coat the sides of joints with the primer and then insert the calking without allowing a sufficient time for the priming solution to dry. It is doubtful if any primer will seal the pores effectively if the primer is not allowed to dry. If the plastic is applied to joints treated with shellac or waterglass before the latter are dry,

the bond may be adversely affected. The drying time of any primer is affected somewhat by the type of surface to which it is applied.

Workmen claim an additional advantage for primers in that they "lay the dust" in joints and thus permit a better bond to be obtained. Dust interferes with bonding, but it can probably be removed at less cost by other means.

Studies were made on 11 commercial primers by applying them to limestone 24 hr before the calking materials were applied. Shrinkage, tenacity, and bond determinations were made on the calking materials after 60 days and compared with those from similar tests in which no primer was used. The results are given in table 6. Five brands of calking with volatile contents from approximately 8 to 22 percent were used in these tests.

The slight reductions in shrinkage caused by various primers cast considerable doubt on their value for improving the performance of calking materials. This is especially true when the calking compound has a high volatile content. In some cases the bond and tenacity were affected adversely. Primers which materially reduced the shrinkage without seriously affecting the other properties were thinned oils, thinned varnishes, and tung-oil paint.

TABLE 6.—Results of tests with calking materials on stone primed with various commercial primers in comparison with the same calking on unprimed stone

	Calking samples 1												
Primer used on stone sur-	18 A-g (7.8%)		15 A-g (21.9%)		11 A-g (8.8%)			14 D-g ⁴ (12.0%)	5703 (8.7%)				
face *	Shrink- age	Tenac- ity ³	Bond 3	Shrink- age	Tenac- ity ³	Bond ³	Shrink- age	Tenac- ity ³	Bond ³	Shrink- age	Shrink- age	Tenac- ity ³	Bond ³
None a b d	% 22 21 20 19 21	OK OK OK F	$OK \\ OK \\ F \\ OK \\ F \\ F$	% 38 38 37 35 37	$F \\ F \\ F \\ F \\ F \\ F$	OK OK F OK F	% 30 24 21 21 28	OK OK OK OK	OK OK OK F	% 26 23 22 20 25	% 24 23 23 19 24	OK F OK OK F	OK OK F F OK
e f g h i	18 18 19 19 23	OK OK OK OK	OK OK OK OK	36 36 37 36	$F \\ F \\ F \\ F \\ F \\ F$	OK OK OK OK	20 20 23 27 28	OK OK OK OK	OK OK OK F	20 20 23 24 26	$20 \\ 20 \\ 23 \\ 22 \\ 24$	OK OK OK F F	OK OK OK OK
j k	$\frac{20}{19}$	${OK \atop OK}$	$OK \\ OK$	38 38	$F \\ F$	$OK \\ OK$	$ 28 \\ 20 $	$\begin{array}{c} OK \\ OK \end{array}$	${}^F_{OK}$	$\begin{array}{c} 23\\22 \end{array}$	$\begin{array}{c} 22\\18\end{array}$	${OK \atop OK}$	${}^{OK}_{OK}$

¹ Serial numbers in table 1 with percentage volatile (weight loss in 60 days) in parentheses. Sample 5703 is not listed in table 1. ² Primers a, b, and j are essentially aluminum-soap solutions. Primers e, f, and h are thinned oils containing aluminum soap. Primers e, f, and h are thinned varnishes.

Primers c and k are shellac and tung-oil paint, respectively. F signifies failure; OK, satisfactory.

Tenacity and bond were not determined for this sample. The test had not been developed at the time these measurements were made.

FIGURE 10.—Effect of priming upon staining property of calking compounds.
Panel of limestone in which the joints were primed for half of the length (right half) and the other half left unprimed, then all filled with a calking mixture of pronounced staining quality. Primers used were water-glass (top), thinned oil (middle), and shellac (bottom).

The results shown in figure 10 indicate that the performance of a staining compound can be improved somewhat by priming, but that primers do not entirely prevent the absorption of oil or the formation of cracks. A point that should be considered is the durability of the primer. Solutions of waterglass (silicate of soda) tend to decompose, on drying and exposure to the air, into silica (more or less hydrated) and sodium carbonate; the silica layer on the masonry may reduce the bond between the calking compound and the masonry and the sodium carbonate may saponify some of the oil in the compound. Shellac is not very durable when exposed to the weather, and there is not much protection to it in a calked joint on the outside of a building. However, it has some desirable features for this purpose in that it dries quickly and forms a film that is temporarily almost impermeable. In general, primers may be considered of value only in preventing or reducing the absorption of oil by porous materials. They do not prevent shrinkage caused by volatile matter in the calking; hence a nonstaining compound cannot be expected to serve its purpose any better in primed joints than in unprimed joints.

IX. DURABILITY

The weathering and aging effects were studied by means of exposure tests on the roof of a building and inspection of actual installations. The indications are that oil mastics do not usually deteriorate by crumbling, as do some masonry mortars, but fail by gradual hardening, bond rupture, or cracking within the compound. Those materials which contain an excess of pigment often form a network of fine cracks and become almost as hard as mortar. Such compounds would probably disintegrate after a period of years, but they fail to function as plastic joint fillers in a much shorter time. In the exposure tests, brittle films formed on the surface of a few specimens, which finally broke and scaled off.

1. EXPOSURE OF STRAIN-TEST SPECIMENS

One series of exposure tests consisted of joints between slabs of limestone, slate, and marble and also between five grades of brick. These accessory materials were chosen for a large range in porosity, the percentage of water absorption being approximately as follows: Limestone, 5; slate, 0.35; marble, 0.16; and the bricks ranged from 1 to 12. Seventeen commercial brands of calking materials made by the larger producers were used. The types of oil used in these compounds were not known. All were gun grade except two. The volatile varied from 1 to 14 percent and pigment from 46 to 85 percent. Six compounds contained whiting in amounts varying from 2 to 79 percent of the total pigment, and seven compounds contained rosin. Several joints between limestone slabs were made with each brand of calking material.

Strain tests were made on the limestone specimens after aging, as follows: first set cured for 30 days indoors; second set exposed to the weather for 30 days; third set exposed to the weather for 1 year. There was no appreciable difference in the results for specimens cured indoors 30 days and those exposed to the weather for 30 days.

Joints made from two brands of calking showed no retrogression after 1 year's exposure, but the other samples showed decreases in strain values ranging from 12 to 100 percent and averaging 60 percent. Joints in the very dense materials (marble and slate) for most tests gave higher strain values than the joints in limestone. However, the results on joints between the dense brick were usually not as high as for joints between more porous grades. Since the removal of vehicle from the plastic may seriously affect durability, the lower strain value with the dense brick seems to indicate that the capillarity of these was greater than for the more porous grades. Very dense materials like marble probably exert a strong capillarity, since they often show oil penetrations extending an inch or more away from the joints. However, the amount of oil removed from the plastic in producing such stains is small, because the total porosity of the marble is very low.

Observations on the specimens that had been exposed to the weather for 1 year gave some indications of the causes of deterioration. In several specimens the plastics had started to separate at the sides of joints on account of shrinkage and poor bond. Decreases in strain values on aging may indicate that the materials have hardened excessively or that the bonding value has retrogressed.

2. Exposure of Rigid Joints

Seventeen brands of calking materials in grooved blocks of limestone have been studied after exposure to the weather for periods varying from 2 to 8 years. The stone blocks measured 4 by 4 by 2½ in. with two grooves ¾ in. wide and ¾ in. deep. Both grooves in each were filled with one sample of calking, the only difference being that one groove was primed and the other unprimed. Some brands of calking in the exposures were represented by more than 100 specimens. The inspection of these specimens consisted in rating them as to tightness of the joints, consistency of the calking, and general appearance.

Most brands showed large differences in results for different samples. This may be partly accounted for by the fact that some manufacturers produce compounds of several different compositions. However, there are also marked variations in the products of manufacturers who apparently use only one formula. For instance, a firm which evidently uses 1 formula has 75 specimens in these tests. There are 21 specimens in the group that have been exposed for an average of 7 years, and 15 of them were judged to be in a passable condition. In the group exposed for an average of 3 years there were 17 specimens, and only 7 of them were rated as passable. Similar variations occur in most other brands, as illustrated in figure 11.

A few specimens of various products were found to be in good condition after exposure for 6 or 8 years. The composition of the calking materials used in these tests was not determined, and hence it is not possible to draw any definite conclusions regarding the effects of various ingredients on durability. Conclusions based on the assumption that any brand was made according to a single formula over a period of years are of doubtful value. The tests give indications that it is possible to make a plastic calking compound that will give satisfactory service for more than 8 years, but for consistently satisfactory performance it may be necessary for some producers to control the fabrication process more carefully.

One conclusion that seems to be justified by these tests is that coloring pigments are detrimental to durability. All specimens in which the calking was white or blue in color were very hard and showed a network of fine cracks.

3. INSPECTION OF BUILDINGS

Inspections were made of calking materials in 33 structures located in 18 cities, the farthest south being Galveston, Tex., the most northerly being Williamsport, Pa. The brands of calking were known for most of these and 15 different



FIGURE 11.—Rigid joints in limestone after exposure to the weather.

Two specimens of each brand were selected from a large number to represent the best results (left) and the poorest (right) for each of 12 products. The numbers above each group signify the same brands as in table 1.

products were represented, but no specific information regarding their composition was available. The plastics were in contact with various materials, such as granite, marble, limestone, brick, mortar, steel, bronze, and wood.

The observations on actual installations agree, in general, with laboratory results and observations on exposure specimens in that the plastics bond somewhat better to porous materials than to nonporous materials or to materials of very low porosity. Plastic joints between brick and steel were noted where excessive shrinkage had caused the calking compound to separate from the steel. An instance of slumping, as in figure 6, was found where a gun-grade calking material was used between marble and metal. Excessive hardening of the plastics in marble construction was noted where sufficient coloring pigment had been used in the compounds to produce very light colors. In several cases it appeared that the calking was less durable in contact with mortar than where it was in contact with stone. The calking in one case where it was used between sheets of lead had failed completely. The installations examined were from 2 to 8 years old and in about 50 percent of these the calking appeared to be in good condition, whereas that in about 20 percent was poor. These observations indicate that calking compounds can be made which will give good service for more than 6 years. No definite differences in performance could be attributed to different climatic conditions.

X. SUMMARY

Seventy-two commercial brands of plastic calking compounds were examined by chemical and microscopic methods, and the effects of different treatments of oils in numerous trial mixtures were studied by means of certain laboratory tests.

The compounds were studied (1) by laboratory tests simulating the conditions of service, (2) by exposure tests, and (3) by inspection of several structures showing the materials in use for periods up to 8 years.

New tests have been devised which are simpler than the tests now in use, and the properties so determined are thought to give more specific information on quality.

Certain conditions affecting the durability and performance have been given attention. In this connection various types of primers have been tested to determine their effects on shrinkage, bond, and tenacity of the plastic calking compounds. The performance of these products on various types of building materials with particular reference to porosity and capillarity has been studied. Although the work has not been completed, the following statements seem to be justified:

1. A considerable variety of ingredients can be utilized in the plastic calking compounds with good results. For the vehicle certain blown oils such as soybean, cottonseed, linseed, and fish seem to give the most satisfactory results. When mixed with properly treated oils, the pigments may consist of any of the common fillers, such as calcite, talc, asbestine, etc., with asbestos fibers. These can be used in almost any proportions provided that not less than 5 percent is fiber of a suitable grade. Coloring pigments—such as lithopone, zinc oxide, or titanox—should be limited to amounts that will not cause the compound to craze or harden too rapidly. Volatile thinner in the vehicle causes shrinkage of the compound and if used in excessive amounts, "bleeding," premature hardening, or loss of tenacity may occur.

2. Physical qualities desired are low shrinkage, low rate of hardening, tenacity, adhesiveness, and ability to retain the vehicle; also, the consistency should be such that the compound will work easily but not flow, after placing in the joints. The formation of a skin within 3 days after placing in the joints is desirable.

3. Hardening of the plastics reduces their ability to function under service conditions and causes higher bonding stresses when the joint is in tension. The average rate of hardening of 93 samples of proprietary compounds was 55 percent, the maximum being 87 percent and the minimum 15 percent. Since the value of a calking compound depends on its remaining soft, the rate-of-hardening test is believed to be a good indication of durability.

4. Shrinkage is caused mainly by evaporation of volatile matter and absorption of a part of the vehicle by porous materials in contact with the calking. In the usual commercial compounds, shrinkage continues over several months and sometimes amounts to more than 25 percent of the original volume. The first manifestation of shrinkage of calking compounds is the formation of concave joints or cracks. Excessive shrinkage causes separation from the sides of joints or, in materials of poor tenacity, the formation of cracks within the plastic. The average shrinkage of trade products for 15 days in contact with limestone was, for 104 gun-grade materials, 22 percent; and for 26 knife-grade materials, 8 percent. Since shrinkage causes strains in the calking sufficient, in many cases, to cause bond failures regardless of structural movements, it seems evident that a low limit for shrinkage should be a requirement.

5. The adhesion or bonding value of plastics varies appreciably with the type of adjacent material, that is, they bond more satisfactorily to porous materials than to materials of low porosity. The value of the compound in this respect seems to depend to some extent upon the type of pigment (filler) used.

6. The consistency required for different installations may vary somewhat with such considerations as joint widths, the type of material in contact with the calking, and the temperature at the time of application. If the calking does not sag or flow within a few hours after the joints are filled, sagging is not apt to occur later. Troubles of this kind are more frequent in joints between nonabsorptive materials than in joints between absorptive materials. The addition of thinner, as sometimes practiced at the building site to make compounds more workable, is apt to cause undesirable results. For cold-weather application the producers recommend softening the calking by setting the container in hot water.

7. Staining of porous materials by plastic calking indicates that oils were of too low viscosity or that too much thinner was used in the compound. Staining of masonry materials by the calking is objectionable in two ways:
 (1) The appearance of the masonry is impaired.
 (2) If appreciable amounts of oil are removed from the calking, shrinkage and hardening result. Oil stains in materials having a porosity

of 10 percent or more usually disappear after a few months of weathering, but in denser materials they disappear very slowly.

8. The density of commercial calking mixtures varies from 1.3 to 2.5 (10.8 to 20.8 lb per gal). The lighter mixtures probably contain a considerable amount of entrapped air which may increase the shrinkage and rate of hardening.

9. Priming the surfaces of porous materials before placing the calking may reduce staining and adversely affect bond and tenacity. When primers are used, they should be allowed to dry before the calking is applied. Tests on various primers indicate that thinned varnishes and thinned oils are the most satisfactory types.

10. Inspection of masonry structures and exposure specimens indicates that it is possible to make calking compounds that will last for at least 8 years. Studies on specimen joints that have been exposed to the weather for several years indicate that each of the trade preparations varied in quality, resulting in large differences in durability.

WASHINGTON, July 21, 1939.

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