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16. ABSTRACT
The development of a prototype rapid set epoxy adhesive to bond raised pavement markers to Portland cement concrete and asphaltic concrete in California freeways and secondary roads was first reported in June 1969 as State Specification 68-F-44. Since then, four formula revisions have been made to improve adhesion and application characteristics. The current specification, designated as State Specification 721-80-42, has been in use since 1972.

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DEPARTMENT OF TRANSPORTATION
DIVISION OF HIGHWAYS
TRANSPORTATION LABORATORY
5900 FOLSOM BLVD., SACRAMENTO 95819

July, 1974

Translab No. 635150
Federal No. D-5-15

Mr. R. J. Datel
State Highway Engineer

Dear Sir:

Submitted herewith is an interim research report titled:

DEVELOPMENT OF A RAPID SET EPOXY ADHESIVE

FOR CALIFORNIA HIGHWAY MARKERS

By

D. R. Chatto and T. L. Shelly

Under the Supervision of

Donald L. Spellman, P. E.
Chief, Concrete Section

Very truly yours,

JOHN L. BEATON
Chief Engineer, Transportation Laboratory

Attachment

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The investigators express gratitude to the late Mr. Herbert A. Rooney for guidance and consultation, and to Mr. B. J. Chapman for assisting in testing.

The contents of this report reflect the views of the Transportation Laboratory which is responsible for the facts and the accuracy of the data presented herein. The contents do not necessarily reflect the official views or policies of the State of California or the Federal Highway Administration. This report does not constitute a standard, specification, or regulation.

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INTRODUCTION

In 1965, California adopted the system of raised pavement markers for all freeways and a majority of secondary roads. A standard set epoxy adhesive initially used to bond the markers to the pavement, although adequate in performance, was much too slow curing. This resulted in long lane closures which were hazardous in areas where markers were applied to heavily travelled highways. Also, marker installation at temperatures below 50°F was almost impossible. To alleviate the long lane closures and permit markers to be applied at temperatures down to 40°F, the Transportation Laboratory developed an initial rapid setting epoxy adhesive designated as 68-F-44.

Performance of this adhesive when applied by marker placement contractors, under field conditions using automatic mixing machines, lead to a considerable number of adhesive failures. A substantial portion of these failures could be traced to improper proportioning and mixing of the epoxy components but failures also occurred when mixing and proportioning were apparently within specification limits.

Field complaints about the pumping consistency and difficult mixing and proportioning, especially at temperatures around 50°F or less, led to several changes in filler loadings, flexibilizers, and reactive diluents. This report covers the work done in an attempt to improve both the adhesion and flow properties of the original rapid set adhesive.

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CONCLUSIONS

The shortcomings of the first fast setting epoxy adhesive formulated for pavement marker application have been overcome. The falling ball impact strength of the original formula was about 2 ft.lbs. The percent linear shrinkage of the original formula was 0.0144%. The revised formula, 721-80-42, has an impact strength of about 9 ft.lbs. and a shrinkage of 0.0002%.

The use of a small amount of O-cresyl glycidyl ether as a reactive diluent in the A component and the use of resin grade asbestos in both components, have resulted in the required viscosity and thixotropy necessary for good application properties both at low temperatures and at high temperatures.

We have no reports of failures in the field which can be attributed to poor physical properties of this adhesive in over two years of use.

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IMPLEMENTATION

Improvements made in the original rapid set epoxy adhesive formula 68-F-44 have been written into the current specifications 721-80-42. This adhesive system is being manufactured on a contract basis and used whenever conditions require a rapid setting marker adhesive.

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EFFECTIVE SHRINKAGE

High shrinkage rates, primarily during the gelation stage, lead to glue line stresses of considerable magnitude. The rapid set adhesive, 68-F-44, exhibited much higher effective shrinkage than did the slower setting standard epoxy, i.e., 0.0144%, compared to 0.0002% for the standard set epoxy. This was considered to be one of the prime factors contributing to some of the marker losses in road service, especially where the failure had occurred at the glue line interface between the bottom of the marker and the epoxy adhesive.

The percent effective shrinkage was measured by the glass bow method and is described in the Appendix.

Table 3 shows the effect of various amounts of certain fillers on the percent shrinkage. Filler loadings do not appear to affect the shrinkage to any appreciable extent. The reactive system with LP3 has the greatest effect on the shrinkage. The polysulfide polymer LP3 Thiokol shows a pronounced improvement in lowering the shrinkage at 35 phr (parts per hundred of resin).

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IMPACT STRENGTH

Impact strength of the first rapid set adhesive, 68-F-44, is about 2 ft.lbs. The test is basically a gravity impaction of a one pound steel ball on a cured specimen 0.30-inch thick and 1.75 inch in diameter. Examination of Table 3 shows the effect of the LP3 polysulfide polymer and Mobisol 66 on the impact strength.

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THERMAL CYCLING

Some indication of glue line stability was obtained by covering the bottom of a ceramic marker button with an epoxy layer about 1/2-inch thick, curing 24 hours, and then cycling the button from -10°F to 77°F, about 4 hours at each temperature. The adhesive, 68-F-44, with high shrinkage and low impact resistance usually separated at the glue line between the epoxy and the bottom of the ceramic marker after less than 4 cycles. The low shrinkage, high impact resistance formula with 35 phr LP3 Thiokol, after 8 cycles, showed no signs of glue line separation or spalling. Although this is an empirical test, we believe it has some correlation with road performance and adhesion, especially with regard to the hard line at the marker/pavement surface.

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FLOW PROPERTIES

Pavement marker epoxy adhesives usually are proportioned, mixed, and extruded by automatic mixing equipment. Flow properties of adhesive, i.e., viscosity and thixotropy at various temperatures, are important not only for proper proportioning, mixing, and extrusion, but also to prevent excessive flow of the extruded adhesive from under the marker when placed in position in the roadway.

Two thixotropes have been used in the rapid set system, Thixatrol ST, a hydrogenated castor oil, and RG-144, a resin grade asbestos.

Initially, the rapid set adhesive contained RG-144 asbestos for flow control, and it performed satisfactorily. Next, Thixatrol ST was tried as a replacement for RG-144 in the laboratory mixes, although heating of the batch to at least 160°F was necessary to dissolve it. Actually the adhesive had to be heated during manufacture to facilitate the de-aeration of each component so that proper density could be obtained. This formulation pumped and mixed easily, and at first it was thought that it might be superior to RG-144 since it gave a "smoother" product rather than a slightly granular structure obtained with RG-144. However, field complaints that the adhesive was too thin and had excessive after-drip at the extrusion nozzle required several revisions of the formula to increase the viscosity by raising the Thixatrol level. Formula 711-80-44 (Table 1) contained a Thixatrol level high enough to prevent excessive run and after-drip, but developed a "false set" or gelling in the shipping containers. Gelling made loading of the mixing machine very difficult since the material would not "pour" into the holding tanks. Thixatrol ST was deleted from the next revision of 712-80-44 and RG-144 asbestos substituted. There have been no further field complaints on the flow properties of the lastest revision, 721-80-42.

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SHEAR STRENGTH AND MOISTURE SENSITIVITY

Slant shear strength, as determined by Test Method No. Calif. 425, of the original rapid set adhesive (68-F-44) was about 4000 psi. After a water soak for one week, the strength dropped to about 3500 psi. On the latest revision (721-80-42), the shear strength was about 2000 psi, dropping to about 1200 psi after a one week water soak. Improvement of the impact and shrinkage properties has meant an increase in moisture sensitivity. Long term wet shear strength values up to two years water immersion are shown in Figure 3. Also plotted on Figure 3 are water absorption values of the epoxy adhesive. Of interest is the fact that after two years immersion, the shear strength was 150 psi. A duplicate shear block was removed from the water and dried for 18 hours at 120°F and the strength increased to 550 psi. This semi-reversible reaction should be an advantage in road service where markers are seldom completely immersed and if so, for only short periods of heavy rainfall. However, removal of markers has shown that the concrete under the marker is damp for at least several weeks after a rainfall. Therefore, if any failure would occur that was related to the epoxy, one possible cause would be moisture sensitivity of the adhesive.

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TABLE 1

COMPOSITIONS OF RAPID SET SPECIFICATIONS

Material	Parts by Weight					
	68-F-44	701-80-44	702-80-44	711-80-44	712-80-44	721-80-42
Component A						
Shell 828	100	100	100	90	90	90
Epi-Rez 5011				10	10	10
Reg-144 Asbestos	3				3	3
Thixatrol ST		4	5	6		
TiO_2	1.6	3	3	3	3	3
Talc				50	50	50
Antifoam Q		0.01	0.01	0.01	0.01	0.01
Component B						
Dion 3-800 LC	60	60	60	60	60	60
DMP 30	6.0	7.0	7.0	7.0	7.0	7.0
Mobilsol 66	15	14.7	14.7			
2 Ethyl Hexanoic	0.2	0.15	0.15			
Thiokol LP 3				35	35	35
Ballotini Spheres	36.4					
RG-144 Asbestos	2.0				3.0	3.0
Thixatrol ST		0.75	1.0	2.0		
Talc		45.5	47.6	61.9	50.6	50.6
Carbon Black	0.05	0.05	0.05	0.05	0.05	0.10
Antifoam Q		0.01	0.01	0.01	0.01	0.01

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TABLE 2

TYPICAL PROPERTIES OF RAPID SET SPECIFICATION ADHESIVES

Property	68-F-44	701-80-44	702-80-44	711-80-44	712-80-44	721-80-42
Viscosity, cps, A	90,000	80,000	280,000	460,000	250,000	250,000
Viscosity, cps, B	130,000	140,000	280,000	520,000	300,000	300,000
Viscosity, cps, Mixed	100,000	75,000	185,000	320,000	160,000	160,000
Shear Ratio A	2.3	2.0	2.1	2.7	2.5	2.5
Shear Ratio B	2.3	2.1	2.0	2.3	2.6	2.6
Shear Ratio Mixed	1.7	2.2	2.1	2.5	2.6	2.6
Pot Life, Minutes	5	5	5	8	7	7
Rate of cure to 200 psi, Minutes	17	17	17	25	30	30
Slant Shear, 24 hrs., psi		4200	4000	2000	2000	2000
Slant Shear, 24 hrs. + 7 days water, psi		4000	3600	1200	1200	1200
Water Absorption:						
7 Days, %	2.4	2.0	1.8	2.0	2.2	2.2
Tensile, psi				1700	2000	2000
Elongation, %				20	20	20
Hardness, Shore D	80	75	78	73	73	73
Impact Strength, ft.lbs.	2	3	2	9	9	9
Effective Shrinkage, %			0.0144	0.0002	0.0003	0.0003
Tensile Bond to Ceramic Marker, psi	2000	2000	2000	1250	1400	1400
Tensile Bond to Reflective Marker, psi	800	800	800	800	800	800
Tensile Bond to Concrete psi	600	600	600	600	600	600
After 7 days water, psi	500	500	500	400	400	400

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TABLE 3

IMPACT RESISTANCE AND SHRINKAGE VS. FILLER TYPE AND LOADING

Impact Strength, Ft. Lbs.	Percent Shrinkage	phr Loading*						
		Ballotini	Mobilsol 66	Talc	LP3	Versamid 140	EM 308	Mica
2	0.0144	115	14.7					
	0.0095	50		124.6	20			
	0.0022				20			
	0.0220					20		
	0.0113					10	10	
	0.0010					25	25	
8	0.0005			161	30			
8	0.0006	37		119.6	30			
	0.0020			111.6	30			50
9 +	0.0009			61.7	30			30
9 +	0.0008				30			91.9
9 +	0.0004				35			101
9 +	0.0002			101	35			
	0.0002			106.5	35			
	0.0007			136.7	25			

*With 90 parts Shell 828 + 10 parts ER 5011 (O-cresyl glycidyl ether).
Curing agent 60 parts Dion polymercaptan + 7 parts DMP 30

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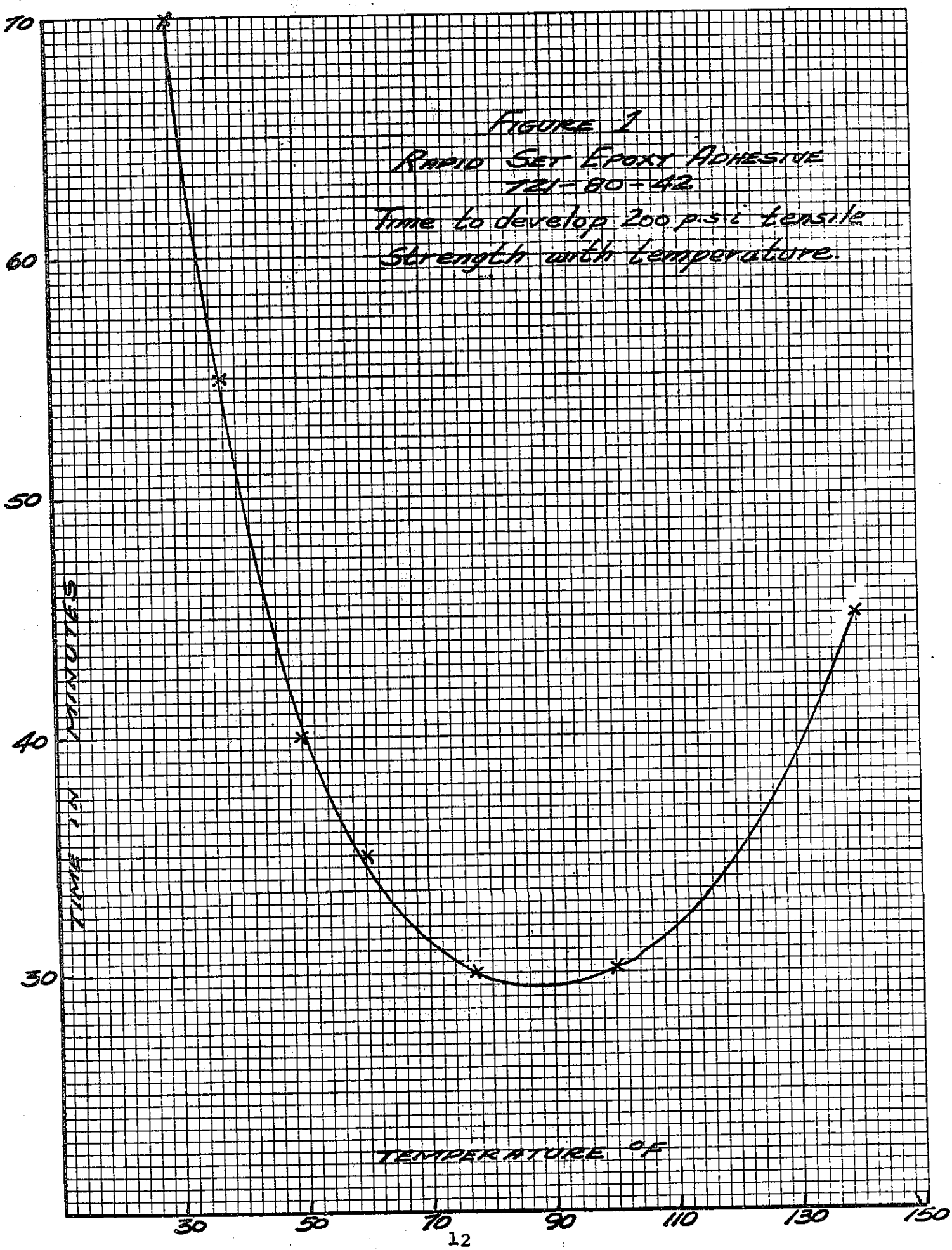

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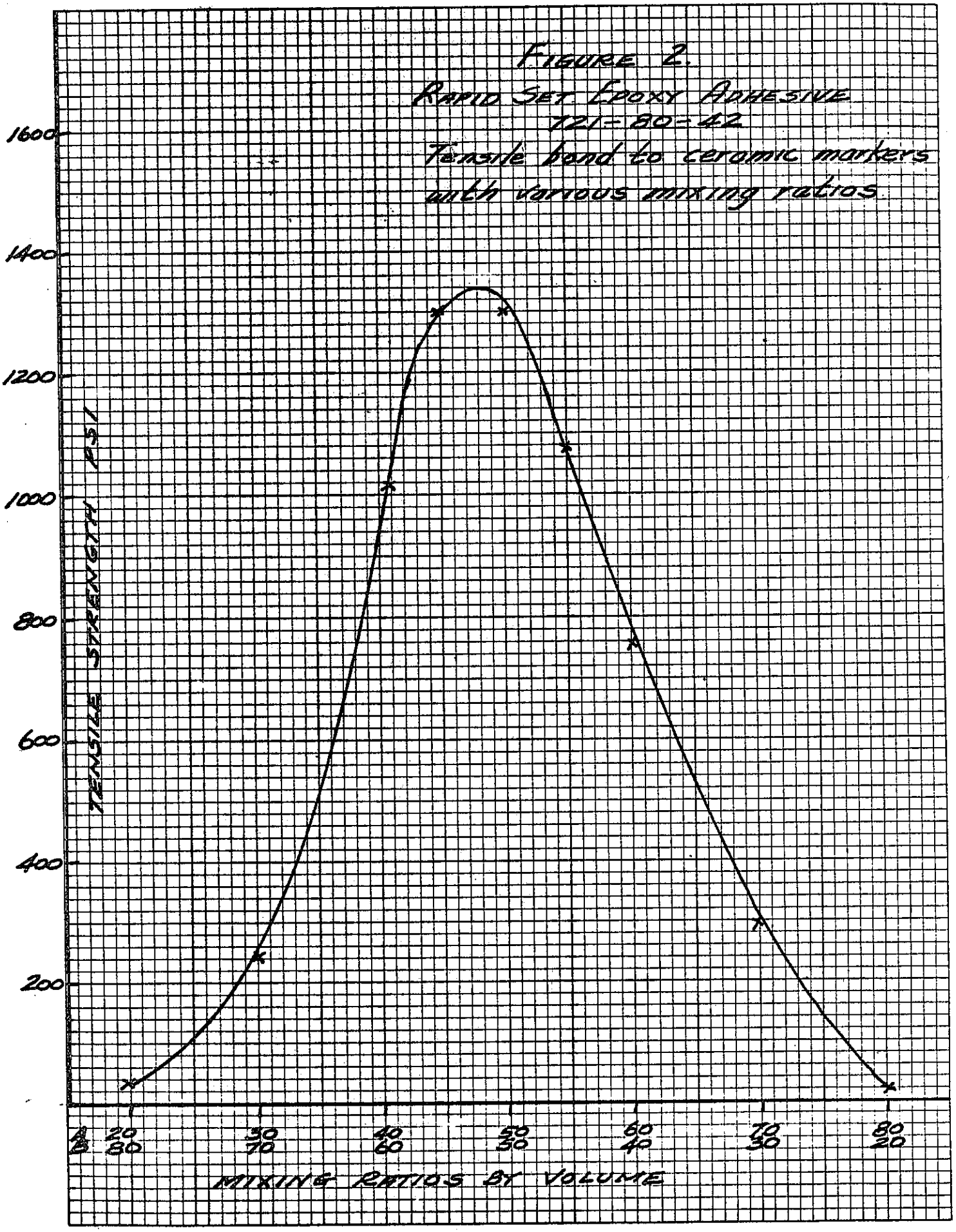

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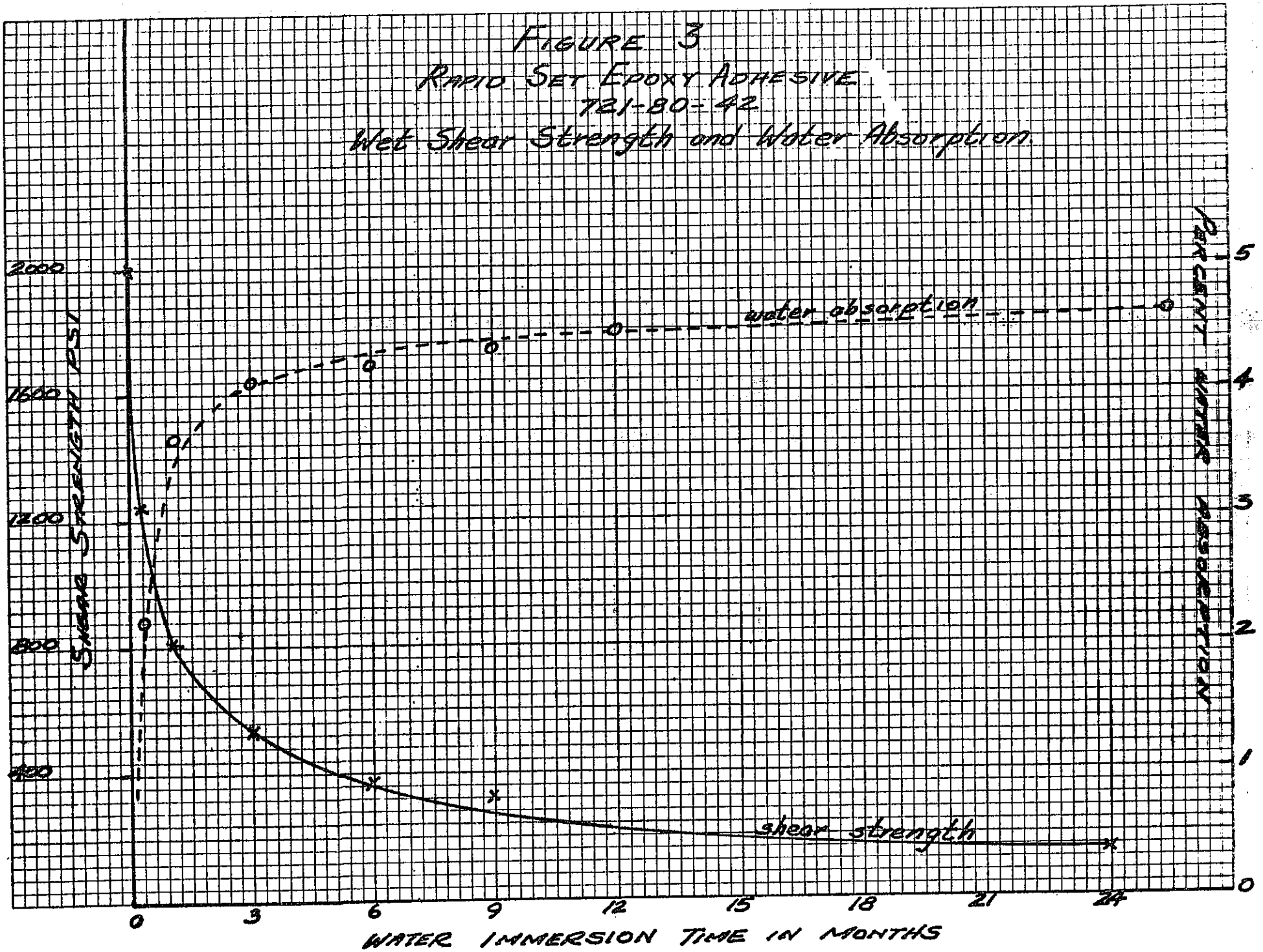

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APPENDIX

Effective Shrinkage

Suitable forms shall be made on a substantial thickness (1/8-inch or greater) of polyethylene sheet and by using polyethylene strips 1/8-inch thick, to provide a space, the inner dimensions of which are 3-1/2" by 9-1/2" by 1/8" deep.

The epoxy components are conditioned to 77°F and a 350-gram batch hand mixed for 5 minutes using care to avoid inclusion of air. The mixed components are slowly poured into the forms (a 350-gram batch makes two test panels) using a slight excess.

Immediately, a 4" by 10" panel of single strength glass, meeting Federal Specification DD-G-451a, Type II, Clear Sheet Glass, which has been thoroughly cleaned with a detergent and water, water flushed, and then carefully wiped two times with a clean cloth moistened with methyl ethyl ketone, is slowly lowered into place to avoid air bubbles at the epoxy-glass interface.

The glass is then weighted, and the epoxy allowed to cure 24 hours at room temperature (77°±5°F) before removal from the form. The result is a laminate of the glass panel and an 1/8-inch film of epoxy, the dimensions of the epoxy being 1/4-inch less on all sides than the dimensions of the glass panel.

The laminate is then aged six days under conditions of ASTM E-41, E-41-57T2(b) which is 77°F and 40 percent relative humidity. At the end of one week after casting, the panel is cycled 30 minutes to 120°F, followed by 30 minutes at the standard temperature in the ASTM method.

At the end of 10 uninterrupted cycles, the panel is brought to the standard room temperature (77°F). One end is then held firmly against a plane surface, and the divergence from plane at the unconfined end shall be measured to the nearest 0.001-inch. The effective percent shrinkage shall be computed as follows:

$$S = 100 - \left(\frac{\sqrt{(\ell)^2 - (a/2)^2}}{\ell} \right) \times 100$$

Where: S = Effective shrinkage, in percent

a = Measured divergence, in inches

ℓ = one-half the length of the glass plate, (5 inches when a 10-inch length is used for the test)

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METHOD FOR TESTING EPOXY RESIN ADHESIVES FOR PAVEMENT MARKERS

Scope

The procedures used for testing epoxy resin adhesives for pavement markers are described in this test method.

This test method is divided into the following parts:

I. Pot Life
II. Infrared Curve
III. Bond Strength Concrete
IV. Tensile Adhesion and Cohesion
V. Brookfield Viscosity
VI. Shear Ratio
VII. Weight in Pounds per Gallon
VIII. Percentage of Entrapped Air
IX. Slant Shear Strength

PART I. POT LIFE

A. Apparatus

1. Unwaxed paper cups, 8 oz., 2 inches ± ¼ inch diameter base (Dixie Cup No. 4338 or equivalent).
2. Unwaxed paper cups, 2 oz., 1¾ inches x 1½ inches at base (Dixie Cup No. 23 or equivalent).
3. Wooden tongue depressor with ends cut square (Puritan No. 705 or equivalent).
4. Stainless steel spatula with blade 6 inches x 1 inch, and with the end cut square.
5. Stop watch, 1 second or smaller divisions.

B. Test Procedure

1. Condition both A and B components to 77 ± 2°F.
2. Stir the separate components vigorously with the spatula to redisperse any settled material.
3. Measure components in a ratio of one 2 ounce paper cup of component A to one 2 ounce paper cup of component B into an 8 ounce paper cup.
4. Start stopwatch immediately and mix the components for 60 seconds using a wooden tongue depressor and taking care to scrape the sides and bottom of cup periodically.
5. Place the sample at 77 ± 2°F on a wooden bench top which is free of excessive drafts.
6. Probe the mixture once with the tongue depressor every 30 seconds, starting two minutes from the time of mixing.
7. The time at which a soft stringy mass forms in the center of the cup is the pot life.

PART II. INFRARED CURVE

A. Apparatus

1. Perkin-Elmer Model 137-B Infracord Spectrophotometer, automatic recording system from 2.5 microns to 15 microns with a two speed recorder. Comparable results can be obtained by other double-beam recording spectrophotometers with similar resolution.
2. Disk holder for a one inch diameter disk.
3. Two sodium chloride crystal disks one inch in diameter.
4. Sorvall SS-3 Automatic Superspeed Centrifuge, or a comparable centrifuge which is able to separate the liquid and solid phases of the epoxy components without previous dilution with solvents.

B. Procedure

1. Place about 15 grams of component A into a stainless steel centrifuge tube.
2. Counterbalance with component B in a second centrifuge tube.
3. Centrifuge the two components at 17,000 rpm until there is a supernatant liquid layer present in each tube. This takes 20 to 30 minutes.
4. Place a drop of component A liquid layer on a sodium chloride disk.
5. Place another sodium chloride disk over the drop, rotate, and press down until the liquid has flowed into a uniform layer of proper thickness between the two sodium chloride disks.
6. Place the disks in the holder and run an absorption curve with the infrared spectrophotometer.
7. More or less liquid may be used between the disks so as to produce a maximum absorption of 0.7 to 1.0 for the strongest absorption point on the curve.
8. Clean the disks with toluene and dry.
9. Repeat steps 4 through 8 with the liquid layer from component B.
10. Compare each curve with the attached absorption curves for standard materials. Two materials are considered to be identical if all of the absorption points agree as to wave length and relative magnitude of the peaks in comparison with the other points of absorption.

PART III. BOND STRENGTH CONCRETE

A. Materials

1. A sandblasted concrete block 12 inches by 12 inches by 3 inches prepared with 7 sack concrete and having a tensile strength in excess of 250 psi.

B. Apparatus

1. Use the same apparatus as specified in Test Method No. Calif. 420 A. Apparatus 1, 2, 3 and 7; or a suitable testing press at a load rate of 5000 lbs./minute.
2. A sandblasted 2" diameter steel rod may be used in place of the pipe cap.
3. Stop watch, 1 second or smaller divisions.

C. Procedure

1. Condition the test equipment, materials and epoxy components for 24 hours at the testing temperature specified.
2. Stir the separate components vigorously for 30 seconds.

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3. Place equal volumes of each component on a tin plate and mix with a plaster trowel or spatula for 60 ± 5 seconds.
4. Immediately start timing.
5. Place the adhesive on the pipe cap, or rod, and the concrete surface.
6. Press pipe cap, or rod, firmly in place and remove the excess adhesive.
7. Just before the required test time insert the hook into the cap (or rod).
8. Proceed according to Part I, Section A-6 of Test Method No. Calif. 420, or use a suitable testing press to determine the bond strength.

PART IV. TENSILE ADHESION AND COHESION

A. Materials

1. Class II polyester marker of current State specification, one per test.
2. Classes III and IV ceramic markers of current State specification, two each per test.
3. Reflective pavement marker of current State specification, three per test.

B. Apparatus

1. Use testing apparatus described in Part III-B above.
2. Cold box capable of maintaining 15 ± 2°F.
3. Oven capable of maintaining 140° ± 2°F.

C. Procedure

1. Stir the separate components vigorously for 30 seconds.
2. Place equal volumes of each component on a tin plate and mix with a trowel or spatula for 60 ± 5 seconds.
3. Place the adhesive on the pipe cap or rod and the surface to be tested.
4. Press the pipe cap or rod firmly in place and remove the excessive adhesive.
5. Cure all specimens for 24 hours at 77 ± 2°F.
6. Proceed according to Part I, Section A-6 of Test Method No. Calif. 420, or use a suitable testing press for the following:
   a. Class II polyester marker, test one only.
   b. Class III ceramic marker, test one only.
   c. Reflective pavement marker, test in triplicate.
7. Post cure one Class III ceramic marker further as follows:
   a. 48 hours at 140°F.
   b. Return to 77 ± 2°F and then place in cold box for 24 hours at 15 ± 2°F.
   c. Return to 77 ± 2°F and test as in 6 above.

PART V. BROOKFIELD VISCOSITY

A. Apparatus and Supplies

1. Model RVT Brookfield Syncro-Electric Viscometer, Brookfield Engineering Laboratories, Stoughton, Massachusetts.
2. Brookfield Helipath Stand Model C with spindles TD, TE and spindle weight.
3. Round, one pint paint cans.
4. Stainless steel spatula having a blade 6 inches by 11 inches with the end cut square.

B. Test Procedure

1. Fill a one pint paint can within one inch of the top with well mixed component A.
2. Condition the material to temperatures required for the test.
3. Stir the material vigorously for 30 seconds with a spatula.
4. Remove entrained air bubbles by vigorous tamping.
5. Insert proper spindle according to specifications.
6. Make viscosity reading at 5 rpm within ten minutes of stirring.
7. Make additional readings at 0.5 and 2.5 rpm for use in Part VI—Shear Ratio.

C. Calculations for 5 rpm readings

1. Reading on 0–100 scale × 4000 = viscosity in centipoise, TD spindle.
2. Reading on 0–100 scale × 10,000 = viscosity in centipoise, TE spindle.

D. Calculations for 0.5 rpm readings

1. Reading on 0–100 scale × 40,000 = viscosity in centipoise, TD spindle.
2. Reading on 0–100 scale × 100,000 = viscosity in centipoise, TE spindle.

E. Calculations for 2.5 rpm readings

1. Reading on 0–100 scale × 8000 = viscosity in centipoise, TD spindle.
2. Reading on 0–100 scale × 20,000 = viscosity in centipoise, TE spindle.

PART VI. SHEAR RATIO

A. Apparatus and Supplies

1. Same as Part V.

B. Test Procedure

1. Same as Part V.

C. Calculations

1. $\text{Shear Ratio} = \dfrac{\text{Viscosity cp at 0.5 rpm}}{\text{Viscosity cp at 2.5 rpm}}$

PART VII. WEIGHT IN POUNDS PER GALLON

1. Follow Federal Test Method Standard No. 141, Method 4184.

PART VIII. PERCENTAGE OF ENTRAPPED AIR

A. Apparatus

1. Round, one quart paint cans.
2. Electric hot plate.
3. Vacuum chamber capable of 30″ mercury which is equipped with valves enabling manual control of

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application and release of vacuum and viewing ports or other means of observing controls under vacuum.

B. Test Procedure

1. Weight in pounds per gallon, Part VII, must be done before proceeding with this test. (W_1)
2. Fill separate round, one quart paint cans half full of each component.
3. Heat cans and contents to 175-180°F.
4. Place into vacuum oven and evacuate until the samples show signs of frothing over.
5. Vibrate or tamp samples to facilitate removal of entrapped air.
6. As frothing subsides, raise vacuum until 30″ is reached.
7. Cool samples to 77 ± 2°F after removing from vacuum oven.
8. Measure the weight in pounds per gallon as done in Part VII. (W_2)

C. Calculations

$$\% \text{ Air} = \frac{W_2 - W_1}{W_1} \times 100$$

See items 8 and 1 above for W_2 and W_1, respectively.

PART IX. SLANT SHEAR STRENGTH

A. Materials

1. Ottawa sand, ASTM C109
2. Portland cement Type II
3. Water

B. Apparatus

1. Suitable mold to make diagonal concrete mortar blocks with a square base with 2 inch sides and having one diagonal face 2 inches x 4 inches starting about ¾ inch above the base. The diagonal faces of two such blocks are bonded together, producing a block of dimensions 2 inches by 2 inches x 5 inches.
2. Blocks made from the following composition:

Ottawa sand, ASTM C109	30.1 lbs.
Portland cement Type II	12.1 lbs.
Water	4.8 lbs.

Cure blocks 28 days in a fog room. Dry and lightly sandblast diagonal faces.

3. Suitable test press.

C. Test Procedure

1. Mix epoxy as described in Part III-C and apply a coat to each diagonal surface. Press diagonal surfaces of each block together by hand and remove excess epoxy adhesive.
2. Align the blocks so that the ends and sides are square and form a block 2 inches x 2 inches x 5 inches. Use blocks of wood or metal against each 2″ x 2″ end to keep diagonal faces from slipping until epoxy hardens.
3. After the required cure time as called for in specification, apply a suitable capping compound to each of the 2 inch x 2 inch bases and test by applying a compression load with a Universal Test Machine or other suitable testing apparatus at the rate of 5000 lbs./min. until failure.
4. Report results in pounds per square inch

$$= \frac{\text{Load in Pounds}}{4}$$

5. For wet shear strength, bond another set of blocks together as described above. Cure 24 hours at 77 ± 2°F, then soak in water for seven days at 77 ± 2°F, and immediately test as described above in C-3.

Recording Test Results

Record all test results on Form HMR T-581

REFERENCES

Test Method No. Calif. 420
Federal Test Method Standard No. 141, 4184

End of Text on Calif. 425-C

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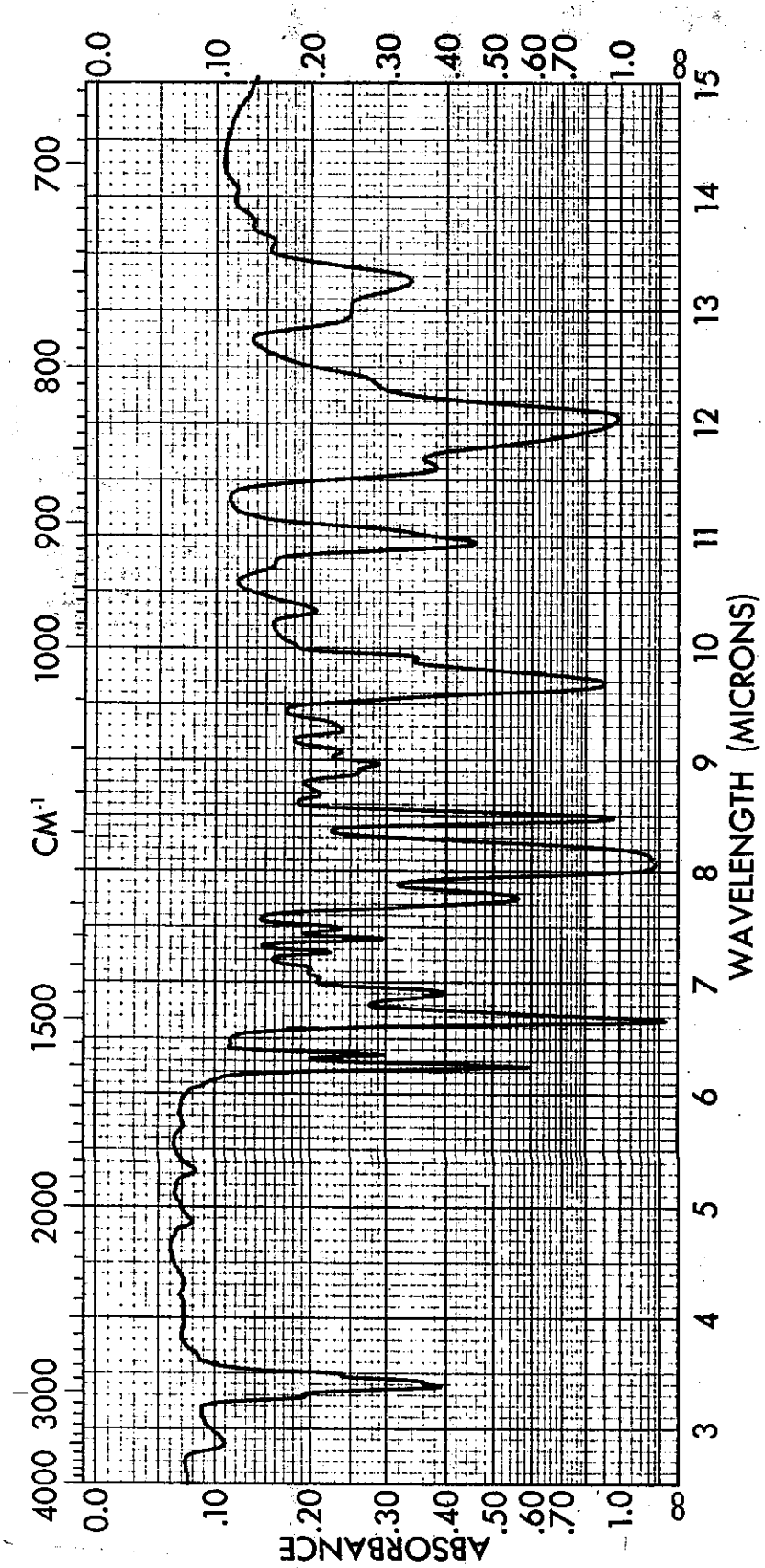

TEST METHOD 425

STANDARD INFRA RED ABSORPTION CURVE

COMPONENT A

RAPID SET ADHESIVE

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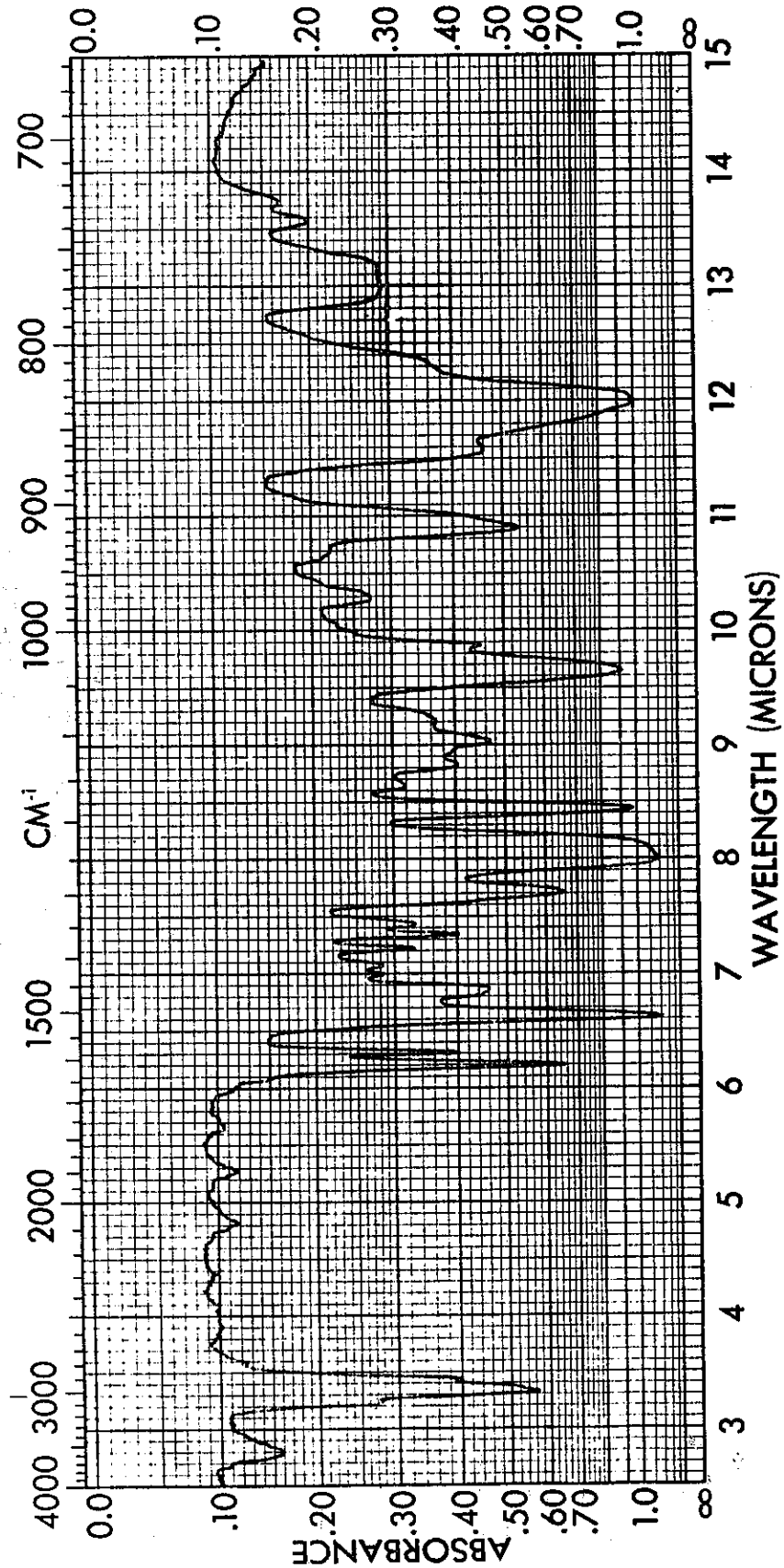

TEST METHOD 425
STANDARD INFRARED ABSORPTION CURVE
COMPONENT A
STANDARD SET ADHESIVE

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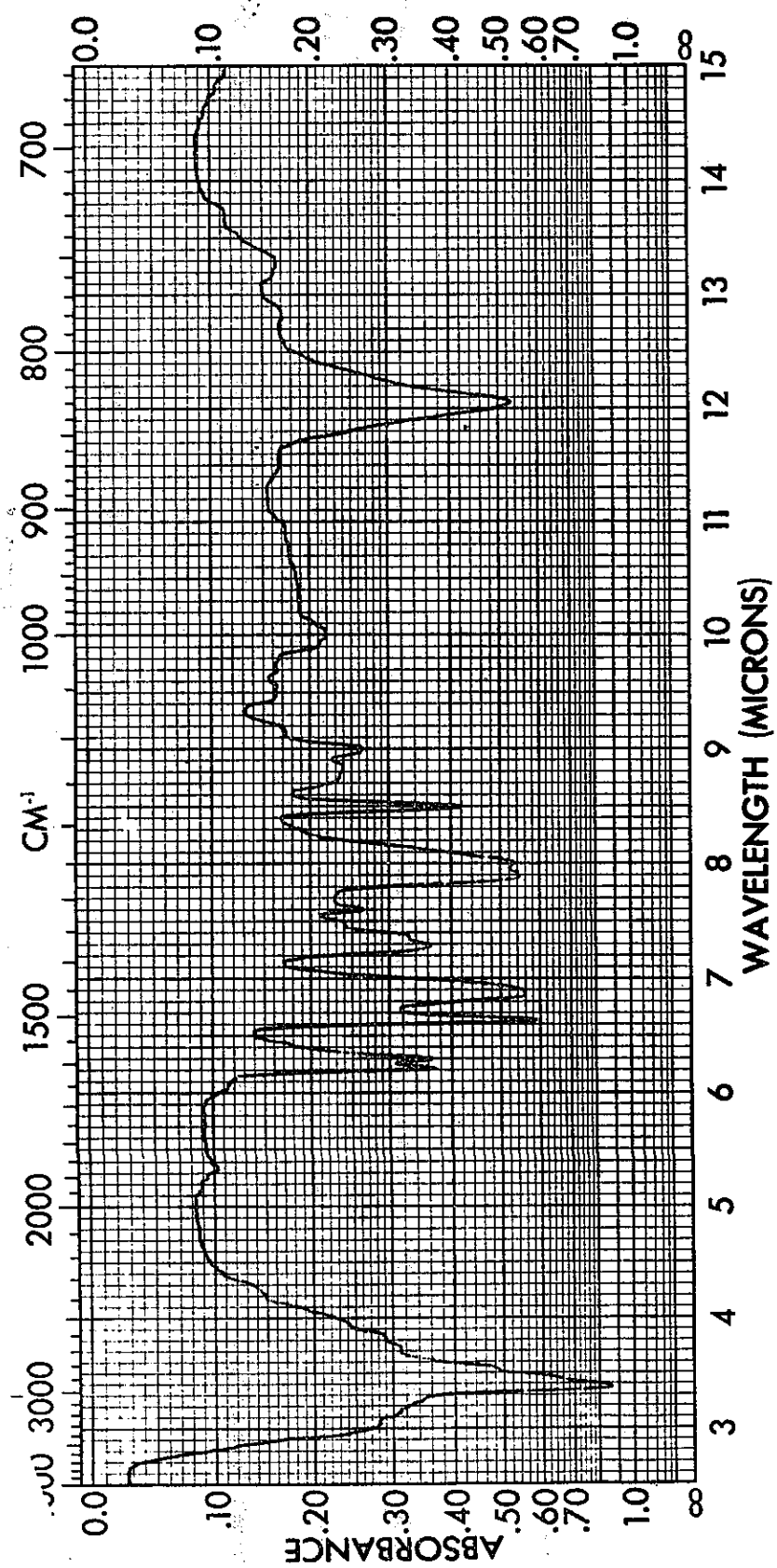

TEST METHOD 425
STANDARD INFRARED ABSORPTION CURVE
COMPONENT B
STANDARD SET ADHESIVE

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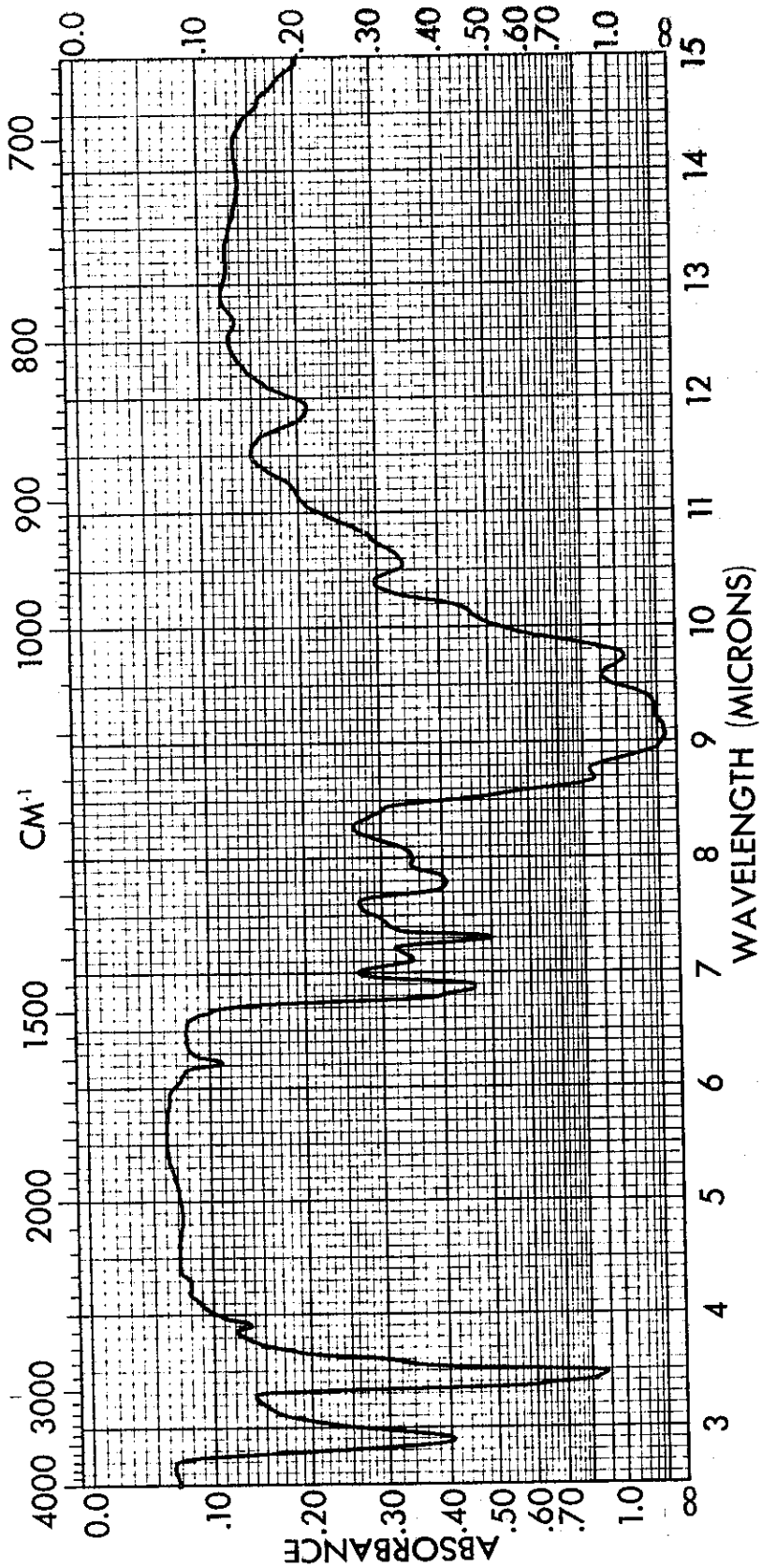

TEST METHOD 425
STANDARD INFRA RED ABSORPTION CURVE
COMPONENT B
RAPID SET ADHESIVE

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