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16. ABSTRACT

The residual elastic strain in an oriented quartzite sample taken from outcrop is measured by means of x-ray diffraction. Two disks cored at right angles to each other are cut parallel to the XY plane and the Z axis respectively of a rectangular coordinate system related to the field orientation. Six solutions for the principal strain axes are calculated by computer analysis. The technique permits a general solution of the three-dimensional state of residual elastic strain in naturally deformed rocks.

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STATE OF CALIFORNIA
DEPARTMENT OF TRANSPORTATION
DIVISION OF CONSTRUCTION AND RESEARCH
TRANSPORTATION LABORATORY

April 1975

FHWA No. D-5-44
TL No. 632134

Mr. R. J. Datel
Chief Engineer

Dear Sir:

I have approved and now submit for your information this final research project report titled:

ANALYSIS OF THE STATE OF RESIDUAL ELASTIC
STRAIN IN QUARTZOSE ROCKS BY X-RAY DIFFRACTION

Study made by . . . . . Geotechnical Branch

Under the Supervision of . . . . . R. A. Forsyth

Principal Investigator . . . . . M. L. McCauley

Co-Principal Investigator . . . . . James Gamble

Report Prepared by . . . . . James Gamble

Very truly yours,

George A. Hill

GEORGE A. HILL
Chief, Office of Transportation Laboratory

Attachment

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ACKNOWLEDGEMENTS

This research project could not have been completed without several personal communications with Dr. Friedman, Texas A & M University. He not only answered the specific questions I asked, but offered suggestions and gave much additional material including the complete annotated computer program which he anticipated would be needed.

At the Transportation Laboratory Messrs. Chas. Frazier, Bill Chapman, Tom Hoover, and Byron Works each contributed his time and special skills to different phases of the project.

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

The purpose of the research project described in this report was to determine if a new X-ray diffraction technique of measuring the state of three dimensional residual elastic strain in quartzose rocks as described by M. Friedman [1,2] could be duplicated at the Transportation Laboratory. Briefly, the X-ray measurements are obtained from two polished quartzite samples (disks) cored at right angles to each other from an oriented hand specimen taken from outcrop. One sample is parallel to the XY plane of a rectangular coordinate system related to the field orientation, and the second sample is oriented parallel to the Z axis. The orientation and magnitude of the principal strain axes are calculated by computer from the seven strain components obtained from X-ray measurements of the two disks.

Dr. Friedman's published articles describing the procedures and conditions for making the measurements were carefully studied and the technique followed as closely as differences in equipment, and the details given in the published material, permitted. The two papers by Dr. Friedman [1,2] describing the techniques are readily available. Therefore a complete description of the new method is not repeated herein. However, to aid in understanding the purpose and need for the work that was done on the research project, the abstract from Dr. Friedman's paper (1967) is presented by the following:

"A new X-ray diffraction technique is described to measure the three-dimensional state of residual elastic strain in quartzose sandstones. The sample consists of any two, polished, 2-inch diameter, sandstone disks cored at right angles to each other from an oriented hand specimen taken from outcrop. One of the disks is oriented parallel to the XY plane of a rectangular coordinate system related to the field orientation, and the second disk is oriented parallel to the Z axis. The d-spacing (corrected for θ-dependent factors) for

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[32$\bar{5}$4] in quartz is determined by computer analysis of fixed-count data obtained by step-scanning the diffraction profile. The strain component parallel to a given direction in space is obtained by comparing the measured d-spacing along that direction with that of strain-free material, i.e., $(d_{obs} - d_u)/d_u = \varepsilon$. From study of the XY disk strain components are determined parallel to the Z axis and along a number of directions inclined at 45° to the XY plane. These are corrected for strain relaxation perpendicular to the XY plane (caused by preparation of the disks) by the component of strain parallel to the Z axis as determined from the second disk. Orientations and magnitudes of the three principal strain axes are calculated via computer from any five of the corrected strain components inclined at 45° to the XY plane and the undistorted value of strain along the Z direction.

"Results to date in study of sandstones deformed during Late Cretaceous indicate that the residual strains were locked in these rocks at the time of deformation. Strain magnitudes in excess of $400 \times 10^{-6} \pm 20 \times 10^{-6}$ have been recorded."

The existence of residual elastic strains (stresses) in some rock masses has been conclusively demonstrated and the magnitude of these stresses, as measured in quartzites, granites, and sandstones, can be large [2]. A special bias of the X-ray technique is that the residual strains stored in the grains (quartz) are measured preferentially to those in the cement. The special advantage of the new technique is that a general solution of the three dimensional state of residual strain is permitted when the principal planes are unknown, as in naturally deformed rocks, and the measurements can be obtained from small oriented samples taken from outcrop. Although the method does not seem to have practical value for highway construction at the present time, continued research may discover new applications for this technique.

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The measurement of residual elastic strain at the Transportation Laboratory was envisaged as the first phase of a two-part project, that if successful, might make it possible to predict the orientation of joint planes in some rock masses prior to excavation. This information or the orientation of joint planes would have been incorporated into the slope design. The proposed second phase, the determination of the relationship between the direction and magnitude of the principal strain axes and the orientation of the joint planes, will not be attempted because computer analysis of the strain measurements did not uniquely define the directions of the greatest and intermediate axes. Another possible application for the information on residual strain in a rock mass would have been to identify seismic refraction velocities that were anomalously high. This knowledge would be beneficial for estimating the excavation characteristics of the rock mass.

Numerous delays encountered in pursuing the project were caused by routine work requirements, shared use of the diffractometer, and difficulty with the computer program.

The cost of determining the magnitude of the residual elastic strain and the directions of the three principal planes by X-ray diffractometry is considerable because of the time required. This can be reduced however, by automatic printout of data, more experience with the computer program and data interpretation, and procedural standardization. The reliability of these data obtained (and the derived solution) is very dependent on properly aligned equipment, sample temperature control, and awareness of the personnel making the study of the need for close attention to procedural details. The possibility of numerical errors being made during the recording, correcting, and calculating of these data is ever present. If undetected, such errors would yield erronous and possibly misleading solutions.

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CONCLUSIONS

Although the findings from this study have no known immediate practical application to the design and construction of highways, on-going and future research efforts may find useful correlations between the residual elastic strain and other measurable engineering properties of rock masses [4].

The residual elastic strain in quartzose rocks can be measured at the Transportation Laboratory by the X-ray diffraction technique. However, some modifications to the existing equipment, and some additional equipment, would be required to efficiently obtain consistantly accurate 2θ measurements.

Temperature control of the sample, a better method of positioning the sample in the X-ray beam, and automatic data printout, are the minimum additional equipment requirements for accurate and efficient data acquisition.

A thorough understanding of the computer program is essential. To understand the computer program, the programmer must understand the basic assumptions of the technique, and how the data were obtained. The relative merits of the "mid-point" method (p.12) versus the nth order polynomial computer analysis (used by Dr. Friedman) to find the "peak" 2θ value is difficult to evaluate; the "mid-point" method is much less complex to program, and this method was used.

The sample requirements are restrictive, e.g., the residual elastic strain can only be measured in rocks with a high content of granular quartz. Further, the residual strain distribution within the aggregate (quartz grains) must be at least statistically homogeneous, so that the X-ray measurements from different locations within the two surfaces can be meaningfully combined [1].

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RECOMMENDATIONS

A recommendation for implementation, or for continued investigative efforts is not warranted at the present time. Recent studies by Friedman [4] in cooperation with the U.S. Bureau of Mines found correlations between the condition of residual elastic strain and the engineering properties of some massive rocks. Thus, on-going and future studies may find a correlation, or direct engineering application of value to highway construction.

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IMPLEMENTATION

Although the X-ray diffraction technique of residual elastic strain measurement in rocks does not appear applicable at present to highway construction, knowledge of residual strain values does appear to have practical application, though indirectly, for underground excavations in granitic rocks. A recent study by Friedman and Bur [4] found that in granitic rocks a good correlation exists between ultrasonic attenuation and velocity fields, and the residual strains measured by X-ray diffraction. Therefore, the possibility exists that sonic probing alone may yield much of the information required for rapid underground excavation. Friedman and Logan [5] had previously noted that for certain loading conditions the state of residual elastic strain in quartzose sandstones controls the orientations of induced tensile and shear fractures.

A considerable body of knowledge now exists concerning the nature of residual elastic strain in rocks, and a precise method of measurement has been designed. It seems assured that continued efforts by the investigators working in this field will disclose new areas of practical application. The method is limited for general application for at least the following three reasons: (1) the strain can be measured only on granitic rocks, quartzites, and quartzose sandstones; (2) few problems will be encountered in highway construction that will justify the considerable expense of obtaining the orientation and magnitude of the principal strain axes by X-ray diffractometry; (3) it will be difficult to retain the expertise that is needed to obtain accurate data and to derive a reliable solution; and (4) there is no assurance that the sample selected for the strain measurements will yield an unequivocal solution, i.e., the magnitude and direction of the three principal strain axes.

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TESTING PROCEDURE

Equipment

The basic equipment used was a General Electric XRD-5 diffractometer in conjunction with Ortec detector control components (pulse height analyzer, ratemeter, dual counter/timer, high voltage power supply). Copper radiation with line focus was collimated by a 1° beam slit, a medium resolution Soller divergence slit, and an 0.2° receiver slit. A filter was not used. A time constant of 3, 35 KVP, and ∿ 35 MA were used.

A General Electric residual stress device was used for translating the counter tube onto the new focusing circle that is generated when the sample is rotated to the 45° positions [1]. A rotating sample holder and stage was included as part of the G.E. residual stress measurement package. The installation, operation, and maintenance of the residual stress device is fully described in General Electric Direction 12834C, Code 3-2166, a G.E. X-ray Department equipment manual. This equipment is shown installed on the spectrogoniometer in Figure one. It is absolutely necessary to precisely align the spectrogoniometer first, as indicated in the instructions, before aligning the translating device. After properly installed, the device remained in alignment and performed very satisfactorily.

The equation for determining the correct positions of the detector for various rotation angles (Ψ) of the sample is given by Norton [10]. Special note should be taken that for a 45° clockwise rotation of the sample, $\Psi = 315°$. This places the beam divergent point (R) at 9.27 inches, a distance much greater than the maximum 5.98 inch range of the translating device. For this study the distance of R (3.54 inches) for the 45° counterclockwise sample rotation was

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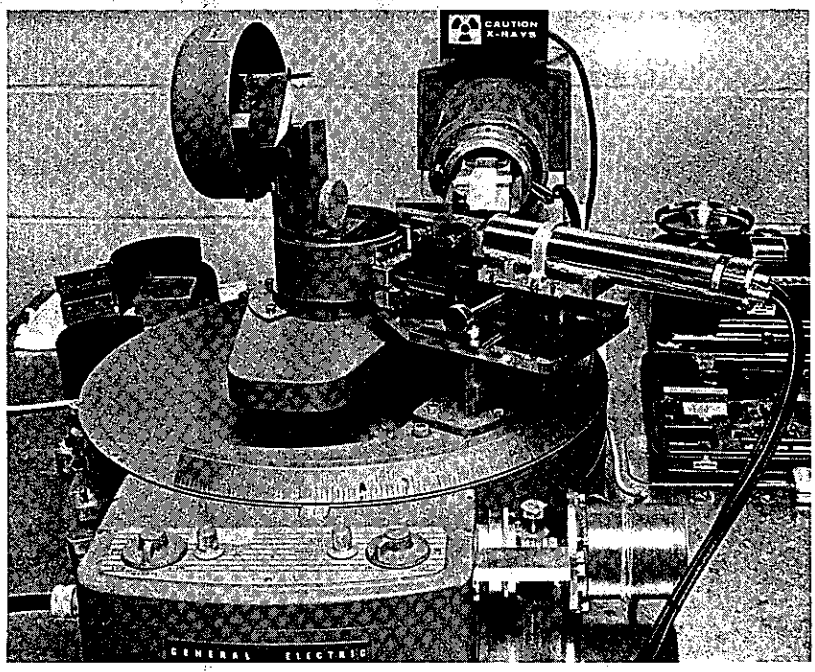

FIGURE 1

General Electric XRD-5 goniometer with sample holder and residual stress device attached.

maintained for both 45° positions. Satisfactory peak intensities were obtained with the sample in this position even though the detector was not on the focusing circle. The source to specimen distance (R_o) for the G.E. XRD-5 diffractometer is 5.73 inches.

No problems were experienced with the sample rotating device except for the very tight fit into the base. This was a problem only because frequent removal of the sample holder was necessary to analyze routine samples. To maintain maximum precision the sample holder should not be removed until all the count data are recorded.

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The scale on the body of the sample holder must be scribed at 45° on the right side of zero. This must be done with precision to ensure that the d-spacing measurements are made between the lattice planes that are inclined 45° to the sample surface.

Modeling clay was used to position the sample on the sample holder stage. Modeling clay has the advantage of cleanness, and can be reused almost indefinitely. Also, the sample can be repositioned with a minimum of difficulty. However, the accuracy with which the sample can be correctly placed in the X-ray beam varied as much as 0.07° 2θ as measured by the position of the 26.64° 2θ quartz diffraction peak on the chart recorder. The accuracy desired was ± 0.01°. Such small differences between successive sample positions are difficult, if not impossible, to detect by visual inspection of the contact between the sample and the post. A visual inspection will, however, eliminate gross misalignment of the sample. Another recourse for diminishing the observed differences in 2θ between successive sample positions is trial-and-error repositioning, but this is a very time-consuming procedure.

The lack of a shutter device for the tube port on the G.E. XRD-5 diffractometer necessitates a tube warm-up period of approximately one-half hour each time the sample is repositioned. This makes even longer the lengthy process of count data acquisition. A new shutter device manufactured by the Diano Corporation for use on the G.E. XRD-5 does not permit continuous tube operation without by-passing the "fail safe" cover shield, a practice that could expose a careless operator to hazardous radiation.

Automatic printout for recording the fixed count data would eliminate the time required to record the counts manually. Lacking this capability the stepping motor purchased for the research project was not used.

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The Ortec detector electronics, the Bicron detector, and the General Electric radial support assembly that were installed on the diffractometer to facilitate the research project, all performed very satisfactorily.

Temperature control of the sample is essential for residual elastic strain measurements in quartz. To obtain accurate and consistent d-spacing measurements the sample temperature should not vary more than 1°C. The thermal expansion of quartz is such that a 1°C change in the temperature will cause a 0.1° change in the 2θ angle [13]. The room at the laboratory in which the diffractometer is located is not temperature controlled and temperature variations of 7°F may occur from day to day, or even from morning to afternoon. This difficulty could not be corrected and it must be assumed that the fixed count data reflect some degree of inconsistency caused by variations in temperature.

Sample

The oriented hand specimen was obtained from the Lakeview quartzite quarry (now Premier Enterprises) located about five miles east of Lone Pine, California. The steeply dipping beds of almost pure quartzite have been exposed along the strike by the quarrying operation (see Figure 2). Four oriented specimens large enough for coring were pried from the free face formed by a smooth bedding plane. Closely spaced joint planes at nearly right angles to the plane of the bedding made the task of obtaining the specimens easier than was anticipated.

At the laboratory the specimen selected for coring was carefully leveled, oriented, and encased in a 18" x 18" x 18" block of concrete. Two cores two inches in diameter were cut from the specimen

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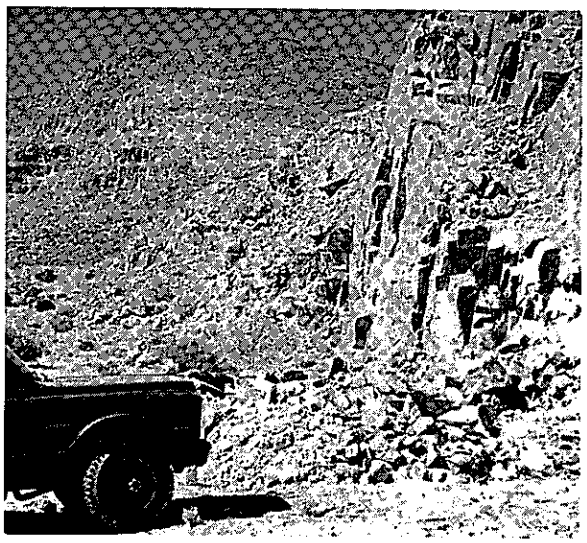

FIGURE 2
Steeply dipping quartzite beds at the
Lakeview Quarry

(see Figure 3) one perpendicular to the XY plane, and one perpendicular to the Z axis of a rectangular coordinate system that relates the specimen to the field orientation. A 2-inch diameter disk was cut from each core, one (1st disk) parallel to the XY plane and the other (2nd disk) parallel to the Z axis of the coordinate system. The disks were carefully marked for orientation and the surface to be irradiated was ground to at least 0.1 inch away from the saw cut using 400 and 600 mesh abrasive. After polishing, the thickness was approximately 0.3 of an inch.

The d-spacing for strain free quartz (du) is determined from a powder sample prepared from the rock specimen [1]. The powder sample was prepared by mixing quartzite powder passing 200 mesh with catalyzed polyester resin and casting as a solid disk (see Figure 4). The quartz powder to resin ratio is not critical,

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FIGURE 3
Method of obtaining the cores from
which the disks were cut.

but should be estimated visually at about 90 to 10 by volume. The size of the disk should be approximately the same as those cut from the cores. The surface to be irradiated must be flat and may require some polishing.

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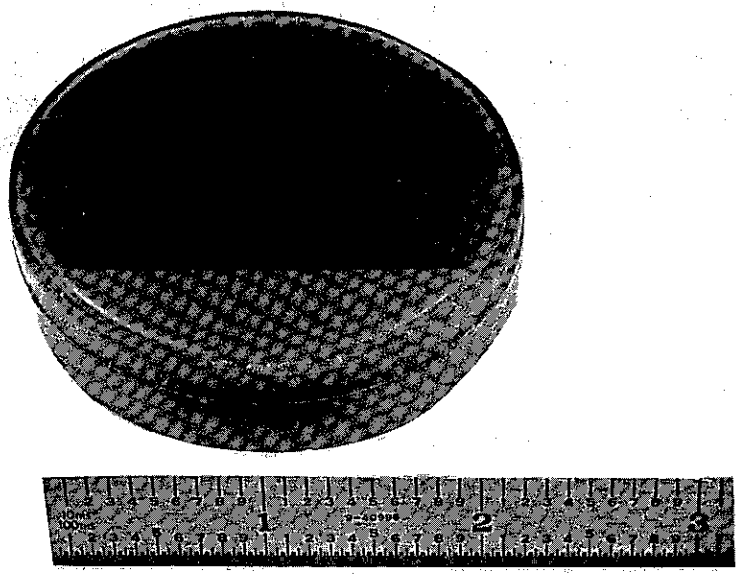

FIGURE 4
Disk prepared from
polyester resin and quartz powder

Data Acquisition

The fixed count data were obtained by step-scanning at 0.5° 2θ intervals across the diffraction peak and recording a 50 second count at each interval. For each direction of measurement the counts for three separate peaks are recorded and averaged. The average of the counts is used to find the "peak" 2θ value for each direction in which measurements were made. Automatic print-out capability was not available and the counts were recorded manually on prepared forms.

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The quartzite fabric was coarse-grained and a search to find an area of the surface with correctly oriented grains for the given direction of measurement was usually necessary. The peaks varied greatly in intensity between irradiated areas of the surface, and those of very low intensity were not used. The procedure followed was to locate a suitable peak visually on the strip chart recorder, then scan the back reflection region from about 151.50° to 154° and select a suitable place between 151.50° and 152.50° to make a background count (average of 3 counts). The search for an acceptable peak together with the attendant repositioning of the sample usually required more time than recording the counts.

The count data are corrected for lorentz, polarization, and absorption factors [14], and the "peak" 2θ values are determined by a mid-point method computer program. The d-spacing for each of the directions measured (both disks) are then found from any table of d-spacings for copper $K\alpha$ 1,2 radiation. The seven strain components E_a to E_f and E_z are calculated from the equation:

$$\frac{d_{obs} - d_u}{d_u} = E_i \, [1],$$

where (E_i) is the strain component along a given direction in the sample (1st disk) normal to the diffracting planes. The orientation of the strain components with respect to the coordinate axes and the disk surface is the same as shown in Figure 3, Friedman [1]. The adjustment for the relaxation of strain parallel to the Z axis, E_z' [1], that must be made to the calculated strains is derived from the equations of Murray and Stein [9]. In the computer program this adjustment is referred to as the gamma correction; its computed value is 10.1×10^{-6}. This numerical value of the adjustment obtained from the equations, is superposed on the unadjusted E_a to E_f and E_z values by the computer program.

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The publication by Murray and Stein is not in general circulation and copies of the required pages were obtained from Dr. Friedman; the University of Kansas Library also has a copy available for loan upon request from other libraries.

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PRINCIPAL STRAINS

The computer program uses the data shown in Table 1, and from this input data the computer derives six solutions for the principal strains, and plots the direction cosines of the principal strain axes. To obtain the orientation of the principal strain axes the direction cosines are converted to degrees and the X, Y, and Z coordinates for six solutions of E_1, E_2 and E_3 are plotted on an upper hemisphere projection of an equal area stereogram. A separate plot is made for each of the six solutions, and the points from each plot transferred to a composite lower hemisphere projection as shown in Figure 5; the plane of the figure is perpendicular to the bedding planes. The plots for the six solutions (annotated to clarify method of plotting the X, Y, Z coordinates) are shown in the Appendix.

Six solutions for the principal strains and the average strain values are given in Table 2. Tensile strains are positive, compressive strains are negative. The magnitude of the strains as compared to those found by Dr. Friedman are very large. The values are so large that one may question their accuracy. If they are inaccurate, the source of the problem is unknown. The fact that <u>all</u> of the values are large suggests to the author that they may be accurate. Such large values cannot be ascribed to the temperature problem inasmuch as a temperature change of 3.3°C (7°F; maximum observed) causes a plus or minus change of only .000045 Å between 153.50 - 153.60 degrees 2θ. This amount of d-spacing change is, however, sufficiently large to change the sign of the strain values for E_a and E_z, and may in part account for the variations in strain between E_a-E_f. The variation in the magnitudes of the respective principal axes for each of the six solutions suggests an inherent heterogeneity in strain magnitudes within the sample, or as stated by Dr. Friedman, "the basic assumption calling for no, or only uniform, partial relaxation within and between surfaces is not wholly valid".

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Significance of Data

The data shown in Figure 5 are significant for the following reasons: (1) The rather tight grouping of the least strain axes near the center of the diagram; (2) The greatest and intermediate strain axes, although widely scattered in direction, are very nearly in the same plane - a plane that is almost at right angles to the mean position of the plane containing the least strain axes. The reason for the scatter in direction of the greatest and intermediate strain axes of more than 180° is not understood. The grouping of the least strain axes about the Z-axis (strike of beds) of the stereogram suggests there is some relationship between the axes and the geological framework. However, a more precise statement concerning the relation of the principal strain axes to the geological framework cannot be made because of the wide directional scatter of the other two axes.

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2θ "Peak" Value (deg.)	d-spacing (Å)	Strain Value (Microinches/in.)	Plane of Measurement
		FIRST DISK	
153.514	.791289	$E_a = -4.17 \times 10^{-5}$	X + 45° Z
153.480	.791350	$E_b = 3.54 \times 10^{-5}$	X + 45° Z
153.383	.7915055	$E_c = 2.32 \times 10^{-4}$	YZ
153.437	.7914145	$E_d = 1.17 \times 10^{-4}$	YZ
153.486	.7913380	$E_e = 2.02 \times 10^{-5}$	XZ
153.399	.7914815	$E_f = 2.02 \times 10^{-4}$	XZ
153.510	.7912950	$E_z = -3.41 \times 10^{-5}$	Parallel to Z Axis
		SECOND DISK	
153.520	.791280	$E_t = 5.31 \times 10^{-5}$	XY
153.440	.791410	$E_w = 1.11 \times 10^{-4}$	XY
153.498	.791314	$E_v = 1.01 \times 10^{-5}$	Parallel to X Axis
		POWDER SAMPLE	
153.494	.791322 (d_u)	0.00	- - -

Table 1. "Peak" values, determined by the computer, and their corresponding d-spaces obtained from the corrected fixed-count data. The strain values were calculated from $\frac{d_{obs} - d_u}{d_u} = E_i$.

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Principal Strains		Direction Cosines		
Solution One				
E_1 =	1092.3	.85531	-.46987	-.21833
E_2 =	-17.6	.00154	.42371	-.90580
E_3 =	-46.0	.51811	.77440	.36313
Solution Two				
E_1 =	261.9	-.07301	-.93006	-.36007
E_2 =	104.9	-.43897	-.29422	.84896
E_3 =	-520.9	.89553	-.22004	.38679
Solution Three				
E_1 =	1087.9	.46173	-.86599	.19200
E_2 =	-3.2	.37404	-.00618	-.92739
E_3 =	-55.9	-.80430	-.50002	-.32106
Solution Four				
E_1 =	236.4	.95408	.12304	.27311
E_2 =	99.7	.20224	.40797	-.89031
E_3 =	-490.2	.22096	-.90467	-.36435
Solution Five				
E_1 =	783.3	.70042	-.71336	-.02302
E_2 =	71.0	.21917	-.18427	-.95813
E_3 =	416.9	.67925	-.67613	.28541
Solution Six				
E_1 =	497.9	-.66446	-.69446	-.27608
E_2 =	-3.2	.37289	.01205	-.92780
E_3 =	-57.4	.64764	-.71943	.25094

Average Principal Strains in Microinches Per Inch

E_i = 659.95 E_2 = 41.90 E_3 = -264.40

Table 2. Principal Strains and the direction cosines solved for six quadric surfaces. The average principal strains is also given.

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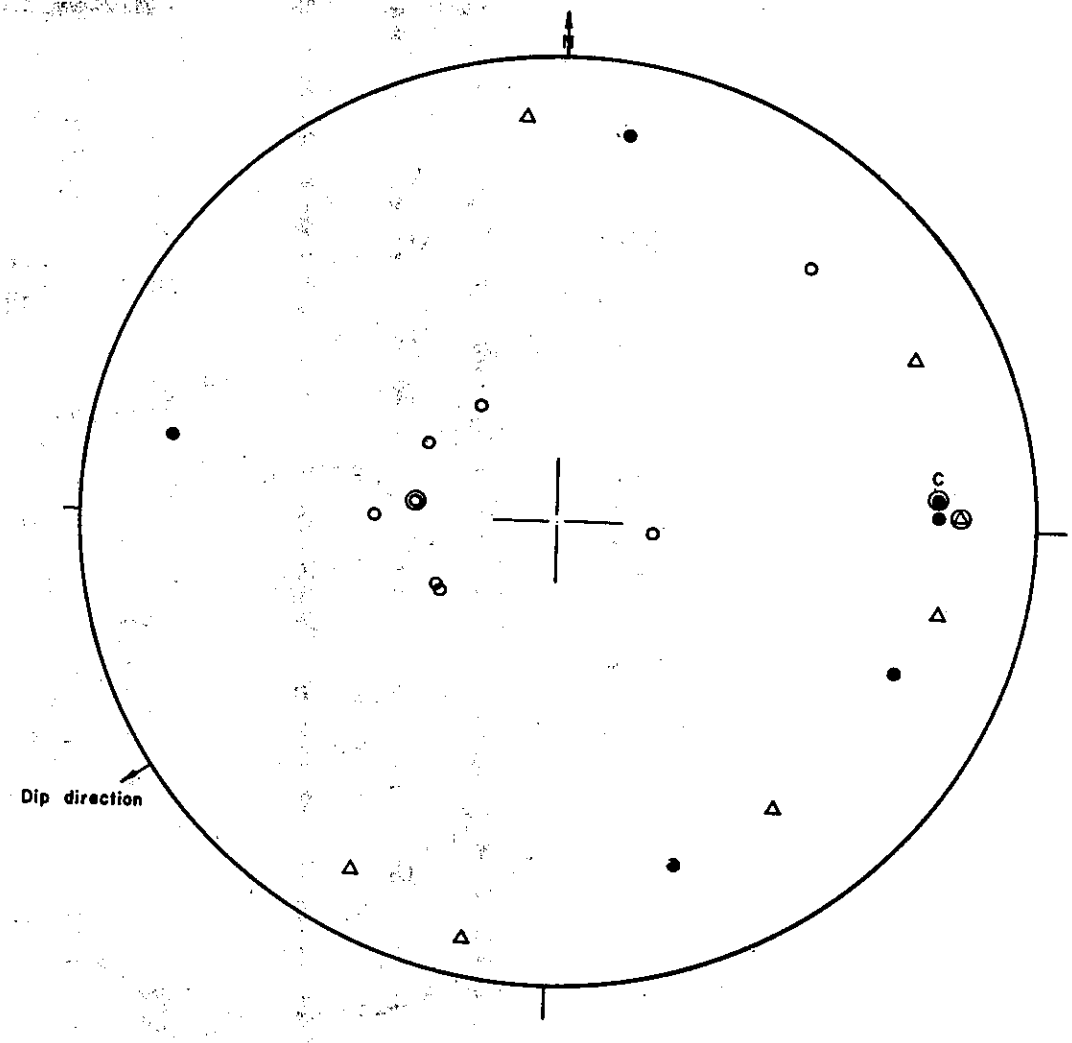

FIGURE 5

Sterogram showing orientations and average magnitudes of the greatest ( ), intermediate ( ), and least ( ) principal strain axis. The plane of the diagram is perpendicular to the bedding with direction of dip as shown. The data are plotted in equal area lower hemisphere projection. The mean position of each of the axis is indicated.

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APPENDIX

Plots for each of six solutions of the principal strain axes, annotated to show method of plotting. The points are plotted on the upper hemisphere of an equal area stereonet. Note that the composite stereogram, Figure 5, is changed to lower hemisphere.

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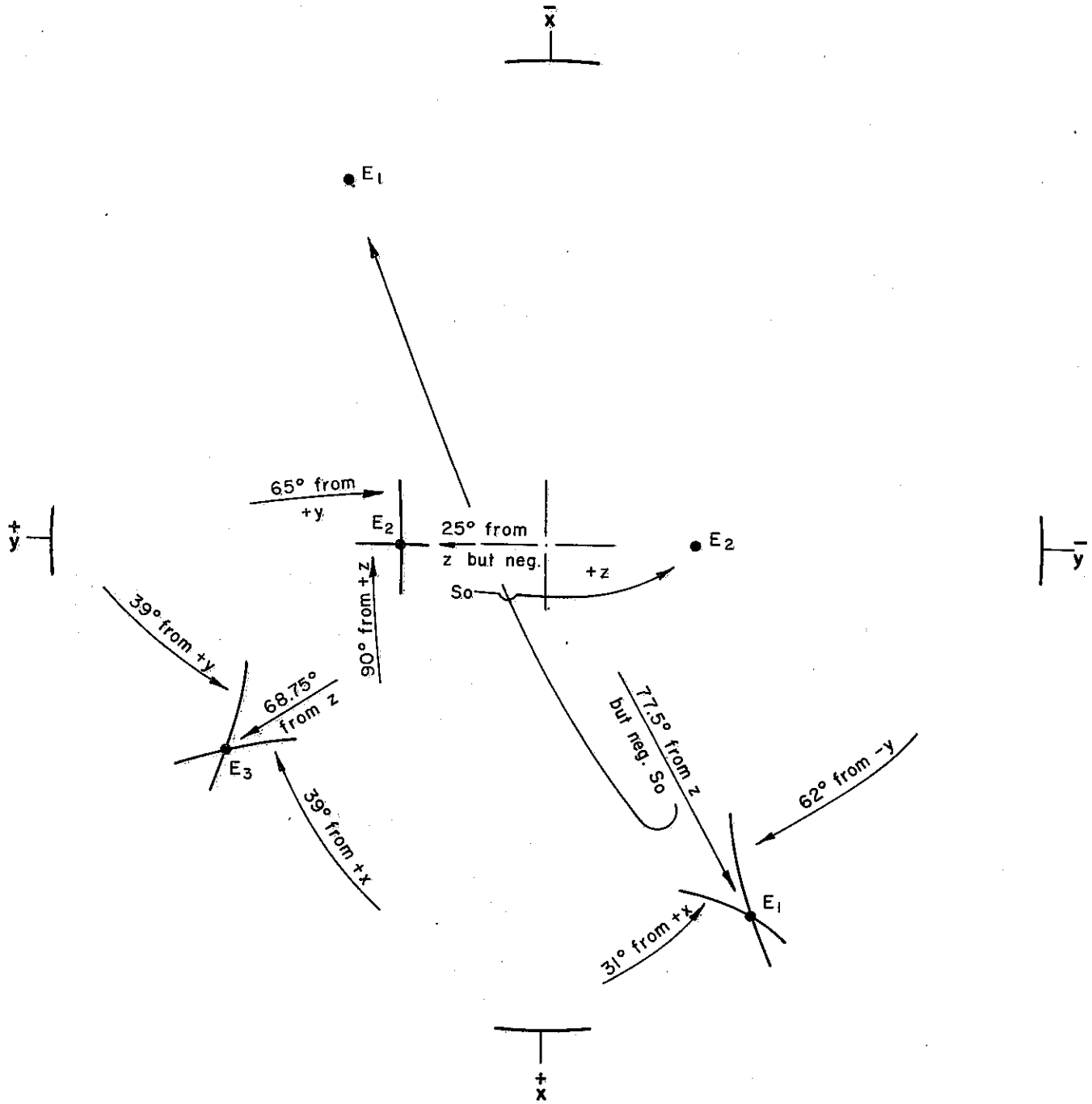

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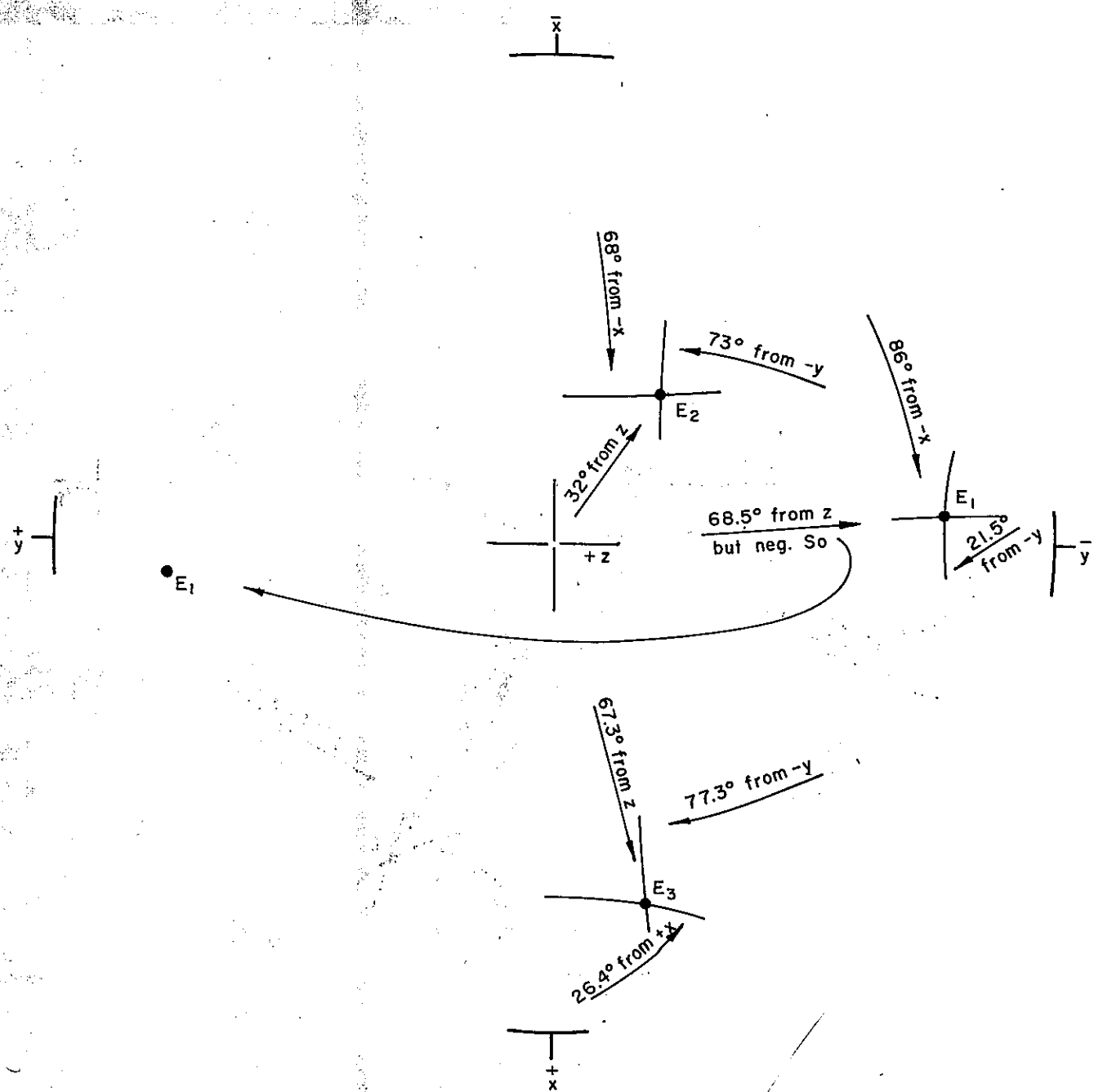

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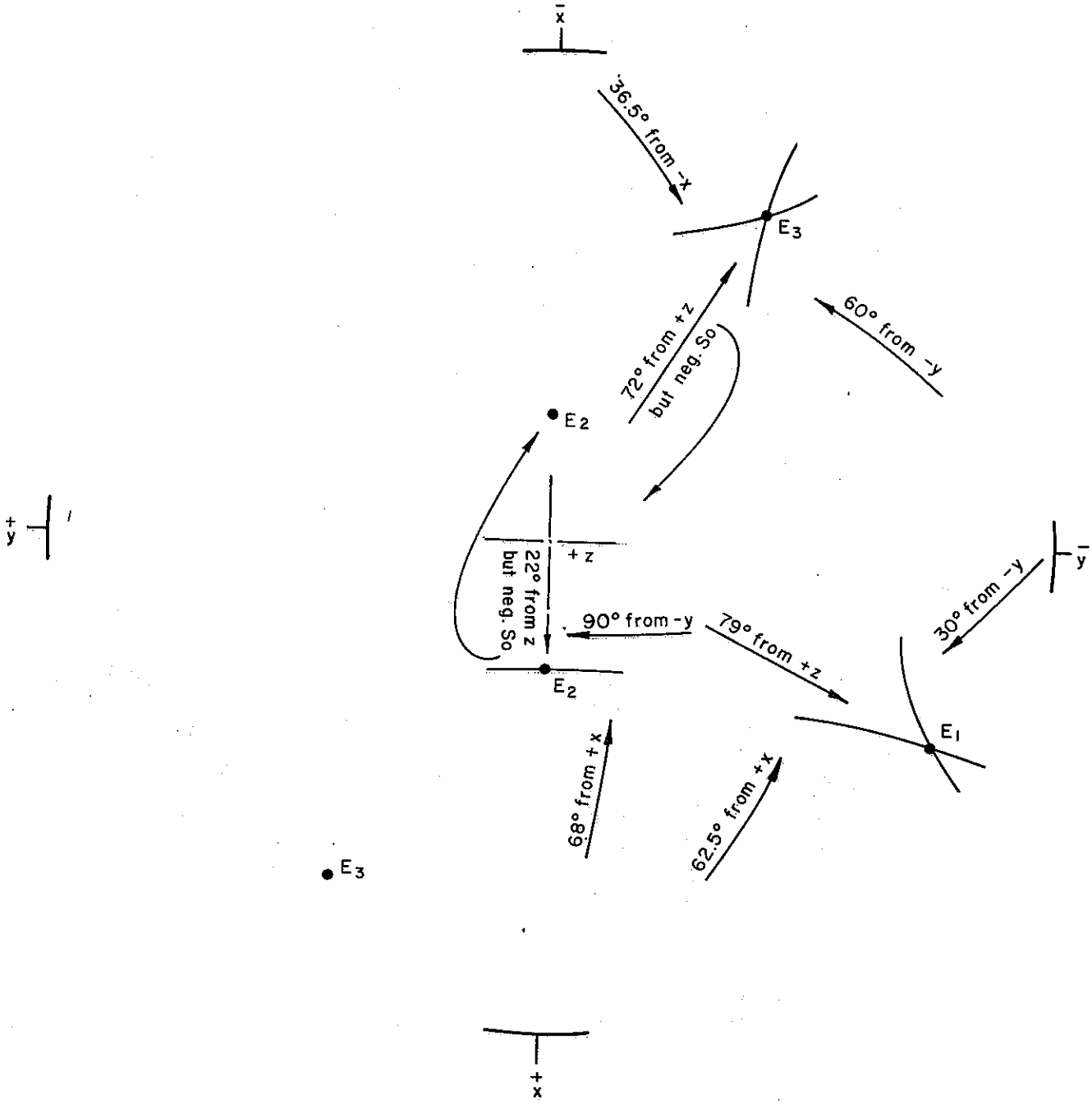

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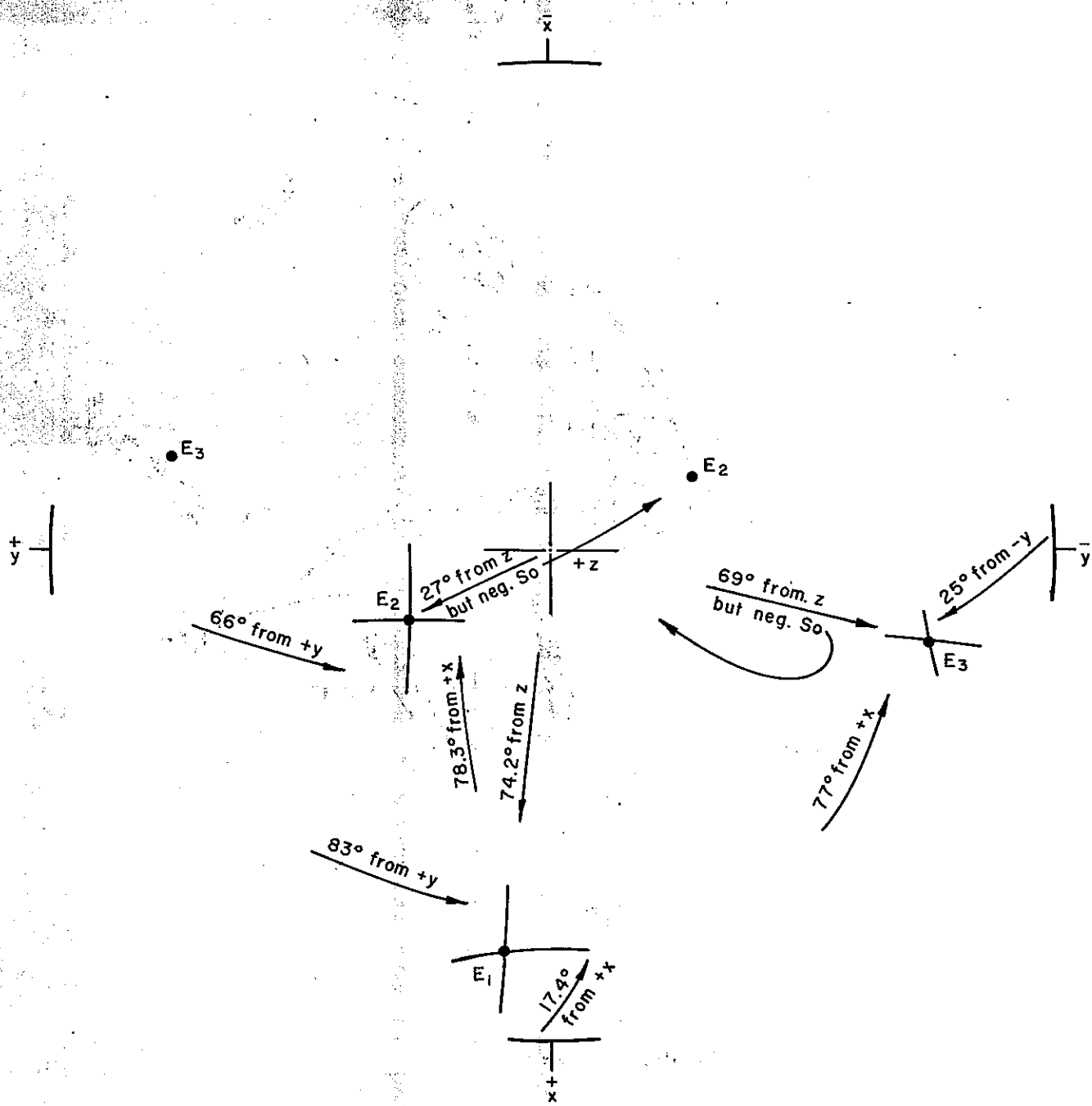

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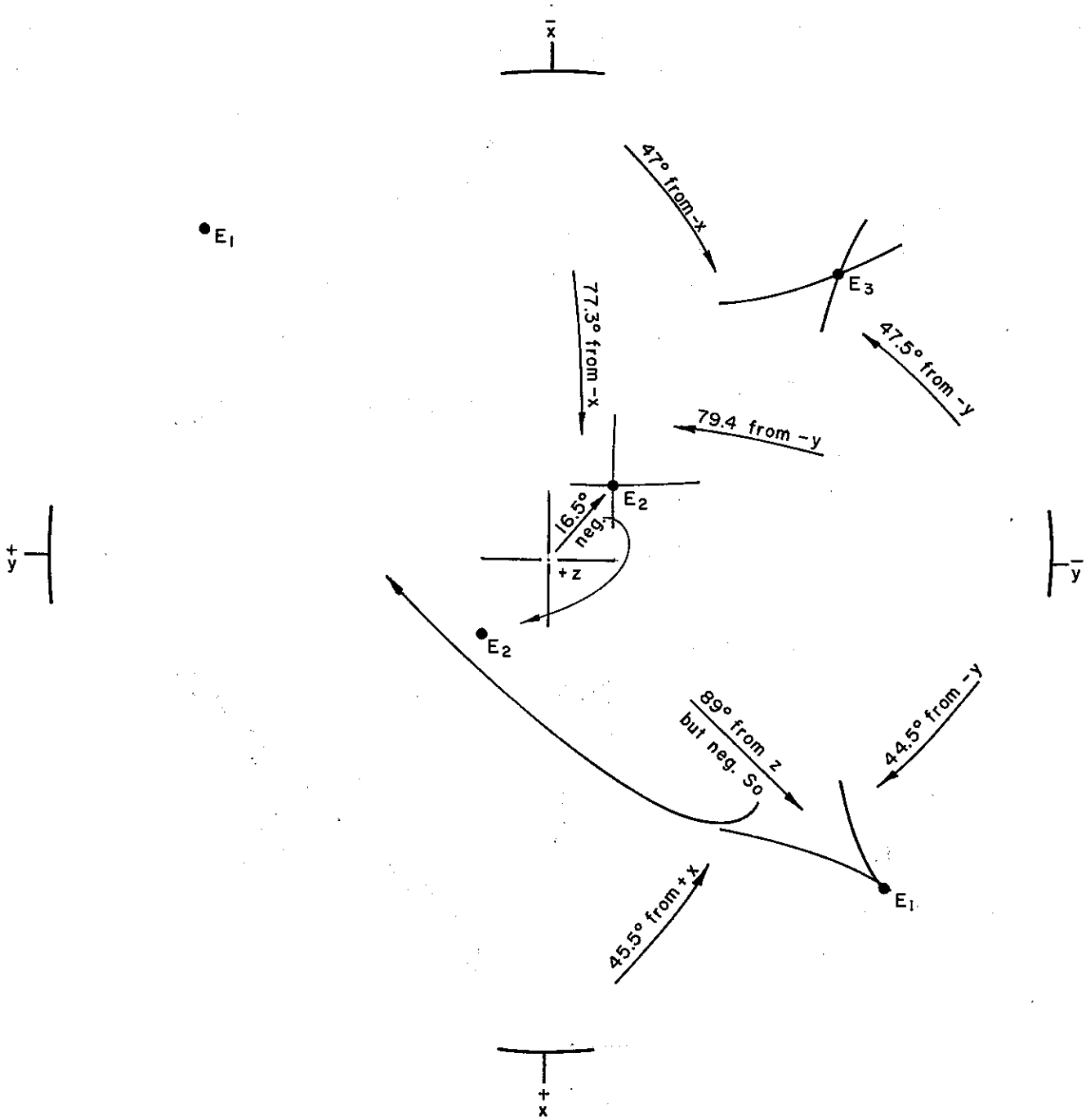

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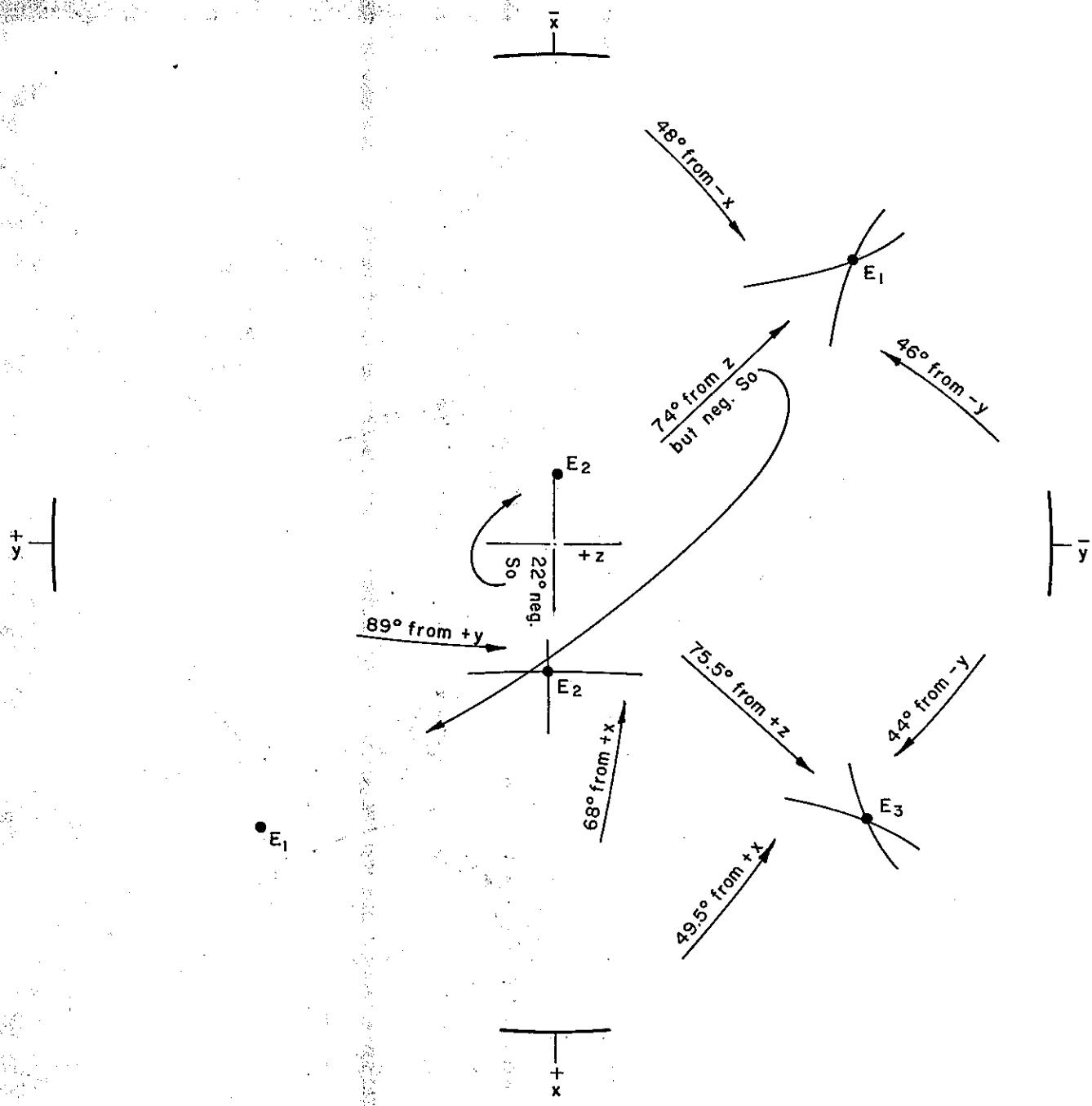

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TECHNICAL REPORT STANDARD TITLE PAGE

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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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STATE OF CALIFORNIA—BUSINESS AND TRANSPORTATION AGENCY

RONALD REAGAN, Governor

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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ACKNOWLEDGEMENTS

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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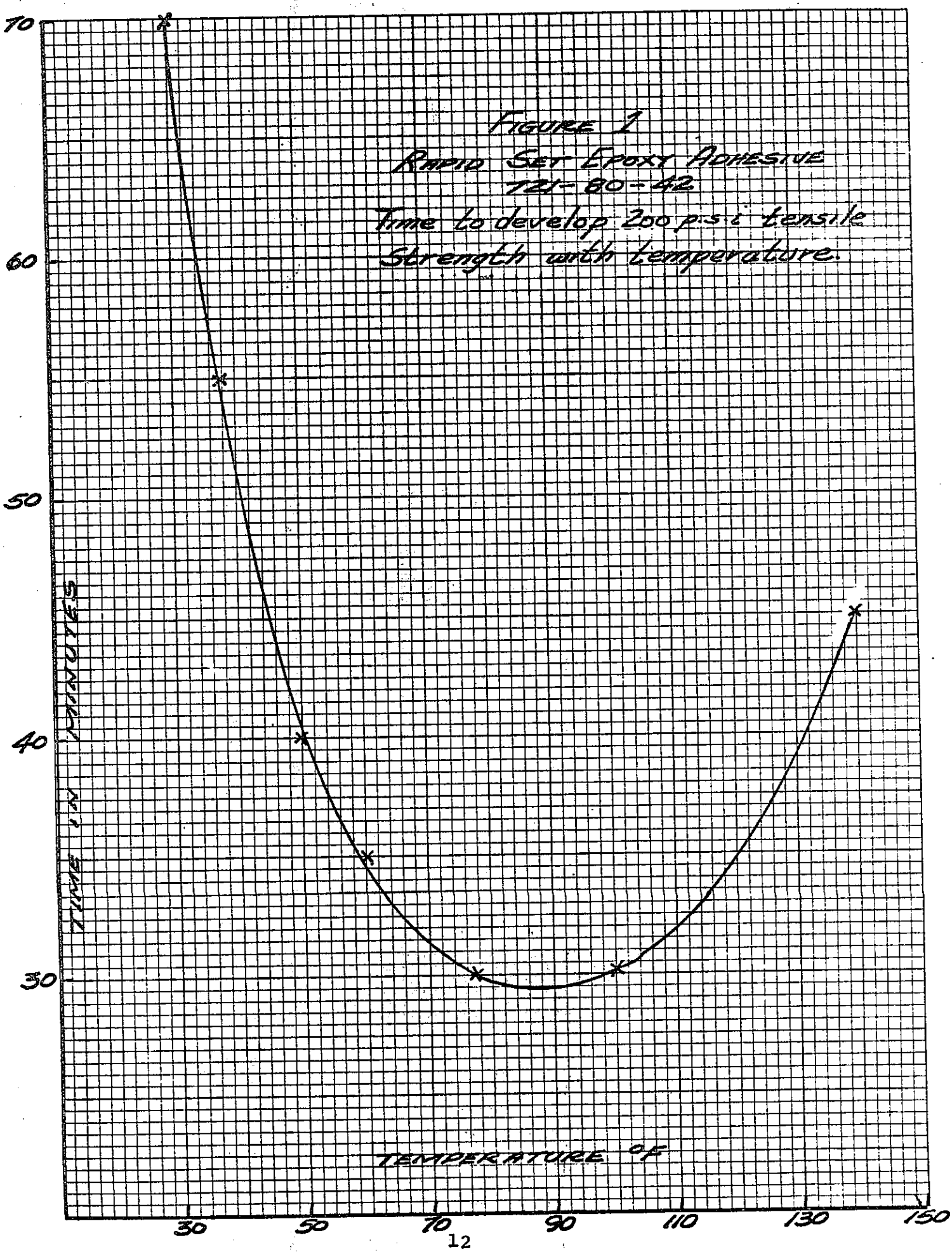

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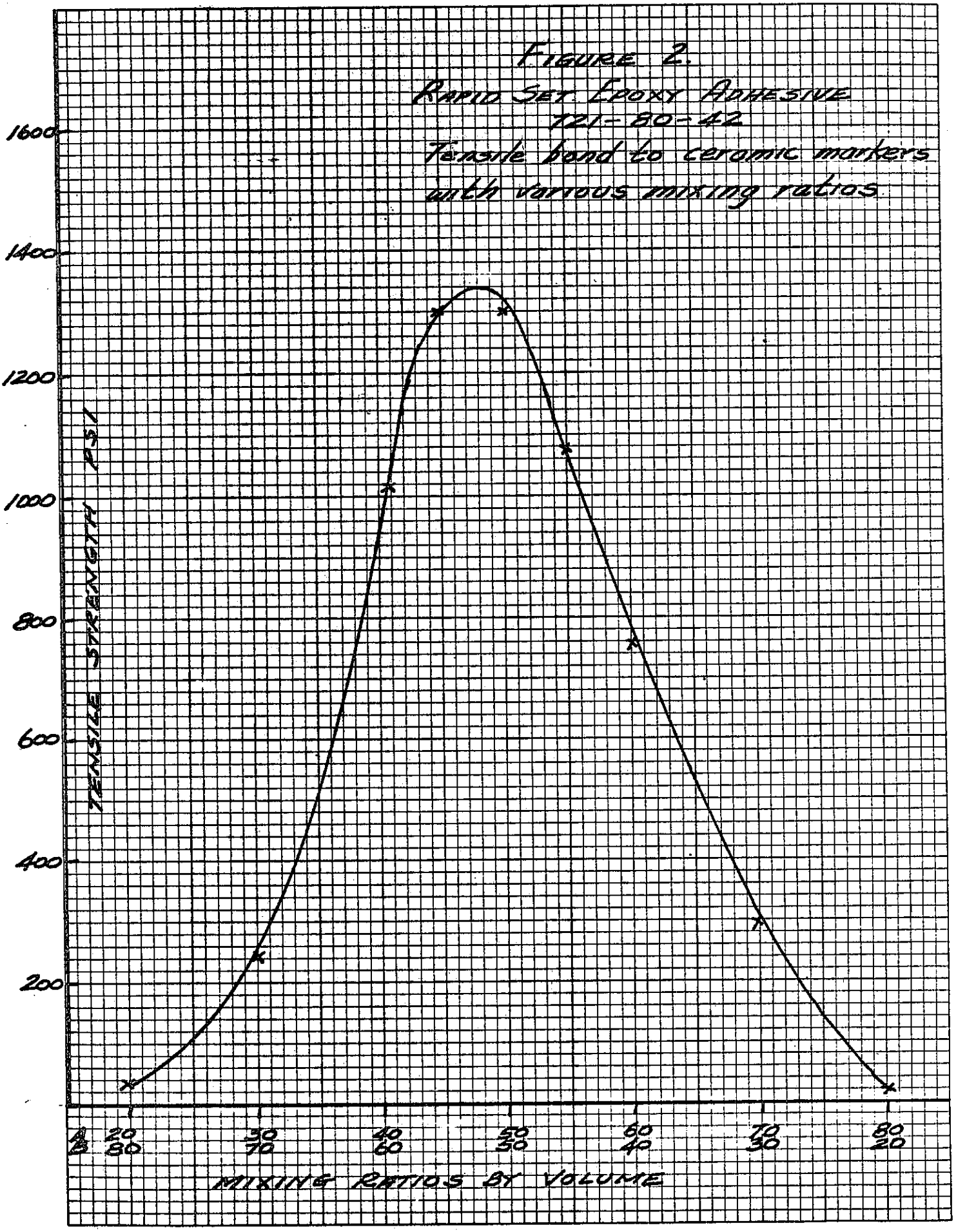

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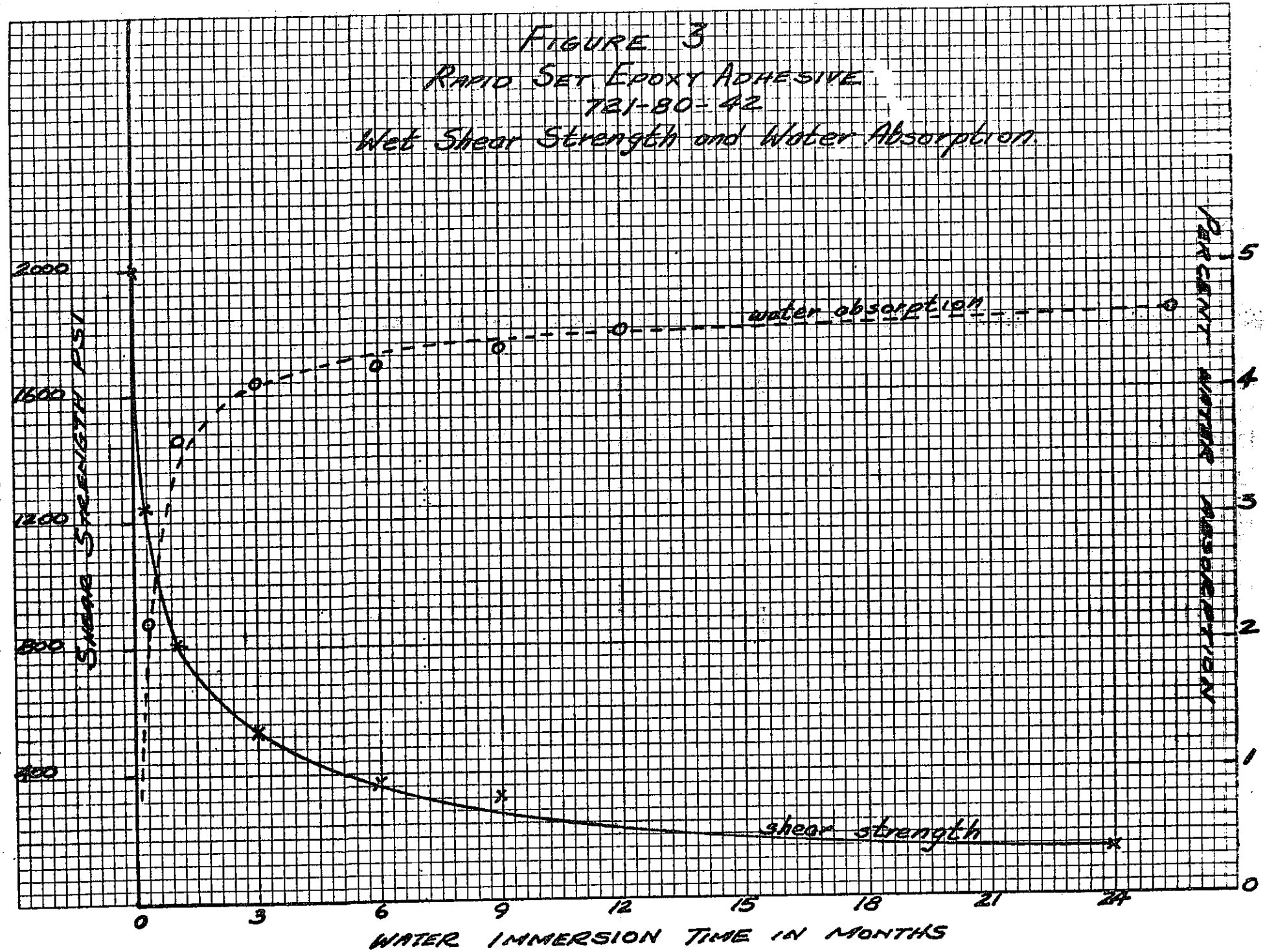

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APPENDIX

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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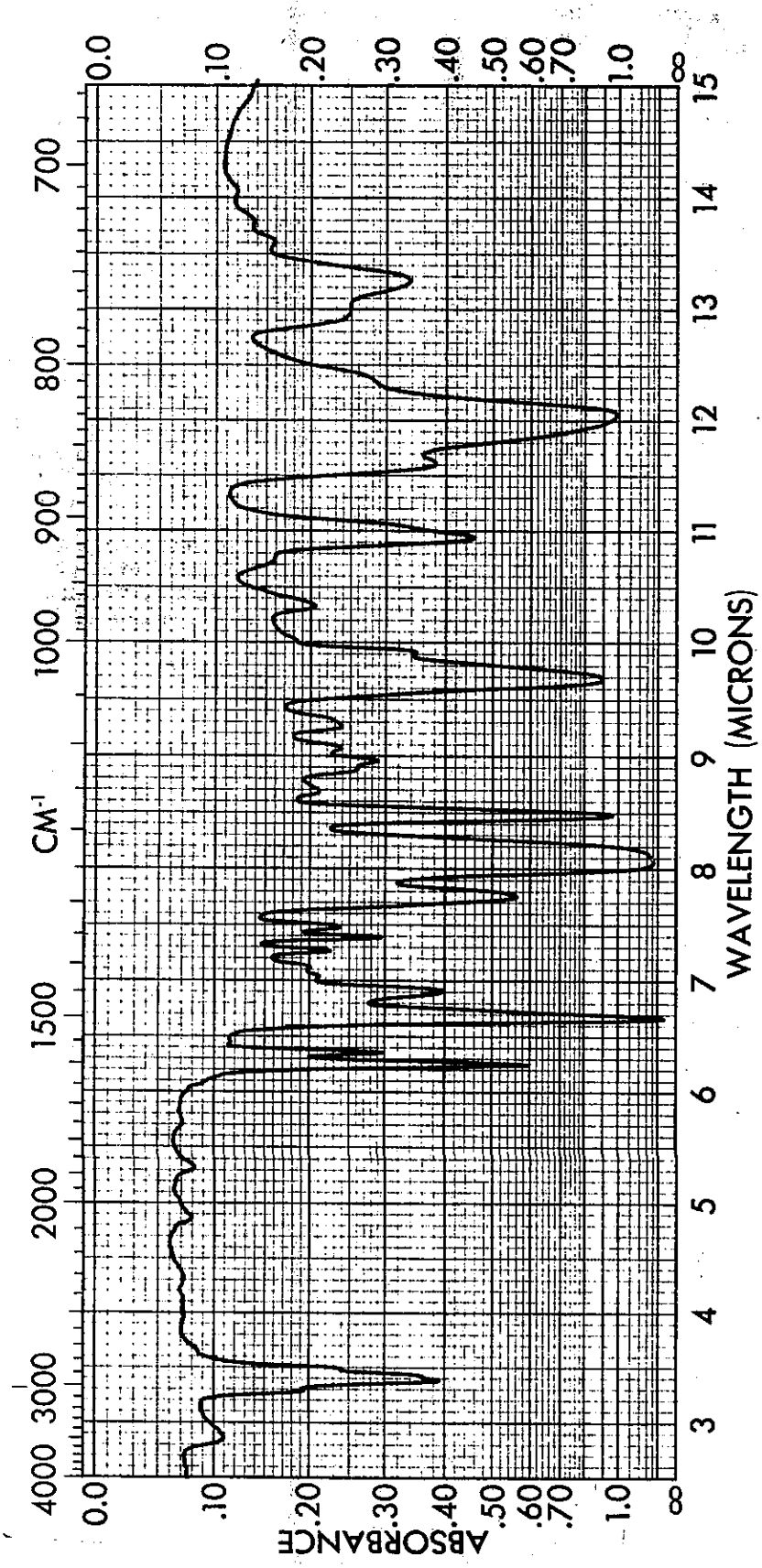

TEST METHOD 425

STANDARD INFRA RED ABSORPTION CURVE

COMPONENT A

RAPID SET ADHESIVE

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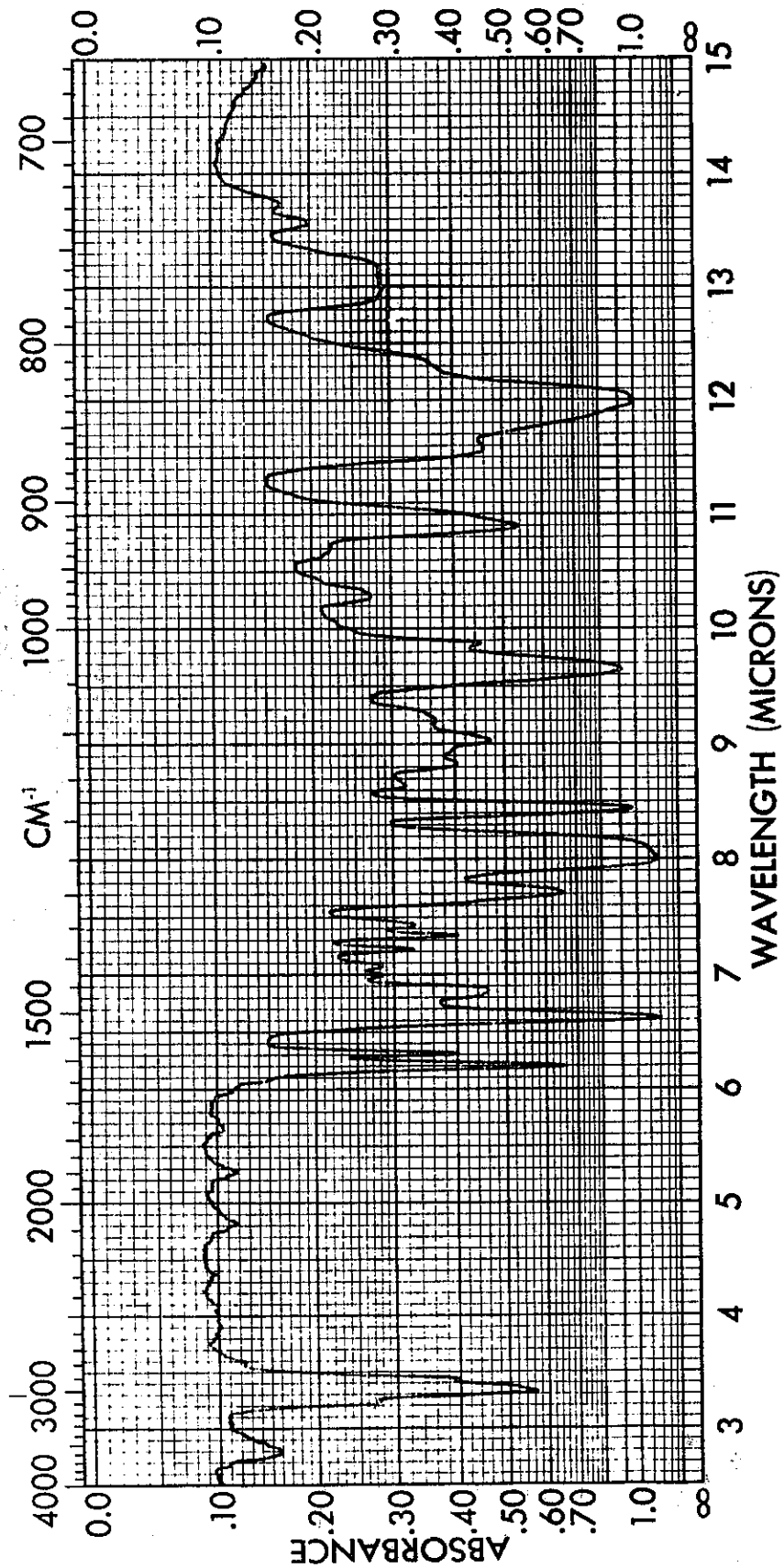

TEST METHOD 425
STANDARD INFRARED ABSORPTION CURVE
COMPONENT A
STANDARD SET ADHESIVE

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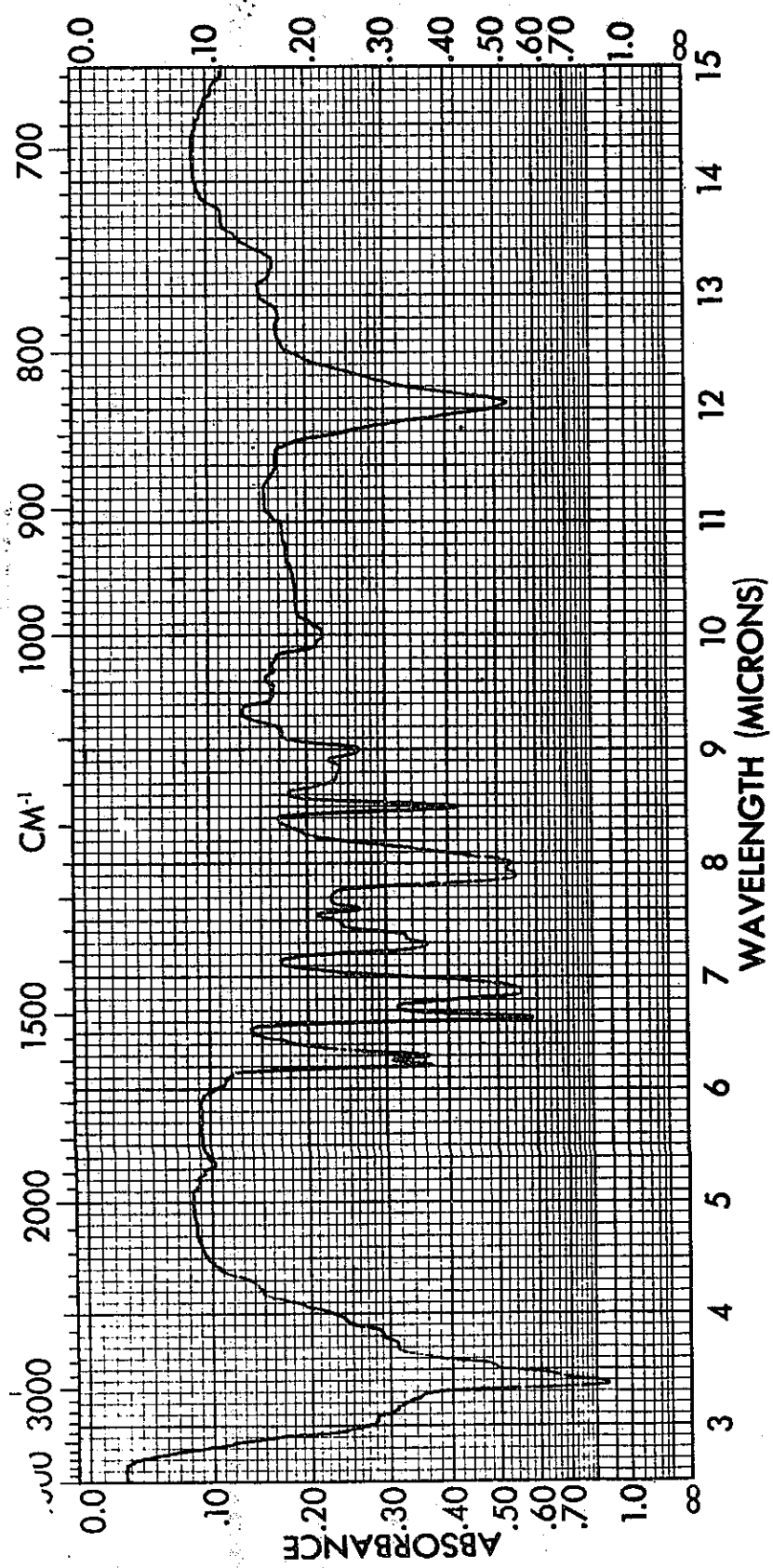

TEST METHOD 425
STANDARD INFRARED ABSORPTION CURVE
COMPONENT B
STANDARD SET ADHESIVE

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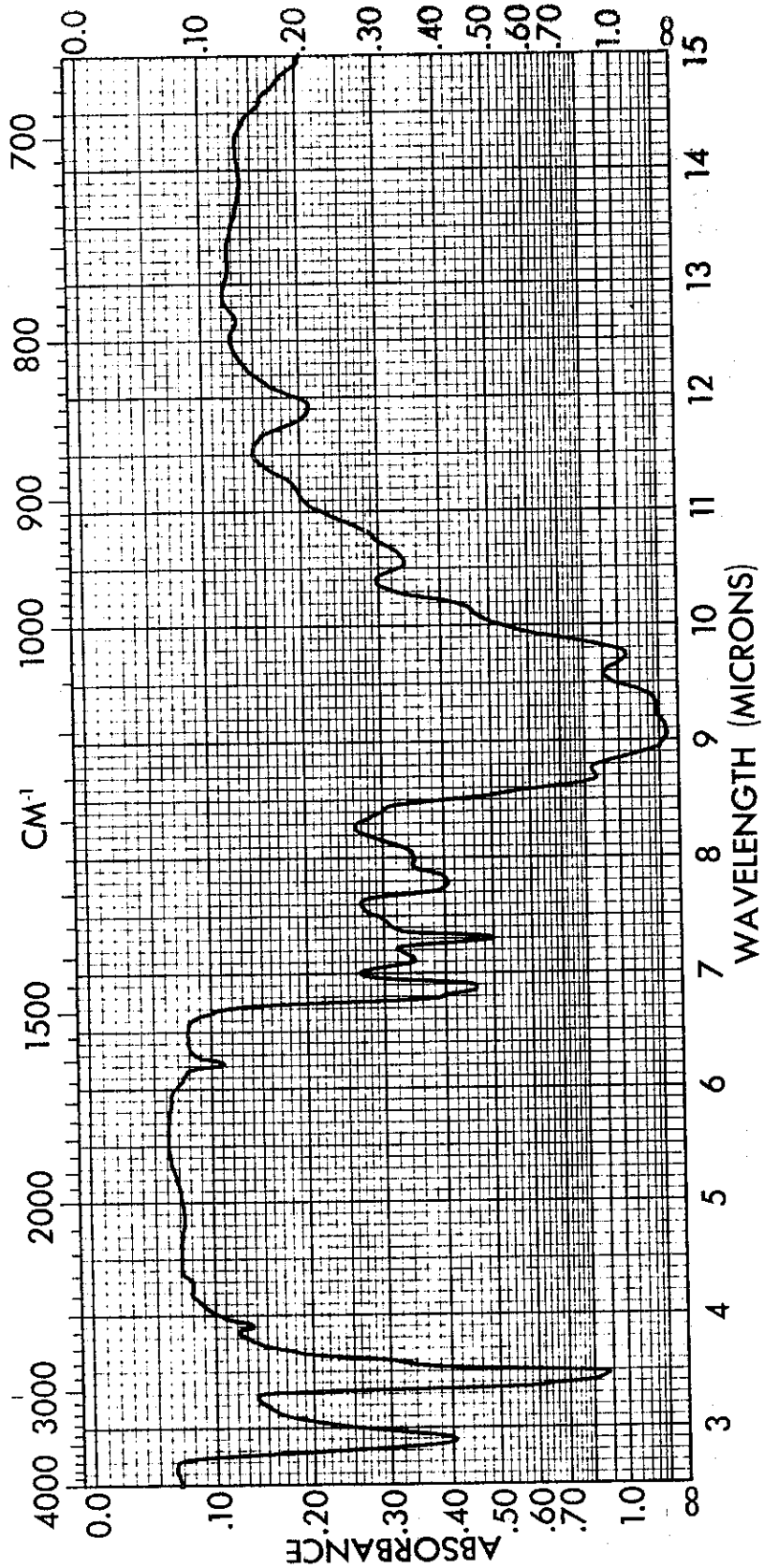

TEST METHOD 425
STANDARD INFRA RED ABSORPTION CURVE
COMPONENT B
RAPID SET ADHESIVE

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