DEVELOPMENT OF EPOXY MATERIALS FOR HIGHWAY USE

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Foreword

The epoxy resins are among the newest of the major plastic materials. The majority of the epoxy resins are produced by reacting two chemicals, bisphenol A and epichlorohydrin. Both of these materials can be obtained from petroleum. The epoxies are thermosetting resins. A thermosetting material is one which can be changed, by application of heat or by chemical means, into a solid product which cannot be melted by application of heat or dissolved in organic solvents. The epoxy resins are changed into the finished plastic by chemical means. They are reacted with curing agents or hardeners to form hard, infusible systems.

The epoxies have certain properties which lend them to construction applications, notably the ability to bond plastic concrete to hardened concrete. In the late 1950's these materials came to the attention of highway personnel. Some of them were used as adhesives and patching materials in the various Districts of the Texas Highway Department. Their potential uses as concrete adhesives, concrete patching compounds, protective coatings and the like were quickly recognized. However, it was soon discovered that many different types of epoxy materials were available, not all of which performed satisfactorily in highway applications. Even when it was possible to write adequate specifications for the type of epoxy desired, there were many different brands available and uniform results were difficult to obtain with these various materials.

In order to make the use of epoxies feasible, the Materials and Tests Division was asked to help by providing suitable specifications. Initial investigation of the proprietary epoxy materials which were available indicated that it would be difficult to obtain brand-name products which would consistently perform well in highway applications. It was decided that it would be best both from the standpoint of performance and economics for the Materials and Tests Division to develop epoxy formulations specifically for highway uses and then purchase these materials on a formulation basis in a manner similar to that in which the Highway Department obtains paint.

This report is presented in three parts. Part I deals with the development of an epoxy adhesive for highway use. Part II describes the development of an epoxy binder for maintenance applications. The distinction made between an adhesive and a binder in this report is as follows: An adhesive is applied in thin coats to material surfaces and acts as a bonding agent between these materials. A binder acts as a cement for various types of aggregate to form a mortar or concrete. Part III is concerned with the weathering and aging characteristics of epoxy adhesive and binder formulations.

PART I

DEVELOPMENT OF AN EPOXY ADHESIVE

Development of Epoxy Materials for Highway Use

Part I

SCOPE

The purpose of this project was to develop an epoxy adhesive specifically for highway use. The adhesive was to be designed mainly for the following uses:

1. Bonding plastic portland cement concrete to existing concrete structures.
2. Bonding cured concrete to existing concrete structures.
3. Bonding steel to hardened or plastic concrete.
4. Bonding steel to steel where high structural strengths are not required.

OBJECTIVES

In order for the epoxy adhesive to perform satisfactorily in the above mentioned applications, it must meet certain requirements. The objective of this project was to develop from the basic raw materials an epoxy adhesive having the desired characteristics. The following requirements were placed on the proposed epoxy adhesive:

1. The adhesive must be able to bond under wet conditions.
2. It must be usable at temperatures between 60 and 105 degrees F.
3. It should be of such a consistency that it can be applied by brush.
4. A one gallon batch should have a usable pot life of not less than 30 minutes at an ambient temperature of 80 degrees F.
5. The adhesive should be sufficiently resistant to flow that a suitable glue line of 20 to 40 mils (0.02 to 0.04 inch) in thickness can be maintained on vertical surfaces.
6. The time of application of plastic (wet) concrete to the epoxy adhesive should not be overly critical.
7. The epoxy adhesive should develop a good bond to steel as well as to concrete.

CONCLUSIONS

As a result of the original investigational work on epoxies, a modified epoxy-polysulfide adhesive was developed. It was designated as Texas Highway Department Epoxy Adhesive A-100 and was used in limited quantities on an experimental basis. This material complied with the requirements for an adhesive set forth in the scope of this project. This formulation was subsequently modified slightly and designated as Epoxy Adhesive A-101. Approximately 250 gallons of this material were purchased and used on one specific project. As a result of weathering and aging tests carried out on the epoxy materials, which are discussed in Part III of this report, it was found that the epoxy-polysulfide systems did not perform as well for our purposes as the polyamide and modified amine-epoxy combinations. As a result of additional investigation

an epoxy adhesive with a combination of a modified amine and a polyamide as the curing agent was developed. This formulation was designated as Epoxy Adhesive A-102 and 475, 3/4 gallon units of this material were purchased. Field experience with this material indicated satisfactory performance except for the fact that it was difficult to apply evenly by brush. Also it was found that almost all of the adhesive material was being used strictly in concrete applications. This was taken into consideration and another formulation, designated as Epoxy Adhesive A-103, was developed. This material was designed to have better handling characteristics. The physical properties of the cured adhesive were quite similar to A-102 with the exception of the impact strength, which was lower. Also, it was somewhat lower in cost than Epoxy Adhesive A-102. Approximately 3000 gallons of this material have been purchased to date at an average cost of about $9.00 per gallon, including inspection and testing expenses. Indications are that this material is performing satisfactorily in the field. The following conclusions concerning an epoxy adhesive formulation specifically for highway use were reached.

1. The best type of epoxy resin among those tested is a low viscosity unmodified type.
2. There is no significant difference between the equivalent resins produced by different manufacturers.
3. Of the curing agents tested, a modified amine or modified amine-polyamide combination was found to be the best.
4. With regard to the fillers tested, minus 325 mesh tabular alumina performed best followed by minus 325 mesh silica flour. Considering the comparative cost of these two materials, silica flour is the better all around filler.
5. The best thixotropic agent among those tested, considering both performance and ease of incorporation into the formulation, is M-5 Cab-O-Sil (colloidal silica).
6. The use of solvents is not recommended because in many cases, they detract from the physical properties of the adhesive.

As a result of this experimental work, the Texas Highway Department has available a comparatively low cost structural epoxy adhesive which will perform well in highway applications.

MATERIALS

The specific materials obtained for possible use in an adhesive formulation are listed in groups according to the various types of materials.

Epoxy Resins

The basic epoxy resin may be one of several types. It may range from a solid at room temperature to a liquid with a viscosity as low as 500 cps. It may contain modifiers to obtain specific properties. The epoxy resins evaluated for use in the adhesive were all liquid resins which could be grouped into three classifications: regular unmodified resins, low viscosity unmodified resins, and modified resins. All of the resins considered were reaction products of bisphenol A and epichlorohydrin. The resins are listed in Table 1.

Table 1

Identification of Resins

Resin Designation	Manufacturer	General Type
Epon 828	Shell Chemical Co.	Regular Unmodified (10,000 - 16,000 cps. viscosity at 25 degrees C.)
Araldite 6010	CIBA Chemical Co.	Regular Unmodified (10,000 - 16,000 cps. viscosity at 25 degrees C.)
Epotuf 6140	Reichhold Chemical Co.	Regular Unmodified (10,000 - 16,000 cps. viscosity at 25 degrees C.)
*Epi-Rez 509	Jones-Dabney Co.	Low Viscosity Unmodified (7,000 - 10,000 cps. viscosity at 25 degrees C.)
Epon 815	Shell Chemical Co.	Modified (500-900 cps. viscosity at 25 degrees C.)
Araldite 506	CIBA Chemical Co.	Modified (500-900 cps. viscosity at 25 degrees C.)
Epi-Rez 5071	Jones-Dabney Co.	Modified (500-900 cps. Viscosity at 25 degrees C.)

*This resin became available after initial evaluation of materials.

Of the above resins, the Epon 828, Araldite 6010, and Epotuf 6140 were rated by the manufacturers as equivalent materials. The Epon 815, Araldite 506, and Epi-Rez 5071 also were rated as equivalent materials. The resins in the latter group contain approximately 10% butyl glycidyl ether as a modifier which is added mainly to lower the viscosity. The butyl glycidyl ether is referred to as a reactive diluent in that it reacts with the curing agent along with the epoxy resin.

Curing Agents

The curing agents or curing agent systems which were considered for use in the adhesive can be placed in the five following categories:

1. Aliphatic amines
2. Modified amines and modified amino-amides
3. Polyamides
4. Polysulfides
5. Aromatic amines

A list of the curing agents obtained for possible use in an adhesive is given in Table 2. It is suggested that the reader refer to this table from time to time in order to identify materials under discussion.

Table 2

Identification of Curing Agents

Curing Agent	Manufacturer	Type	Parts per 100 Parts of Epoxy Resin by Weight Generally Used
Diethylene Triamine	Union Carbide	Aliphatic Amine	9 to 12
Genamid 250	General Mills	Modified Amine	40 to 50
Lancast A	CIBA Chemical Co.	Modified Amine	40 to 50 plus 2 to 3 DMP-30
*DP-134	CIBA Chemical Co.	Modified Amine	100
*Epi-Cure 87	Jones-Dabney	Modified Amine	20
*Epi-Cure 874	Jones-Dabney	Modified Amine	15 to 25
*Epi-Cure 872	Jones-Dabney	Modified Amino-amide	35
*Epi-Cure 855	Jones-Dabney	Modified Amino-amide	40 to 60
*Genamid 2000	General Mills	Modified Amino-amide	40 to 50
Versamid 115	General Mills	Polyamide	50 to 100
Versamid 125	General Mills	Polyamide	50 to 75
Versamid 140	General Mills	Polyamide	40 to 60
Thiokol LP-3	Thiokol Chem. Co.	Polysulfide	50 to 100 plus 5 to 10 DMP-30 or a combination of DMP-10 and DMP-30
DMP-10 (Dimethyl Amino Methyl Phenol)	Rohm and Haas	Aromatic Amine	See above
DMP-30 (Tridimethyl Amino Methyl Phenol)	Rohm and Haas	Aromatic Amine	" "

*These curing agents became available after initial evaluation of materials.

Of the various classifications of amines shown on page 3, the aliphatic amines are chemically the simplest. Epoxies cured with aliphatic amines are rigid, rather brittle materials. The modified amines and the modified amino-amides usually consist of amines which have been partially reacted with epoxy resin or some other material. In some cases these curing agents are blends of partially reacted amines. These curing agents have the advantage of more convenient mixing ratios with the epoxy resins, and usually will produce a cured material that is tougher and more flexible than that produced by the aliphatic amines. The polyamides are produced by reacting various fatty acids with amines. The polyamides have the advantage of a wide range of mixing ratios with the epoxy resins and a corresponding range of properties.

The polysulfides might be thought of as a sort of liquid synthetic rubber. Thiokol LP-3 is a dark colored liquid with a viscosity of 700 to 1400 cps. at 77 degrees F. The ratio of polysulfide to epoxy resin varies depending upon the application and the flexibility desired. The higher the ratio of polysulfide, the more flexible will be the resultant material. The polysulfides react so slowly with the epoxy resins that addition of an amine curing agent is necessary to promote the reaction. Several different amines can be used for this purpose. The curing agent recommended for promoting this reaction at temperatures in the 60 to 100 degree F. range is DMP-30 or a combination of DMP-30 and DMP-10. These curing agents fall into the aromatic amine category. They act as catalysts in promoting the reaction between the epoxy resin and the polysulfide. The amine does not actually become a part of the epoxy-polysulfide polymer which makes up the cured material. The total amount and combination of these catalytic curing agents will determine the pot life and rate of cure.

Fillers

Fillers serve the following purposes when added to epoxy adhesive formulations.

1. They extend the pot life or working time of an adhesive formulation.
2. The proper type and amount of filler can enhance the physical properties of the adhesive.
3. Fillers help provide a thicker glue line where this is desirable.
4. Fillers extend the adhesive formulation, thus reducing the cost.

Fillers have the disadvantage of increasing the viscosity of the formulation and making it more difficult to mix and apply. Also, large amounts of filler can degrade the properties of the adhesive.

The fillers listed in Table 3 were evaluated for possible use in the adhesive formulation.

Table 3

Identification of Fillers

Filler	Manufacturer
China Clay, ASP 103	Minerals and Chemicals Corp. of America
China Clay, ASP 400	Minerals and Chemicals Corp. of America
Aluminum Oxide, Tabular (Minus 325 Mesh)	Aluminum Company of America
Asbestos 7TF1	Johns-Manville Co.
Mica, Alsibronz #12	Franklin Mineral Products Co.
Silica Flour (Minus 325 Mesh)	Pennsylvania Glass Sand Corp.

Thixotropic Agents

Thixotropic or gelling agents help prevent filler settling in the adhesive during storage and give the adhesive sufficient resistance to flow that an adequate glue line may be maintained on vertical surfaces. One of the original requirements for the adhesive to be formulated was that it should be sufficiently resistant to flow that a suitable glue line of 20 to 40 mils (0.02 to 0.04 inch) in thickness might be maintained on vertical surfaces. The thixotropic agents considered were as follows:

Table 4

Identification of Thixotropic Agents

Thixotropic Agent	Manufacturer
Bentone 27	National Lead Company
M-5 Cab-O-Sil	Godfrey L. Cabot, Inc.
Asbestos 7TF1	Johns-Manville

The Bentone 27 is described as an organic compound of a special hydrous silicate mineral while the M-5 Cab-O-Sil is a colloidal silica. The Asbestos 7TF1 has already been listed as a filler. However, this material will thicken and provide flow resistance in an epoxy compound so it was also considered as a thixotropic agent.

Solvents

Organic solvents in an epoxy formulation serve mainly to lower the viscosity of the material and thus make it more workable. Solvents also will extend the

pot life and the cure time of the material. Three solvents, toluene, xylene, and methyl ethyl ketone, were experimented with in adhesive formulations.

PROCEDURE

The first step in developing an epoxy adhesive and a binder was to survey the literature available on the subject of epoxies. A number of technical articles were reviewed along with two of the better known books on the subject - Epoxy Resins by Irving Skeist and Epoxy Resins by Henry Lee and Kris Neville. Following a review of the literature, several manufacturers of the basic epoxy resins and curing agents were contacted concerning the proposed development of materials specifically for highway uses. Several of the companies offered samples of their epoxy resins and curing agents along with technical assistance and advice. The Corps of Engineers and several State Highway Departments who were known to have used epoxy materials in structural applications were also contacted. These agencies were asked for copies of any specifications which they had concerning the epoxy materials and their use. Specifications were received from the Corps of Engineers and the states of California, Washington, and Pennsylvania. These specifications were studied prior to beginning any laboratory work.

After obtaining background information on the epoxies along with samples of raw materials and technical advice concerning their use, the next step was to devise a plan to evaluate the various raw materials and combinations thereof. It was decided to work first on the development of the epoxy adhesive.

Any epoxy compound for use at temperatures between 60 and 105 degrees F. must consist of at least two components - the basic epoxy resin and a curing agent. When these two materials are mixed together, they will react chemically to form the hardened epoxy material. In addition to these two basic ingredients, an epoxy adhesive will ordinarily contain fillers and a thixotropic or flow control agent. Solvents sometimes are a constituent of epoxy adhesive formulations.

It was decided that the best approach to follow in developing an adhesive would be to start with the simplest combination of constituents, the epoxy resin and curing agent, and attempt to evaluate each constituent separately, if possible. In order to evaluate these various constituents and combinations thereof, a series of tests had to be developed which would be indicative of the various properties desired in the epoxy adhesive. Since many different combinations of materials would be considered in developing an adhesive, the tests used should be fairly simple and rapid. Wherever possible, existing equipment was utilized in the testing program. The basic requirement for the adhesive was that it be able to bond plastic portland cement concrete or mortar to existing concrete. In order to determine this property, a variation of ASTM C190-59 (Tensile Strength of Hydraulic Cement Mortars) was devised. This test was designated as Ability to Bond Plastic Portland Cement Mortar to Cured Mortar. The test procedure is as follows:

Ordinary tensile briquettes are prepared according to the method prescribed in the above mentioned ASTM procedure by the Cement Section of Materials and Tests in testing portland cements. These briquettes are loaded in tension until failure occurs. Broken halves of briquettes which on seven day strength

tests failed at 400 pounds or higher are obtained. These halves are allowed to dry for approximately 7 days in the laboratory and the broken surfaces are wire brushed to remove any loose mortar. The epoxy compound to be tested then is brushed on the broken surface. After the epoxy becomes tacky, the briquette halves are placed singly in briquette molds and fresh mortar having a ratio of 1 part high-early strength cement to 3 parts Ottawa sand by weight is molded against the epoxy to form a complete briquette. The resulting briquettes then are cured for 1 day in a humidity cabinet and the remaining time in a water bath according to the procedure set forth in ASTM C190-59. After curing, the briquettes were loaded in tension using a Riehle briquette tester. The load at failure and the type of failure (whether in the mortar or in the bond or in both) was recorded. Figure 1 shows one of the composite briquettes in the testing machine.

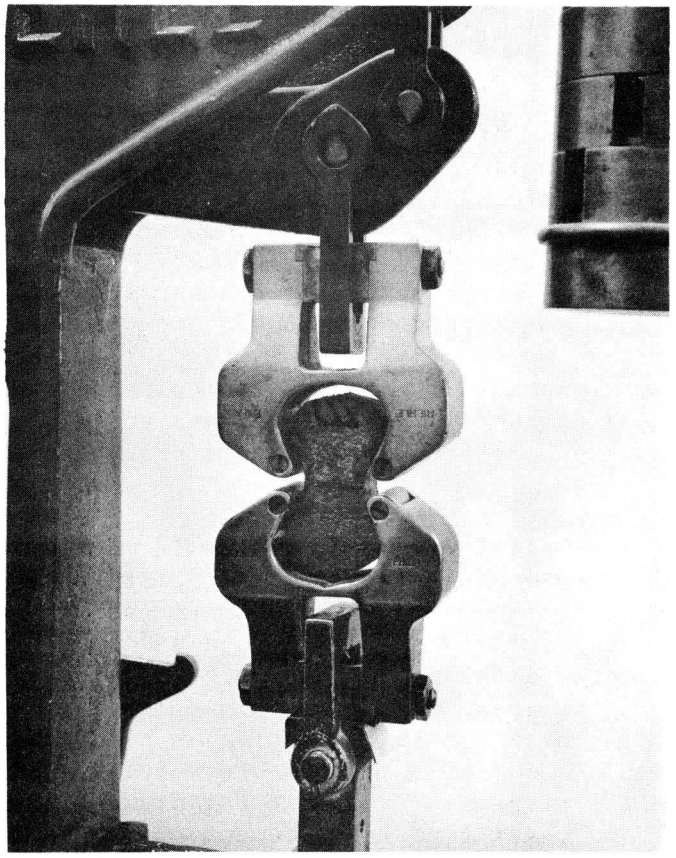

Figure 1.
Briquette Specimen Being Tested to Determine Ability of Epoxy Adhesive to Bond Plastic Portland Cement Mortar to Cured Mortar

It was felt that an epoxy formulation which would bond plastic portland cement concrete to cured concrete successfully would work well for bonding cured concrete to existing concrete structures, so the test just described would give us information on both of these requirements.

In addition to a test to determine ability to bond concrete, tests were needed which would determine the bonding ability of the adhesive to steel and also give more information on the physical characteristics of the various formulations. Several test methods were tried. Among these tests was an adhesive tensile test. For this test, standard 3 inch depth, 5.7 lbs. per foot I-Beam was cut into 2 inch sections and one flange cut off each section to give a T-shaped specimen with a flange area 2" x 2-3/8". A ½" diameter hole was drilled in the center of the web on each of the sections. The flat surface of the flange was prepared by one of several methods and the epoxy to be tested was then applied to the prepared surfaces of two of the sections and the sections were bonded together in a jig to form an adhesive tensile test specimen. The specimens were allowed to cure for approximately 7 days at room temperature and, by means of loose-pin linkages connected to the holes in the webs, were subjected to tensile loading until failure occurred. Figure 2 shows one of these specimens in the process of being tested.

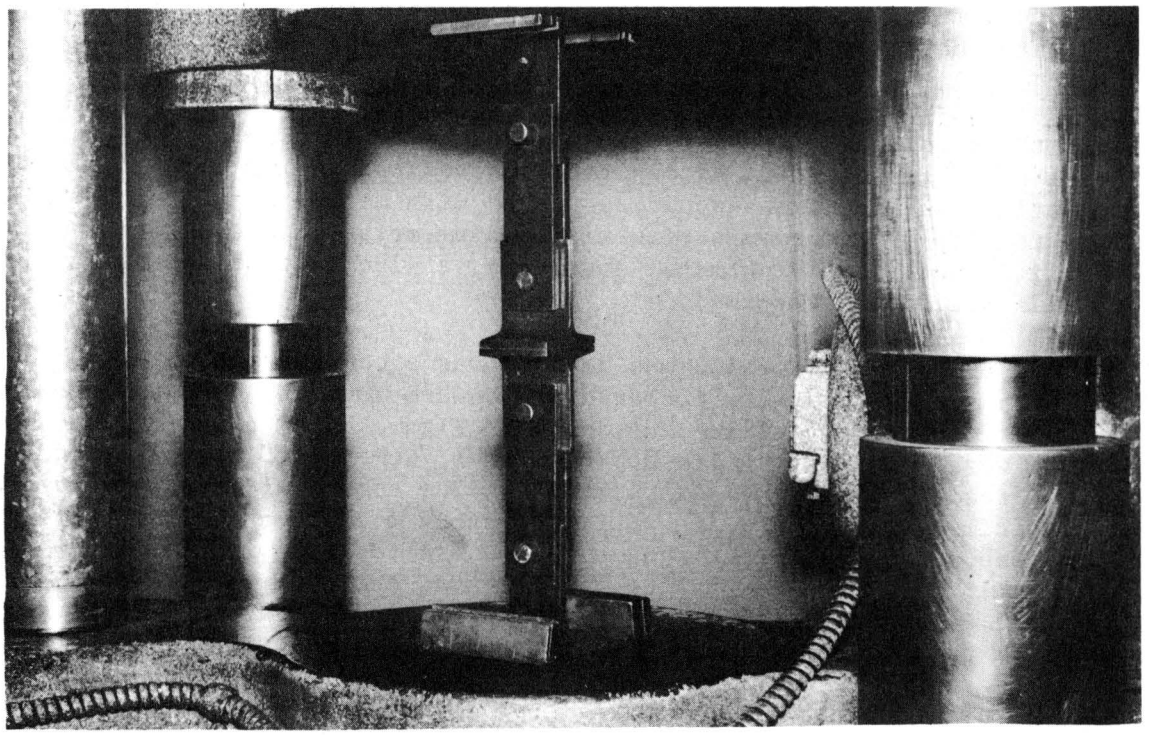

Figure 2.
Adhesive Tensile Test

The adhesive tensile test was used in the early evaluation of epoxy systems, but was abandoned later in favor of other test methods, some of which are described in ASTM Standards. However, valuable information concerning the effect of surface preparation and glue-line thickness on adhesive strength was obtained from this test.

Results are shown in Table 5 for an adhesive formulation of the general type recommended by Thiokol Chemical Company, using various types of surface preparation and two different glue line thicknesses. In the "rotoblast" process which was used to prepare the surfaces of some of the specimens, steel shot is impinged against the surface of the metal. It was found that fragments of the steel shot remained imbedded in the surface of the specimens, resulting in loss of adhesion of the epoxy to the test specimen and consequently, comparatively low strengths. The sandblasting was done with a synthetic grit, Garnet Blasting Abrasive "Gem Blast" 60 mesh (No. 45 to No. 74 U. S. Standard Screens), marketed by Clemtex, Inc. The blasting was done at a gun pressure of 50 to 75 psi using a ¼-inch diameter nozzle.

The results shown in Table 5 indicate that sandblasting, followed by degreasing with sodium metasilicate or methyl ethyl ketone is superior to the other methods of surface preparation considered. The results also indicate that higher strengths can be obtained with thin glue lines.

On the basis of the information obtained from this test, it was decided that the bonding surfaces of all metal adhesive test specimens would be prepared by sandblasting as described above. Following the sandblast operation, the specimens would be washed with methyl ethyl ketone, since this is simpler than degreasing with sodium metasilicate. In all of the adhesive tests involving metal specimens, a glue line of approximately 10 mils would be maintained.

Another adhesive test which was devised was a compressive shear test. Two inch steel cubes were prepared and bonded together with the epoxy under test to form a test specimen as shown in Figure 3. The surfaces to be bonded were sandblasted with No. 60 Garnet "Gem Blast" as discussed above and degreased with methyl ethyl ketone. The epoxy was then applied and the specimen set up in a jig until the epoxy reached an initial set. The total bonded area was four square inches. After allowing the epoxy to cure, a load was applied to the top block as shown in the diagram. This resulted in a compressive shear stress on the bonded areas. The jig shown in the diagram was used to prevent lateral movement. This method was used in testing several of our early formulations but was discarded because of difficulty in preparing good specimens and obtaining reproducible results.

Table 5

Adhesive Tensile Test Results

Method of Surface Preparation	Glue Line Thickness mils	Stress at Failure Avg. PSI	Type of Failure
Rotoblasted and washed with methyl ethyl ketone	10	922	Loss of adhesion to the steel
Ground to white metal and washed with methyl ethyl ketone	10	1596	75% failure in the epoxy, 25% loss of adhesion to the steel
Sandblasted and degreased with a hot 10% solution of sodium metasilicate, then dried in a 212 degree F. oven	10	1840	Failed completely in the epoxy
Sandblasted then acid etched with 10% phosphoric, washed and dried in a 212 degree F. oven	10	1151	75% loss of adhesion, 25% failure in the epoxy
Sandblasted and washed with methyl ethyl ketone	10	2005	Failed completely in the epoxy
Sandblasted and washed with methyl ethyl ketone	25	1611	Failed completely in the epoxy

Note: The formulation used in this test was as follows:

Formulation No. 1

100 parts by weight Shell 828
100 parts by weight minus 325 mesh silica flour
75 parts by weight Thiokol LP-3
10 parts by weight DMP-30

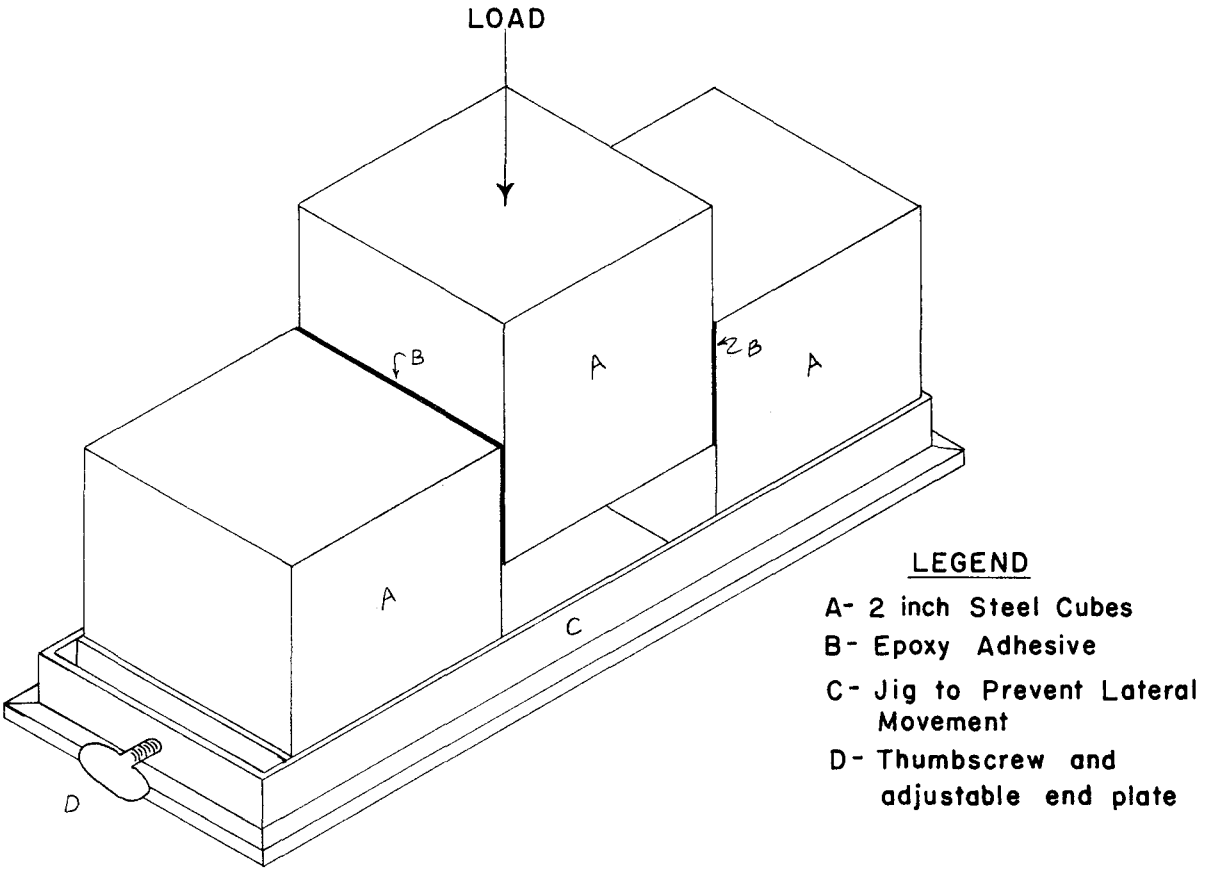

Figure 3.
Compressive Shear Test Specimen
and Retaining Jig

One of the tests which was found to be practical was the Adhesive Shear Strength Test as outlined in ASTM D1002-53T (Strength Properties of Adhesives in Shear by Tension Loading - Metal to Metal.) In this test, specimens of the type shown in Figure 4 are subjected to tensile loading until failure occurs. The alternative of preparing individual specimens rather than multiple panels of specimens was followed. The bonding surfaces of all specimens used in this test were prepared by abrasive blasting as discussed above, followed by washing with methyl ethyl ketone to remove any grease or oil that might be present. A glue line of approximately 10 mils was maintained in preparing the tensile shear specimens.

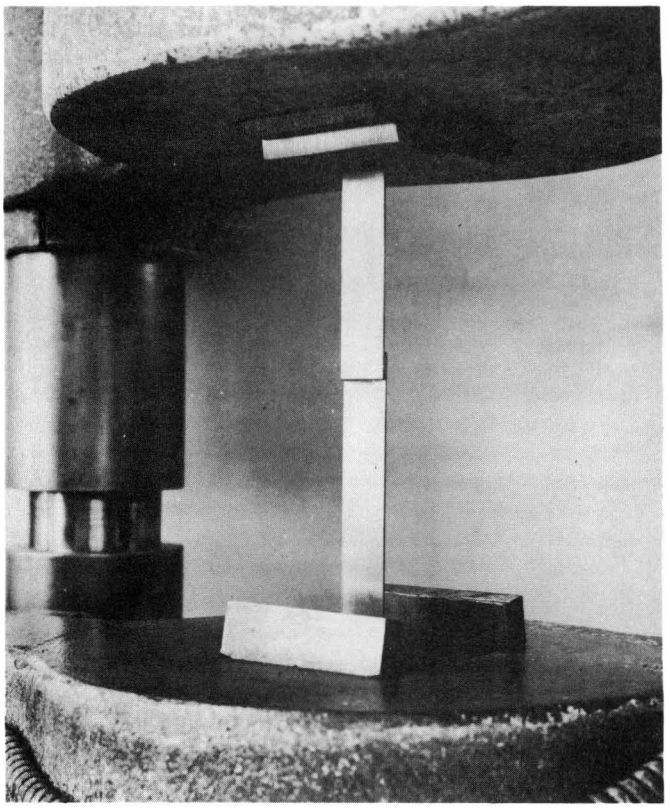

Figure 4.
Adhesive Shear Strength Specimen
in Process of Being Tested

Another test which was found to be suitable was an adhesion test described in ASTM D1062-51 (Cleavage Strength of Metal-to-Metal Adhesives). In this test, a tensile load is applied perpendicular to and at one edge of the bonded area. The bonded area is one square inch. Figure 5 shows a cleavage specimen and the test grips. Figure 6 shows a specimen in the process of being tested. The surfaces of the test specimens to which the adhesive was to be applied were prepared by the same process used in the adhesive shear strength test.

A third test which we used in evaluating the various adhesive formulations was a water absorption test. The water absorption value for an epoxy material indicates completeness of cure or reaction. A low water absorption value indicates that the amount of unreacted epoxy resin and/or curing agent present is comparatively small. In our opinion, the water absorption test also gives an indication as to the weathering and aging characteristics of epoxy materials. Generally, a material with a high water absorption value will not have very good weathering and aging properties. The test used is as described in ASTM D570-57T (Water Absorption of Plastics) with the exception of the specimen size. The

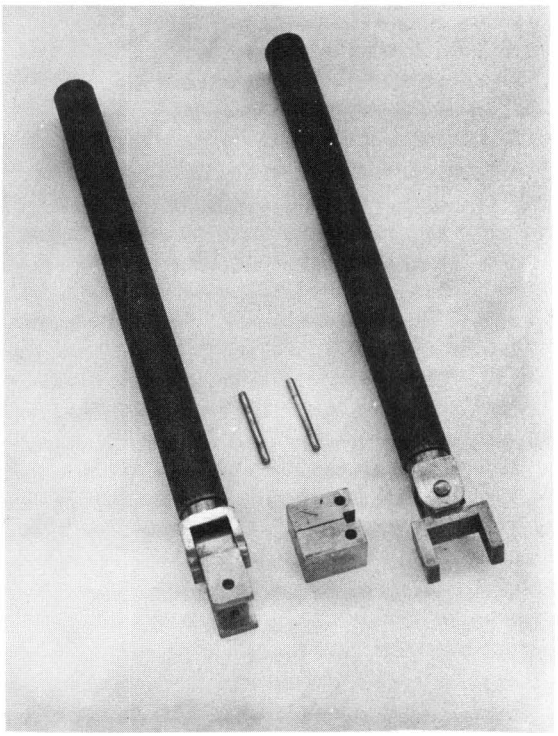

Figure 5.
Cleavage Specimen
and Test Grips

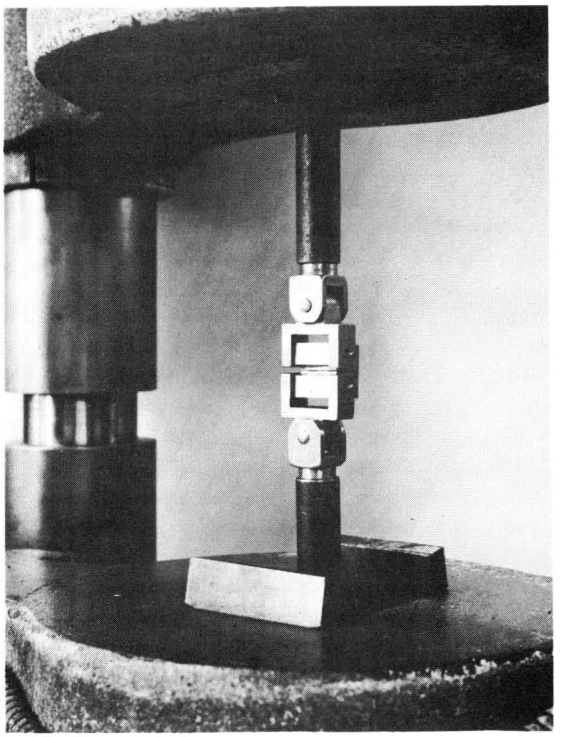

Figure 6.
Cleavage Specimen in the
Process of Being Tested

specimens used were disks 2-3/4 inches in diameter and approximately ¼-inch in thickness. After being allowed to cure for 7 days at 70-80 degrees F., the specimens were immersed in distilled water for 24 hours at 23 degrees C. and the weight gain determined.

For applications involving steel to steel, it was felt that impact resistance would be important, so an impact test was devised and used in evaluating the various adhesive formulations. This test involved dropping a one pound steel ball onto a specimen of the epoxy material. The specimens were of the same type as those prepared for the water absorption test. Both sides of the disks were ground to give plane parallel surfaces. The thickness after grinding was 0.30 inch ± 0.03 inch. The disks rested directly on a concrete floor and the steel ball was dropped on the center of the disk. The initial drop was from a height of 5 feet and then from successively greater heights, increasing in ½ foot increments, until the disk failed. The height at which failure occurred was recorded as the impact strength in ft.-lbs.

In order to determine the working time or pot life of a formulation, a pot life test was devised. The initial method of determining pot life was to

mix a 100 gram batch of the adhesive in a 6 ounce metal ointment can and time the reaction from the initiation of mixing of the two components. The components were stirred together for three minutes with a glass rod. The initial temperature of the two components and the ambient temperature during the test was between 75 and 80 degrees F. The can was placed on an asbestos composition laboratory table top and after 20 minutes time had elapsed the can was tipped at a right angle to the table top and the time for the material to flow to the rim of the can was measured. This was done every two minutes until the end of the pot life was reached. The termination of pot life was taken arbitrarily as the time at which 5 seconds was required for the material to flow to the rim of the can after it had been tipped. This method of determining pot life was used in developing Epoxy Adhesive A-100 and A-101. In developing Epoxy Adhesives A-102 and A-103, the pot life determination was modified as follows. Rather than tipping the can and timing flow of the material to the rim, the can was left on the table top and probed every two minutes with a glass rod. The point at which gelled material began to form was taken as the end of the useful pot life.

The finished adhesive formulation must have sufficient gel, or thixotrophy, to keep fillers from settling during storage and to provide a sufficient glue line on vertical surfaces. The test used to determine the gel of the formulation at 75 $\pm$ 2 degrees F. is as follows:

The two components of the epoxy adhesive are mixed together for 5 minutes and then applied to a smooth clean steel plate to form a panel of epoxy material 2 inches wide, 4 inches in length and 0.10 inch (100 mils) in thickness. A removable form of the proper dimensions is used in placing the epoxy on the steel plate. The adhesive is poured into the form and the excess struck off level with the top edge and then the form removed. Immediately after forming the epoxy material, the steel panel is placed in a vertical position, the 4 inch dimension of the epoxy panel perpendicular to the horizontal. Not more than 7 minutes is allowed to elapse between the initiation of mixing and the placing of the steel panel in the vertical position. After the epoxy has cured, the average thickness of material remaining within the original 2 x 4 inch area of the panel is determined.

Of the tests described, those which appeared most indicative of the properties desired and which were, therefore, used in developing and evaluating various adhesive formulations were as follows:

1. Ability to Bond Plastic Portland Cement Mortar to Cured Mortar.
2. Strength Properties of Adhesives in Shear by Tension Loading - Metal to Metal (ASTM D1002-53T). (In this report, this test will be referred to simply as the Adhesive Shear Test and the results will be referred to as the Adhesive Shear Strength.)
3. Cleavage Strength of Metal-to-Metal Adhesives (ASTM D1062-51). (In this report, this test will be referred to simply as the Cleavage Test and the results will be referred to as the Cleavage Strength.)
4. Water Absorption of Plastics (ASTM D570-57T). (The results will be reported as Water Gain, Percent by Wt.)

5. Falling Ball Impact Test. (The results will be reported as Impact Strength, Ft.-Lbs.)
6. Pot Life Test (100 gram batch at 75-80 degrees F.) (The results will be given in minutes.)
7. Determination of Gel (Thixotrophy). (The results will be given as the average thickness in mils of cured material remaining on the test panel.)

In summary, the concrete bonding test would indicate which formulations would be suitable for bonding plastic concrete to existing concrete or to steel. The adhesive shear and cleavage tests would indicate which formulations had possibilities for bonding steel to steel and would also give us information regarding the ultimate strengths of various formulations which we could not determine from tests involving concrete inasmuch as almost all epoxy formulations exhibit higher strengths than concrete. The water absorption test would indicate the completeness of reaction between the epoxy resin and the curing agent. It would be desirable to have as low a water absorption as would be consistent with the other physical properties desired. The impact test was included because it was felt that an epoxy adhesive to be used to bond steel in structural applications should have a fair degree of impact resistance. The pot life test was needed in order to determine whether or not a particular formulation would have a sufficiently long working life.

At this point it would be well to consider the repeatability of the tests described. In Table 6 below, data is presented on the repeatability obtained for the results presented in this report.

Table 6

Repeatability of Tests Performed on Epoxy Adhesive Formulations

Test	Maximum Average Deviation for a Set of Two or More Specimens, Percent	Average of the Average Deviations for All Sets of Specimens, Percent
Adhesive Shear	12.1	3.9
Cleavage	9.7	2.2
Water Gain	16.7	7.4
Impact	14.2	4.4

In a very few instances deviations from average values greater than the maximums shown above were obtained. These results were discarded because of obvious errors in preparation of the adhesive formulation or the test specimens.

DISCUSSION

After deciding on the series of tests described, evaluation of the epoxy materials was begun. The first step ordinarily would be to select the basic epoxy resin which would be most suitable. As already has been stated, since every formulation must consist of at least an epoxy resin and a curing agent, these two materials would have to be considered in combination. From literature reviews and discussion with various manufacturers, it was found that the unmodified type of epoxy resin with 10,000 to 16,000 cps. viscosity was most often recommended for epoxy adhesive formulations to be used with concrete. It was decided to combine this one type of resin with the various curing agents to determine the best curing agents for our purpose. In the various formulations tested, this resin is referred to as high viscosity unmodified resin or simply HVUR. The various curing agents combined with the epoxy resin were tested for their ability to bond plastic concrete to hardened concrete. A minimum of six briquettes were prepared for each combination. Half of the specimens were tested after three days cure and half after seven days cure. The results are shown in Table 7.

Of the curing agents tested, the Lancast A plus DMP-30, and Thiokol LP-3 plus DMP-30 systems performed best for bonding plastic concrete mortar to hardened concrete. These two curing agent systems were chosen for further evaluation. The Versamid 115 and 125 were eliminated from further consideration, not only because of poor performance, but also because of their high viscosity which would make them unsuitable for use in a brushable adhesive.

The results of additional tests on the Lancast A + DMP-30, and the Thiokol LP-3 + DMP-30 systems are shown in Table 8. These results indicated that the LP-3 + DMP-30 system had somewhat better overall physical characteristics. On the basis of these results, we chose to work with the LP-3 + DMP-30 curing agent system.

After choosing a curing system, the epoxy resin was reconsidered. Our initial work was with a high viscosity unmodified resin. Specimens were prepared using a modified epoxy resin along with the polysulfide and DMP-30 curing agent system. The formulation was as follows:

Formulation No.	Composition
8	100 pbw modified epoxy resin 50 pbw Thiokol LP-3 10 pbw DMP-30

The results of the tests performed are shown in Table 9.

Table 7

Ability to Bond Plastic Portland Cement Mortar to Cured Mortar

Formulation No.	Composition	Avg. Strength of Briquettes, PSI 3 Days Cure	7 Days Cure	Type of Failure 3 Days Cure	7 Days Cure
1	100 pbw HVUR + 70 pbw Versamid 115	235	305	100% in Bond	100% in Bond
2	100 pbw HVUR + 50 pbw Versamid 125	315	340	100% in Bond	100% in Bond
3	100 pbw HVUR + 45 pbw Versamid 140	240	340	100% in Bond	100% in Bond
4	100 pbw HVUR + 45 pbw Genamid 250	345	385	50% in Mortar	50% in Mortar
5	100 pbw HVUR + 45 pbw Lancast A + 3 pbw DMP-30	405	410	90% in Mortar	100% in Mortar
6	100 pbw HVUR + 12 pbw Diethylene Triamine	360	525	100% in Bond	50% in Bond
7	100 pbw HVUR + 50 pbw LP-3 + 10 pbw DMP-30	485	460	75% in Mortar	75% in Mortar

Table 8

Physical Properties of Formulations 5 & 7

Test	Formulation No. 5	7
Adhesive Shear Strength, PSI, Avg.	2786	3190
Cleavage Strength, PSI, Avg.	1310	1365
Water Absorption, Percent by Wt., Avg.	0.18	0.19
Impact Strength, Ft.-Lbs., Avg.	5.5	7.0

Table 9

Properties of Formulation 8

Test	Results Obtained
Ability to Bond Plastic Portland Cement Mortar to Cured Mortar	
Avg. Strength of Briquettes, PSI, 3 Days Cure	340
Type of Failure	65% in Mortar
Avg. Strength of Briquettes, PSI, 7 Days Cure	410
Type of Failure	85% in Mortar
Adhesive Shear Strength, PSI, Avg.	3047
Cleavage Strength, PSI, Avg.	1340
Water Gain, Percent by Wt., Avg.	0.26
Impact Resistance, Ft.-Lbs., Avg.	7.5

The above results when compared with the results obtained for an unmodified resin do not indicate any significant difference in the modified and the unmodified resins. It was decided that an unmodified epoxy resin - polysulfide system catalyzed with DMP-30 or a mixture of DMP-30 and DMP-10 would be the basis of our adhesive.

At this point an evaluation of the various brands of unmodified and modified epoxy resins which were available was undertaken to determine if there was any significant difference between equivalent resins produced by different manufacturers.

The formulation used to evaluate the unmodified resins was as follows:

Formulation No.	Composition
9	100 pbw epoxy resin 100 pbw minus 325 mesh tabular aluminum oxide 75 pbw Thiokol LP-3 10 pbw DMP-30

All specimens were cured for seven days at 70-80 degrees F. prior to testing. The results obtained are shown in Table 10.

Table 10

Comparison of High Viscosity Unmodified Epoxy Resins

Test	Results Obtained		
	Ciba 6010	Epotuf 6140	Shell 828
Adhesive Shear Strength, PSI, Avg.	3140	2948	3023
Cleavage Strength, PSI, Avg.	1345	1390	1355
Water Gain, Percent by Weight, Avg.	0.16	0.14	0.15
Impact Resistance, Ft.-Lbs., Avg.	9.0	9.0*	9.0*

*Limit of test equipment without failure occurring.

The above test results do not indicate any significant difference in the three unmodified resins tested.

The formulation used to evaluate the modified resins was as follows:

Formulation No.	Composition
10	100 pbw epoxy resin 100 pbw minus 325 mesh tabular aluminum oxide 60 pbw Thiokol LP-3 8 pbw DMP-30

All specimens were cured for seven days at 70-80 degrees F. prior to testing. The results obtained are shown in Table 11.

No significant difference in the three unmodified resins was indicated by the results obtained.

Further work which needed to be done in developing an adhesive included establishing the ratio of epoxy resin to the polysulfide, and the amount and combination of catalytic curing agents that should be used. We also had yet to consider the fillers and thixotropic or flow control agents which should be used.

Table 11

Comparison of Modified Epoxy Resins

Test	Results Obtained Ciba 506	Epi-Rez 5071	Shell 815
Adhesive Shear Strength, PSI, Avg.	2940	2915	2985
Cleavage Strength, PSI, Avg.	1400	1485	1385
Water Gain, Percent by Wt., Avg.	0.18	0.20	0.14
Impact Resistance, Ft.-Lbs., Avg.	9.0*	9.0*	9.0*

*All specimens withstood limit of test equipment without failure occurring.

With regard to the properties of the finished adhesive, the type and amount of filler to be used is interconnected with the ratio of epoxy resin to polysulfide used in the formulation. In view of this fact, these variables would almost have to be considered simultaneously. It was decided to evaluate first the various fillers available, using one basic resin and curing agent combination.

Fillers

The evaluation of the various fillers was carried out using the following epoxy resin - curing agent formulation:

Formulation No.	Composition
11	100 pbw HVUR 75 pbw Thiokol LP-3 10 pbw DMP-30

In all cases, the filler was mixed with the resin. In order to insure complete wetting of the filler, the resin and filler mixture was heated to 160 degrees F. for about 10 minutes and the hot resin and filler combination stirred until a smooth uniform mix was obtained. After preparing and cooling the resin-filler mixes, the polysulfide and catalyst were added and test specimens prepared from the resulting adhesives. Test results for various fillers and filler loadings are shown in Table 12. All test specimens were cured for 7 days at 70-80 degrees F. prior to testing. The filler loadings shown are parts by weight of filler per 100 parts by weight of epoxy resin.

The amount of loading for each filler is limited in part by how much it will increase the viscosity of the formulation. The mica and asbestos both increased

the viscosity of the formulation quite markedly. The aluminum oxide and silica flour had the least effect on viscosity for a given filler loading. The china clays were intermediate in their effect on the viscosity or workability of the formulation.

Considering the test results obtained along with the workability of the resulting formulation, the minus 325 mesh tabular aluminum oxide performed best. However, this combination was too viscous to meet the requirement of a brushable consistency. We would either have to cut back drastically on filler loading, use a solvent to lower the viscosity, or use a modified rather than an unmodified resin. Since a filler loading of approximately 100 pbw of aluminum oxide per 100 pbw epoxy resin appeared to be most desirable from both the standpoint of economics and strength, it was felt that use of a solvent or a modified resin would be a better solution to the problem. We had already done some experimentation with solvents in adhesive formulations. The results are discussed below.

Solvents

A limited amount of work was done incorporating organic solvents in an epoxy-polysulfide formulation with minus 325 mesh aluminum oxide as a filler, and the same formulation with ASP 400 china clay as the filler. Approximately 5 pbw of toluene, xylene, and methyl ethyl ketone were incorporated individually into each formulation. The test for ability to bond plastic portland cement concrete to cured portland cement concrete was used to evaluate the effect of these solvents.

In the case of the formulation containing aluminum oxide as the filler, it was found that with the toluene and xylene, almost always some solvent would be trapped in the glue line, resulting in an inferior bond. The methyl ethyl ketone could be used without solvent being trapped provided the glue line was not too thick and sufficient open time was allowed. The formulation containing the china clay as the filler performed somewhat differently. It was found that regardless of the type of solvent used or open time allowed, some solvent would remain in the glue line. The china clays have a strong affinity for organic materials (organophyllic). This apparently explains why solvent would always remain in the formulation containing the china clay.

Since solvents would tend to make critical the time of placement of the plastic portland cement concrete against the adhesive film, it was decided that the use of solvents would be unwise.

The alternative to the use of solvents to obtain a more workable formulation was to change to a modified resin in the adhesive formulation. Our original comparison of the unmodified and the modified epoxy resins indicated little difference from the standpoint of strength. In view of the better workability which could be obtained with a modified resin, we chose to switch to this type of resin for our adhesive.

Table 12

Filler Evaluation

Filler Used	Adhesive Shear Strength, PSI, Avg.	Cleavage Strength PSI, Avg.	Water Absorption Percent by Wt., Avg.	Impact Strength Ft.-Lbs., Avg.
No filler	3210	1240	0.22	9.0*
50 pbw aluminum oxide (-325 mesh)	3828	1245	0.20	9.0
100 pbw aluminum oxide (-325 mesh)	4110	1355	0.16	9.0*
25 pbw asbestos 7TF1	3520	1275	0.36	9.0
50 pbw asbestos 7TF1	3492	1145	0.39	8.5
75 pbw ASP 103 china clay	3408	1200	0.26	5.5
100 pbw ASP 103 china clay	3022	1110	0.22	5.0
75 pbw ASP 400 china clay	3150	1395	0.20	7.5
100 pbw ASP 400 china clay	3535	1360	0.16	7.5
25 pbw mica, Alsibronz #12	3162	1020	0.19	5.0
50 pbw mica, Alsibronz #12	2558	710	0.32	8.5
50 pbw silica flour (-325 mesh)	2915	1458	0.18	9.0*
100 pbw silica flour(-325 mesh)	2820	1340	0.13	9.0*
80 pbw aluminum oxide (-325 mesh) + 20 pbw asbestos 7TF1	3666	1295	0.17	8.5
60 pbw aluminum oxide (-325 mesh) + 40 pbw asbestos 7TF1	3346	1205	0.13	9.0
80 pbw Al_2O_3 (-325 mesh) + 20 pbw ASP 103 china clay	3460	1210	0.14	8.5
60 pbw Al_2O_3 + 20 pbw ASP 103 china clay	3500	1250	0.16	8.5

*All specimens withstood limit of test equipment without failure occurring.

At this point, the epoxy resin, the filler, and approximate filler loading for the adhesive formulation had been selected. The next step was to decide on the ratio of polysulfide to epoxy resin which would be the most desirable. A range of polysulfide concentrations from 50 to 75 pbw per 100 pbw epoxy resin was investigated. The following formulation was used to evaluate the polysulfide content.

Formulation No.	Composition
12	100 parts by weight modified epoxy resin 100 parts by weight minus 325 mesh tabular aluminum oxide 7 parts by weight DMP-30

Test results for the various concentrations of polysulfide are shown below:

Table 13

Results of Varying Polysulfide Content in Formulation 12

Test	Polysulfide Content 50 pbw	60 pbw	65 pbw	75 pbw
Adhesive Shear Strength, PSI., Avg.	3047	3300	3297	3100
Cleavage Strength, PSI., Avg.	1340	1362	1280	1170
Water Gain, Percent by Wt., Avg.	0.13	0.15	0.14	0.16
Impact Strength, Ft.-Lbs. at Failure, Avg.	8.5	9.0*	9.0*	9.0*

*Withstood limit of test equipment without failure occurring.

From the results shown above, a polysulfide content of 60 to 65 parts by weight polysulfide per 100 pbw epoxy resin would be the most desirable. We chose to use 60 pbw of polysulfide per 100 pbw epoxy resin in the adhesive formulation.

The curing agents used with the epoxy-polysulfide systems were DMP-10 (Dimethyl amino methyl phenol) and DMP-30 (Tridimethyl amino methyl phenol). In the investigational work with the polysulfide systems up to this point, only the DMP-30, in a ratio of 7 to 10 pbw to 100 pbw of epoxy resin, had been used.

The adhesive formulation at this point was as follows:

Formulation No.	Composition
13	100 pbw modified epoxy resin 100 pbw -325 mesh tabular aluminum oxide 60 pbw Thiokol LP-3 polysulfide

The above formulation was cured with the various combinations and amounts of DMP-10 and DMP-30. Test results obtained on the resulting adhesives are shown in Table 14. All the specimens were cured for seven days at 70-80 degrees F. prior to testing.

Table 14

Curing Agent	Test Results: Adhesive Shear Strength, PSI, Avg.	Test Results: Cleavage Strength, PSI, Avg.
3.5 pbw DMP-10 + 3.5 pbw DMP-30	2636	1270
4.0 pbw DMP-10 + 4.0 pbw DMP-30	2567	1185
5.0 pbw DMP-30	2612	1085
6.0 pbw DMP-30	2852	1238
7.0 pbw DMP-30	3081	1322
7.5 pbw DMP-30	3704	1405
8.0 pbw DMP-30	3667	1410
9.0 pbw DMP-30	3009	1315
10.0 pbw DMP-30	2792	1155

The above results indicate that 7.0 to 8.0 pbw of DMP-30 per 100 pbw modified epoxy resin would develop the optimum strength properties in the cured adhesive. Tentatively, 8 pbw DMP-30 per 100 pbw of resin was chosen as the curing agent for the adhesive formulation. This resulted in the following adhesive formulation:

Formulation No.	Composition
14	100 pbw modified epoxy resin 100 pbw -325 mesh tabular aluminum oxide 60 pbw Thiokol LP-3 polysulfide 8 pbw DMP-30

The pot life, or working time, desired for the adhesive would also have a definite bearing on the type and amount of curing agent used. The adhesive formulation had a pot life of 32 minutes when tested by the procedure described earlier in this report. One of the original requirements for the adhesive was that a one gallon batch have a pot life of at least 30 minutes at 80 degrees. A batch of the adhesive (Formulation No. 14) was prepared in two components, the resin component consisting of the epoxy resin and minus 325 mesh tabular aluminum oxide, and the hardener component consisting of Thiokol LP-3 and DMP-30. Sufficient material was prepared that when the two components were mixed together, the total amount of material would be approximately 10 pounds, or a little less than one gallon. Both components were brought to a temperature of 80 degrees F. and were then mixed by hand for 5 minutes in a gallon container. The material was allowed to react completely in the gallon container. The ambient temperature during the reaction was approximately 80 degrees F. The end of the pot life would be the point at which the material first began to gel or set up. The mass was probed with a glass rod every two minutes beginning 20 minutes after initiation of mixing. The first signs of gel were noted 34 minutes after initiation of mixing. Thus our adhesive formulation had a pot life or working time of approximately 34 minutes at 80 degrees F. for a one gallon batch, and would comply with our initial requirement regarding pot life or working time. Since the reactions involving epoxy resins and curing agents are exothermic (that is, heat is given off by the reaction), and since the reaction rate is speeded up by heating, leaving the material in the gallon bucket resulted in a build-up of heat which shortened the pot life or working time. In actual practice, the material would be spread out soon after mixing which would lengthen the working time somewhat.

On the basis of the strength tests and the pot life determinations, we chose to use 8 parts by weight DMP-30 per 100 parts by weight epoxy resin as the catalyst in the adhesive formulation.

Thixotropic Agents

The final item needed in the adhesive formulation was a thixotropic or gelling agent. The thixotropic agents considered were M-5 Cab-O-Sil, Bentone 27, and Asbestos 7TF1. The Asbestos 7TF1 already had been experimented with as a filler. When used in concentrations of 10 to 20 pbw per 100 pbw epoxy resin, this material will impart good resistance to flow, but makes the formulation difficult to brush out. Also, the asbestos appeared to lower the strengths of the formulation. It was decided to eliminate Asbestos 7TF1 from further consideration as a thixotropic agent. The effectiveness of Bentone 27 and M-5 Cab-O-Sil as thixotropic agents in Adhesive Formulation No. 14 was determined by performing the series of tests shown in Table 15. The thixotropic agent concentrations shown are parts by weight per 100 parts by weight epoxy resin. The Bentone 27 was incorporated into the resin portion of Formulation No. 14. It was pre-wet with 1 pbw 50% ethyl alcohol and 50% water and then stirred into the epoxy resin. The resulting mixture was put through a three-roll paint mill twice to achieve maximum effectiveness from the Bentone 27. For the 6 pbw M-5 Cab-O-Sil, all of this material was

incorporated into the resin component. The Cab-O-Sil was stirred in by hand along with the filler and then the mixture was ground for approximately 5 minutes at 2000 rpm on a Cowles Dissolver. During the last minute of grinding, one pbw water was added to aid in gel formation. For the 8 pbw M-5 Cab-O-Sil, 6 pbw was incorporated into the resin component as stated above and the remaining 2 pbw were incorporated into the hardener component. The hardener component was also ground on a Cowles Dissolver for approximately 3 minutes. No water was added to the hardener component.

Table 15

Physical Properties of Formulation No. 14 with Various Thixotropic Agents

Test	5 pbw Bentone 27	6 pbw M-5	8 pbw M-5 Cab-O-Sil
Thixotrophy, Mils of Cured Material Remaining on Test Panel, Avg.	55	38	45
Adhesive Shear Strength, PSI, Avg.	3100	3020	2975
Cleavage Strength, PSI, Avg.	1300	1205	1225
Water Gain, Percent by Wt., Avg.	0.15	0.14	0.16
Impact Resistance, Ft.-Lbs., Avg.	9.0*	9.0*	9.0*

*Specimens withstood limit of test equipment without failure occurring.

The results of the test performed indicated that the Bentone 27 gave a better gel, with less impairment of the strength properties of the adhesive.

Samples of the adhesive components gelled with Bentone 27 and with M-5 Cab-O-Sil were retained in sealed containers in the laboratory to observe the stability of the gel formed. It was found that the Bentone 27 gels were more stable than the gels formed with the M-5 Cab-O-Sil. However, the gels formed with the M-5 Cab-O-Sil retained sufficient thixotrophy to prevent filler settling and to prevent run-off when the adhesive was applied to vertical surfaces.

In view of the more complicated compounding required to obtain satisfactory gels with Bentone 27, the decision was made to use M-5 Cab-O-Sil as the thixotropic agent in the adhesive formulation. The concentration selected was 8 pbw per 100 pbw of epoxy resin, 6 pbw to be incorporated into the resin component and 2 pbw to be incorporated into the hardener component.

A portion of the Cab-O-Sil was placed in the hardener component so that its viscosity would not differ quite so widely from that of the resin component. It was found that if there is a wide difference in viscosity or consistency of the resin and hardener components, it is extremely difficult to obtain good mixing. There is a tendency for lumping to occur. For optimum mixing, the two components should have the same viscosity or consistency.

The adhesive formulation was now complete and was as follows:

Resin Component:	100 pbw modified epoxy resin
	100 pbw minus 325 mesh tabular aluminum oxide
	6 pbw M-5 Cab-O-Sil
	1 pbw water
Hardener Component:	60 pbw Thiokol LP-3 Polysulfide
	8 pbw DMP-30
	2 pbw M-5 Cab-O-Sil

This formulation was designated as Texas Highway Department Epoxy Adhesive A-100. The mixing ratios for Adhesive A-100 were as follows:

3 parts by weight resin component
1 part by weight hardener component

or 2 parts by volume resin component
1 part by volume hardener component

It was decided that the adhesive should be packaged so that mixing could be done in one of the original containers. One-half gallon of the resin component was packaged in a gallon container and one quart of the hardener component was packaged in a quart container. Because of the corrosiveness of the polysulfide, the hardener component was packaged in cans with corrosion resistant linings. This packaging combination resulted in a 3/4 gallon unit which could be mixed in the resin component container.

One hundred units of Epoxy Adhesive A-100 were purchased on a competitive bid basis in order that the adhesive might be tried in the field. The cost per 3/4 gallon unit, including inspection and testing, was $7.50.

Soon after this material was purchased, the need for a fairly large quantity of epoxy adhesive for bonding new to old concrete on a maintenance project arose. As a result of further work in the laboratory and information received from the field, some slight modifications were made in the Epoxy Adhesive A-100 formulation prior to furnishing it for this job. The amount of M-5 Cab-O-Sil was reduced to 6 pbw per 100 pbw of epoxy resin and glycerol was substituted for water as an aid in gel formation. It was found that glycerol in small quantities (on the order of 0.3 pbw per 100 pbw epoxy resin) was more effective than water as an aid in forming gels with M-5 Cab-O-Sil. The modified adhesive

formulation was as follows:

Resin Component:	100 pbw	modified epoxy resin
	102 pbw	minus 325 mesh tabular aluminum oxide
	4 pbw	M-5 Cab-O-Sil
	0.3 pbw	glycerol
Hardener Component:	60 pbw	Thiokol LP-3 Polysulfide
	8 pbw	DMP-30
	2 pbw	M-5 Cab-O-Sil

This formulation was designated as Epoxy Adhesive A-101. Approximately 250 gallons of this material were purchased on a competitive bid basis at a cost of $9.55 per gallon including inspection and test charges.

Reports on these adhesives from the field indicated that when properly used, these materials generally performed quite well.

Concurrently with our program of developing an epoxy adhesive and binder for highway use, we were also investigating the weathering and aging characteristics of epoxy materials. This program is discussed in Part III of this report. As a result of the information obtained from the weathering and aging tests, it appeared that these epoxy-polysulfide adhesives would not give the long range performance needed. It would be necessary to develop an adhesive with better aging and weathering properties. The epoxy adhesive containing Lancast A as a curing agent had exhibited fairly good performance on initial tests and had exhibited good weathering and aging properties. However, the Lancast A formulations did not have quite as good strength properties as would be desired. Also, it was found that the Lancast A tended to separate and form a sediment on storage. It was thought that this might result in poor storage characteristics of the adhesive.

During the time that the developmental work was being done on the epoxy adhesive, the various chemical companies engaged in producing epoxy resins and curing agents were developing new materials. The epoxy resin manufacturers had developed an unmodified epoxy resin of 7,000 to 10,000 cps. viscosity, compared with the 10,000 to 16,000 cps. viscosity unmodified resins with which we originally had worked. A typical resin of this type which was obtained for experimental use was Epi-Rez 509 manufactured by Jones-Dabney. Two new curing agents also were obtained. These were Epi-Cure 872, manufactured by Jones-Dabney, and DP-134, manufactured by Ciba.

In the process of developing a more satisfactory adhesive, these new materials were evaluated. In order to compare the Epi-Rez 509 with the other unmodified resins tested, Formulation No. 9 was used. (See page 19)

Test specimens prepared with Epi-Rez 509 as the epoxy resin in this formulation were cured for 7 days at 70-80 degrees F. Results of the tests performed are shown in Table 17.

Table 17

Results of Tests on Low Viscosity Unmodified Epoxy Resin

Test	Results
Adhesive Shear Strength, PSI, Average	3045
Cleavage Strength, PSI, Average	1380
Water Gain, Percent by Wt., Average	0.14
Impact Resistance, Ft.-Lbs., Average	9.0

The above results are essentially the same as were obtained for the higher viscosity unmodified resins previously tested and thus this type of resin could be considered equivalent to the other unmodified resins with regard to physical properties of adhesives into which these materials might be incorporated.

The Ciba DP-134 and Jones-Dabney Epi-Cure 872 curing agents were combined with epoxy resin in the ratios recommended by the manufacturers and tested for their ability to bond plastic concrete to hardened concrete using the briquette test. The results obtained are shown in Table 18.

Table 18

Ability to Bond Plastic Portland Cement Mortar to Cured Mortar

Formulation No.	Composition	Average Strength of Briquettes, PSI		Type of Failure	
		3 Days Cure	7 Days Cure	3 Days Cure	7 Days Cure
15	100 pbw regular unmodified epoxy resin + 100 pbw DP-134	200	250	100% in Bond	100% in Bond
16	100 pbw regular unmodified epoxy resin + 35 pbw Epi-Cure 872	430	475	100% in Mortar	100% in Mortar

The results indicated that the DP-134 would be unsuitable in an adhesive which would be used mainly to bond plastic portland cement concrete to hardened concrete. The Epi-Cure 872 showed considerable promise for use in a concrete adhesive and was evaluated further. Typical results of additional tests on Formulation 16 are shown in Table 19.

Table 19

Physical Properties of Formulation 16

Test	Results Obtained
Adhesive Shear Strength, PSI	2900
Cleavage Strength, PSI	1540
Water Gain, Percent by Wt.	0.10
Impact Resistance, Ft.-Lbs.	5.5

The Epi-Cure 872 formulation exhibited good physical properties with the exception of the impact strength, which was not as good as would be desired. It was decided that several adhesive formulations would be prepared using the Epi-Cure 872 and blends of Epi-Cure 872 with Versamid 140, the polyamide which prior tests had indicated to be the most suitable for use in a concrete adhesive. A formulation containing a blend of Versamid 140 and Epi-Cure 855, which is one of the components of Epi-Cure 872, also was tested. The information regarding fillers and thixotropic agents, obtained in the original developmental work on concrete adhesives, was used in preparing these formulations. The composition of the formulations prepared are as follows:
(The abbreviation LVUR is used to indicate Low Viscosity Unmodified Epoxy Resin)

Formulation 17

Resin Component:
- 100 pbw LVUR
- 63 pbw minus 325 mesh tabular alumina
- 3 pbw M-5 Cab-O-Sil
- 0.2 pbw glycerol

Hardener Component:
- 35 pbw Epi-Cure 872
- 18 pbw minus 325 mesh tabular alumina
- 3 pbw M-5 Cab-O-Sil
- 0.1 pbw glycerol

Mixing Ratios:
- 3 pbw resin component
- 1 pbw hardener component

or

- 5 parts by volume resin component
- 2 parts by volume hardener component

Formulation 18

Resin Component:
- 100 pbw LVUR
- 62 pbw minus 325 mesh silica flour
- 4 pbw M-5 Cab-O-Sil
- 0.2 pbw glycerol

Hardener Component:
- 35 pbw Epi-Cure 872
- 19 pbw minus 325 mesh silica flour
- 2 pbw M-5 Cab-O-Sil
- 0.1 pbw glycerol

Mixing Ratios:
- 3 pbw resin component
- 1 pbw hardener component

or

- 5 parts by volume resin component
- 2 parts by volume hardener

Formulation 19

Resin Component:
- 50 pbw LVUR
- 50 pbw modified epoxy resin
- 90 pbw minus 325 mesh tabular alumina
- 4 pbw M-5 Cab-O-Sil
- 0.3 pbw glycerol

Hardener Component:
- 60 pbw Versamid 140
- 20 pbw Epi-Cure 872
- 15 pbw minus 325 mesh tabular alumina
- 2 pbw M-5 Cab-O-Sil
- 0.1 pbw glycerol

Mixing Ratios:
- 2 pbw resin component
- 1 pbw hardener component

or

- 10 parts by volume resin component
- 7 parts by volume hardener component

Formulation 20	Formulation 21	Formulation 22
Resin Component:	Resin Component:	Resin Component:
100 pbw LVUR	100 pbw LVUR	100 pbw LVUR
90 pbw minus 325 mesh tabular alumina	79 pbw minus 325 mesh tabular alumina	18 pbw minus 325 mesh tabular alumina
4 pbw M-5 Cab-O-Sil	4 pbw M-5 Cab-O-Sil	2 pbw M-5 Cab-O-Sil
0.3 pbw glycerol	0.2 pbw glycerol	0.2 pbw glycerol
Hardener Component:	Hardener Component:	Hardener Component:
60 pbw Versamid 140	30 pbw Epi-Cure 855	40 pbw Versamid 140
20 pbw Epi-Cure 872	20 pbw Versamid 140	25 pbw Epi-Cure 872
15 pbw minus 325 mesh tabular alumina	9 pbw minus 325 mesh tabular alumina	56 pbw minus 325 mesh tabular alumina
2 pbw M-5 Cab-O-Sil	2 pbw M-5 Cab-O-Sil	3 pbw M-5 Cab-O-Sil
0.1 pbw glycerol	0.1 pbw glycerol	0.2 pbw glycerol
Mixing Ratios:	Mixing Ratios:	Mixing Ratios:
2 pbw resin component	3 pbw resin component	1 part by weight or volume resin component
1 pbw hardener component	1 pbw hardener component	1 part by weight or volume hardener component
or	or	
3 parts by volume resin component	9 parts by volume resin component	
2 parts by volume hardener component	2 parts by volume hardener component	

In these six formulations, the total amount of filler was held close to 100 pbw per 100 pbw epoxy resin. The amount of filler present in each component was adjusted to control the consistency and to provide a convenient mixing ratio. In cases where less than one complete unit of adhesive is to be used in the field, there is usually no way to measure out the proper amount of each component by weight, so it is important that the adhesive have a simple mixing ratio by volume.

Formulations 17 and 18 are essentially the same except that minus 325 mesh tabular alumina was used as the filler in Formulation 17 and minus 325 mesh silica flour was used as the filler in Formulation 18. This was done to compare the performance of these two fillers in a formulation with a modified amino-amide as the curing agent.

When the polyamides and modified amines or modified amino-amides (see Table 2, page 4), are used as curing agents in a formulation for bonding plastic portland cement concrete to hardened portland cement concrete, the manufacturers of these curing agents do not recommend the use of a modified epoxy resin of the type used in Epoxy Adhesives A-100 and A-101. This means that in this type of formulation a regular unmodified or a low viscosity unmodified epoxy resin would have to be used. In order to obtain a brushable formulation the low viscosity unmodified epoxy resin would be the logical choice. However, the low viscosity unmodified epoxy resins still give a formulation which is more viscous than would be desired. In Formulation 19, a 50-50 blend of a modified

epoxy resin and a low viscosity unmodified resin was tried. All of the other formulations were prepared using a low viscosity unmodified resin.

These six formulations were tested for their ability to bond plastic portland cement concrete to hardened concrete using the briquette test. The results are given below:

Table 20

Ability to Bond Plastic Portland Cement Mortar to Cured Mortar

Formulation No.	Average Strength of Briquettes, PSI			Type of Failure		
	1 Day Cure	3 Days Cure	7 Days Cure	1 Day Cure	3 Days Cure	7 Days Cure
17	395	435	480	100% in Mortar	100% in Mortar	100% in Mortar
18	360	435	510	100% in Mortar	100% in Mortar	100% in Mortar
19	315	305	295	100% in Bond	75% in Bond	90% in Bond
20	305	405	395	100% in Bond	75% in Mortar	75% in Mortar
21	95	170	250	100% in Bond	100% in Bond	100% in Bond
22	390	455	500	100% in Mortar	100% in Mortar	100% in Mortar

The above results indicate that Formulations 17, 18 and 22 are the most satisfactory for bonding plastic portland cement concrete to hardened concrete. Additional tests were performed on all six of these formulations to determine their physical properties. The results of these tests are shown below. All these data were obtained by testing specimens cured for seven days at 70-80 degrees F.

Table 21

Physical Properties of Formulations 17 through 22

Test	Formulation 17	18	19	20	21	22
Adhesive Shear Strength, PSI, Avg.	2768	2536	3474	3002	3183	3063
Cleavage Strength, PSI, Avg.	1140	1220	1710	1732	1660	1525
Water Gain, Percent by Wt., Avg.	0.04	0.07	0.12	0.15	0.09	0.08
Impact Resistance, Ft.-Lbs., Avg.	5.0	5.0	8.5	9.0	5.0	8.5

Comparing the results obtained for Formulations 17 and 18 it does not appear that there is a great deal of difference between the tabular alumina and silica flour as fillers. There is enough difference in the average adhesive shear strengths to indicate a slight advantage for the tabular alumina. Considering all the tests performed, Formulation 22 appeared to be the best. This formulation was subjected to further tests with results as follows:

Pot Life of One gallon
Batch at 80 degrees F. - - - Approximately 45 minutes

Thixotrophy
(Mils of cured material
remaining on test panel) - - 85

On the basis of all the tests performed, Formulation 22 was chosen to replace Epoxy Adhesive A-101, and was designated as Epoxy Adhesive A-102.

The adhesive again was packaged so that mixing could be done in one of the original containers. Three pints each of the resin and hardener components were packaged in gallon containers. Since the resin component was the least viscous of the two components, it was to be added to the hardener component and mixing done in the hardener component container.

Four hundred and twenty-five units of Epoxy Adhesive A-102 were purchased on a competitive bid basis and placed in the regional warehouses for maintenance use. The cost per 3/4 gallon unit, including inspection and testing, was $6.75.

Reports from the field on this material indicated that it was performing satisfactorily. The main problem with this material, as far as the field personnel were concerned, was its handling properties. Epoxy Adhesive A-102 was a little too viscous for good application by brushing. The fact that most of this material was applied to rough concrete surfaces aggravated the problem. In most cases, thicker glue lines were being obtained than the 20 to 40 mils which was desirable, resulting in a waste of expensive material. Also, it was found that on storage, some of the resin component was losing most of its gel, resulting in filler settling. Additional work in the laboratory indicated that it is difficult to obtain dependable gels on an unmodified resin with M-5 Cab-O-Sil. This seems to be especially true if the filler content is low. The degree of gelation would vary between batches, and a few of them would turn out to be unstable. The hardener component exhibited good gel retention during storage. It was found that the polyamide-modified amine curing agents could be gelled with M-5 Cab-O-Sil without using any glycerol or other material as an aid in gel formation. These gels are quite stable on storage.

Additional work was done in order to obtain a material with better handling and storage properties. Another new curing agent recommended for use in concrete adhesives had been developed by General Mills. This material is designated as Genamid 2000. It is an amino-amide type curing agent. The Genamid 2000 was combined with low viscosity unmodified epoxy resin in the

ratio recommended by the manufacturer, and tested for ability to bond plastic portland cement concrete to hardened concrete using the briquette test. The results obtained are shown below.

Table 22

Ability to Bond Plastic Portland Cement Mortar to Cured Mortar

Formulation	Composition	Average Strength of Briquettes, PSI 1 Day Cure	7 Days Cure	Type of Failure 1 Day Cure	7 Days Cure
23	100 pbw LVUR + 45 pbw Genamid 2000	330	460	100% in Mortar	75% in Mortar

The above results indicated that Genamid 2000 had possibilities as a curing agent for a concrete adhesive. However, the viscosity of Genamid 2000 was on the order of 1900 cps. at 80 degrees F., and it was felt that an even lower viscosity would be necessary to produce an adhesive with good application characteristics. Blends of Genamid 2000 with the lower viscosity curing agents such as Epi-Cure 872 and Epi-Cure 855 were tried in adhesive formulations. Some of the polyamides and modified amino-amide curing agents which already had been considered were mixed in new combinations and tried in adhesive formulations.

Two other curing agents which had not been used previously were used in small amounts in some of these formulations to speed the reaction. These materials were Epi-Cure 87 and Epi-Cure 874, both modified amines manufactured by Jones-Dabney.

Information concerning the actual uses to which the adhesive was being put in the field revealed that almost all of the material was being used to bond concrete to concrete and very little was being used to bond metal to concrete or metal to metal. Therefore, it was decided that the adhesive could have somewhat lower strength properties, especially impact resistance, without detracting from its performance. In view of this, minus 325 mesh silica flour again was considered as a filler. The comparatively high cost of the minus 325 mesh tabular alumina did not justify the small increases in strength properties which could be obtained by using it, especially in view of the field use pattern that was being set for the adhesive.

The formulations which were prepared and tested are shown on the following pages.

In almost all of these formulations, somewhat more than the stoichiometric amount of curing agent was used. This is one way of obtaining a more flexible, impact resistant formulation and this was the reason an excess of some of the curing agents was used.

In all of these formulations with the exception of 24, the filler content was balanced between the resin and hardener components to give a mixing ratio of one part resin component to one part hardener component by volume. For Formulation 24, the mixing ratio was 2 parts resin component to 3 parts hardener component by volume.

In these various formulations, in addition to comparing the various curing agents and combinations thereof, minus 325 mesh tabular alumina is compared with minus 325 mesh silica flour as a filler, and various filler loadings are considered. In some of these formulations, glycerol was used as an aid in gel formation and in others it was eliminated.

Formulation 24

Resin Component:
- 100 pbw LVUR
- 18 pbw minus 325 mesh alumina
- 2 pbw M-5 Cab-O-Sil

Hardener Component:
- 50 pbw Epi-Cure 855
- 40 pbw Versamid 125
- 10 pbw Epi-Cure 87
- 78 pbw minus 325 mesh alumina
- 2 pbw M-5 Cab-O-Sil

Formulation 25

Resin Component:
- 100 pbw LVUR
- 20 pbw minus 325 mesh alumina
- 2 pbw M-5 Cab-O-Sil

Hardener Component:
- 35 pbw Epi-Cure 872
- 25 pbw Epi-Cure 855
- 75 pbw minus 325 mesh alumina
- 2 pbw M-5 Cab-O-Sil
- 0.2 pbw glycerol

Formulation 26

Resin Component:
- 100 pbw LVUR
- 15 pbw minus 325 mesh alumina
- 2 pbw M-5 Cab-O-Sil
- 0.1 pbw glycerol

Hardener Component:
- 40 pbw Epi-Cure 872
- 20 pbw Versamid 140
- 67 pbw minus 325 mesh alumina
- 3 pbw M-5 Cab-O-Sil
- 0.2 pbw glycerol

Formulation 27

Resin Component:
- 100 pbw LVUR
- 30 pbw minus 325 mesh alumina
- 2 pbw M-5 Cab-O-Sil
- 0.1 pbw glycerol

Hardener Component:
- 40 pbw Epi-Cure 872
- 20 pbw Versamid 140
- 82 pbw minus 325 mesh alumina
- 3 pbw M-5 Cab-O-Sil
- 0.2 pbw glycerol

Formulation 28

Resin Component:
- 100 pbw LVUR
- 15 pbw minus 325 mesh alumina
- 2 pbw M-5 Cab-O-Sil
- 0.1 pbw glycerol

Hardener Component:
- 30 pbw Epi-Cure 872
- 30 pbw Versamid 140
- 67 pbw minus 325 mesh alumina
- 3 pbw M-5 Cab-O-Sil
- 0.2 pbw glycerol

Formulation 29

Resin Component:
- 100 pbw LVUR
- 30 pbw minus 325 mesh alumina
- 2 pbw M-5 Cab-O-Sil
- 0.1 pbw glycerol

Hardener Component:
- 30 pbw Epi-Cure 872
- 30 pbw Versamid 140
- 82 pbw minus 325 mesh alumina
- 3 pbw M-5 Cab-O-Sil
- 0.2 pbw glycerol

Formulation 30

Resin Component:
100 pbw LVUR

Hardener Component:
20 pbw Epi-Cure 872
20 pbw Epi-Cure 855
53 pbw minus 325 mesh alumina
2 pbw M-5 Cab-O-Sil
0.2 pbw glycerol

Formulation 31

Resin Component:
100 pbw LVUR

Hardener Component:
40 pbw Genamid 2000
15 pbw Epi-Cure 855
60 pbw minus 325 mesh alumina
4 pbw M-5 Cab-O-Sil

Formulation 32

Resin Component:
100 pbw LVUR

Hardener Component:
40 pbw Genamid 2000
15 pbw Epi-Cure 855
52 pbw minus 325 mesh silica
3 pbw M-5 Cab-O-Sil
0.15 pbw glycerol

Formulation 33

Resin Component:
100 pbw LVUR

Hardener Component:
35 pbw Epi-Cure 872
20 pbw Epi-Cure 855
52 pbw minus 325 mesh silica
3 pbw M-5 Cab-O-Sil
0.15 pbw glycerol

Formulation 34

Resin Component:
100 pbw LVUR

Hardener Component:
35 pbw Epi-Cure 872
20 pbw Epi-Cure 855
58 pbw minus 325 mesh alumina
4 pbw Cab-O-Sil
0.15 pbw glycerol

Formulation 35

Resin Component:
100 pbw LVUR

Hardener Component:
35 pbw Epi-Cure 872
20 pbw Versamid 140
52 pbw minus 325 mesh silica
4 pbw M-5 Cab-O-Sil
0.1 pbw glycerol

Formulation 36

Resin Component:
100 pbw LVUR

Hardener Component:
30 pbw Epi-Cure 855
15 pbw Versamid 140
10 pbw Epi-Cure 874
52 pbw minus 325 mesh silica
3 pbw M-5 Cab-O-Sil

Formulation 37

Resin Component:
100 pbw LVUR

Hardener Component:
37 pbw Genamid 2000
18 pbw Epi-Cure 855
51 pbw minus 325 mesh silica
4 pbw M-5 Cab-O-Sil

Formulation 38

Resin Component:
100 pbw LVUR
20 pbw minus 325 mesh silica
3 pbw M-5 Cab-O-Sil

Hardener Component:
37 pbw Genamid 2000
18 pbw Epi-Cure 855
75 pbw minus 325 mesh silica
3 pbw M-5 Cab-O-Sil

All fifteen of these formulations were tested to determine their ability to bond plastic portland cement concrete to hardened concrete using the briquette test. The results obtained are shown in Table 23.

Table 23

Ability to Bond Plastic Portland Cement Mortar to Cured Mortar

Formulation No.	Average Strength of Briquettes, PSI		Type of Failure	
	1 Day Cure	7 Days Cure	1 Day Cure	7 Days Cure
24	300	340	100% in Bond	100% in Bond
25	295	365	50% in Mortar	50% in Mortar
26	160	255	100% in Bond	100% in Bond
27	200	220	75% in Bond	100% in Bond
28	135	225	100% in Bond	75% in Bond
29	110	90	100% in Bond	100% in Bond
30	295	370	50% in Mortar	50% in Mortar
31	355	490	100% in Mortar	100% in Mortar
32	365	550	100% in Mortar	100% in Mortar
33	360	480	100% in Mortar	75% in Mortar
34	430	440	100% in Mortar	75% in Mortar
35	240	400	100% in Bond	75% in Mortar
36	370	480	100% in Mortar	100% in Mortar
37	350	450	100% in Mortar	100% in Mortar
38	375	460	75% in Mortar	85% in Mortar

Based on the results of the briquette tests, Formulations 30 through 38 were chosen for further consideration. Results of additional tests on these formulations are shown on the following pages. Based on the results of the physical tests performed on Formulations 30 through 38, Formulations 31 and 32 would appear to be the best adhesives. The difference in these two formulations was the filler, minus 325 mesh tabular alumina being used in 31 and minus 325 mesh silica flour being used in 32. Because of the lower cost of the formulation containing the silica flour, it would be the more desirable of the two. Formulation 32 had one disadvantage in that it did not have quite as long a pot life as would be desirable. In Formulation 37, the ratio of Genamid 2000 to Epi-Cure 855 was changed slightly to give a more workable material with a

longer pot life. Also, in Formulation 37, the glycerol was eliminated because, as mentioned earlier, it was found that stable gels could be obtained on modified amine-polyamide combinations using M-5 Cab-O-Sil without any glycerol or other material to aid in gel formation. In both Formulation 32 and 37, the possibility of obtaining unstable gels with M-5 Cab-O-Sil in the resin component was eliminated by not incorporating any filler in this component. The resin component would be prepared simply by repackaging pure epoxy resin. Another advantage to this, aside from eliminating the gel problem, was that it would eliminate a mixing operation for the formulator and thus should reduce the cost of manufacture. In Formulations 32 and 37, the amount of filler used was approximately 50 pbw per 100 pbw of epoxy resin. This was less than had been established as an optimum amount in earlier work. However, it was felt that the better handling characteristics of these formulations would offset the disadvantage of the lower filler content.

Table 24

Physical Properties of Formulations 30 through 38

Test	Formulation No. 30	31	32	33	34	35	36	37	38
Adhesive Shear Strength, PSI, Avg.	3272	3259	3286	2906	2872	2172	2874	2976	2749
Cleavage Strength, PSI, Avg.	1240	1585	1530	1672	1602	1572	1565	1542	1444
Water Gain, Percent by Wt., Avg.	0.14	0.09	0.09	0.06	0.07	0.10	0.18	0.06	0.08
Impact Resistance, Ft.-Lbs., Avg.	5.0	7.1	7.0	5.6	5.8	5.5	5.2	6.0	5.8
Pot Life of 100 Gm. Batch @ 75 Degrees F., Minutes	84	56	48	60	78	58	62	58	66
Consistency or Workability at 75-80 Degrees F.	Fair	Good	Good	Good	Good	Fair	Good	Good	Fair

Formulation 37 did not evidence quite as good physical properties as 32, but it was felt that the overall properties of Formulation 37 were better. One of the requirements for a good concrete adhesive was that it not be critical with regard to time of application of the plastic concrete. Additional briquette specimens were prepared using Formulation 37 to bond plastic portland cement to hardened concrete. Instead of waiting until the epoxy became tacky before applying the fresh mortar, it was molded against the epoxy-coated face

of the cured briquette halves 30 minutes after initiation of mixing of the adhesive components. The ambient temperature and the temperature of the materials was 75 degrees F. (In the previous test to determine ability to bond plastic concrete to hardened concrete, this formulation required approximately one hour at 75 degrees F. to become tacky.) The resulting briquettes were cured in the usual manner and then tested with results as shown in Table 25.

Table 25

Ability to Bond Plastic Portland Cement Mortar to Cured Mortar

Average Strength of Briquettes, PSI			Type of Failure		
7 Days Cure	14 Days Cure	42 Days Cure	7 Days Cure	14 Days Cure	42 Days Cure
260	290	570	100% in Bond	100% in Bond	75% in Mortar 25% in Bond

These results show that if the fresh concrete should be placed prior to the optimum time, the reaction between the resin and hardener component will be slowed, and the adhesive will not set nearly as fast. However, a satisfactory cure and bond eventually will be attained.

Formulation 37 was chosen to replace Epoxy Adhesive A-102 and was designated as Epoxy Adhesive A-103. This is the adhesive currently being used by the Texas Highway Department in maintenance work. This material is being packaged in 3/4 gallon and 1½ gallon units. The 3/4 gallon unit consists of 3 pints of resin in a ½-gallon container and 3 pints of the hardener in a gallon container. The 1½ gallon unit consists of 3 quarts of resin in a gallon container and 3 quarts of hardener in a 2-gallon container. Both of these units are designed so that the resin component can be added to the hardener component and mixing accomplished in the hardener component container.

Approximately 3000 gallons of Epoxy Adhesive A-103 have been purchased to date by the Texas Highway Department at an average cost of approximately $9.00 per gallon, including inspection and testing expenses. Reports from the field indicate that this material is performing satisfactorily.

In the appendix to this report is a copy of the specification which currently is being used to purchase this material on a competitive bid basis. Also included is a copy of the use instruction pamphlet which is issued to the personnel actually engaged in the use of this adhesive.

The Materials and Tests Division is attempting to keep in close touch with the maintenance forces who are engaged in the use of this material. Changes in the current formulation may be indicated at some time in the future as a result of suggestions by the field personnel, based on their experience and needs. It is also possible that new developments in the field of epoxy adhesives will enable us to improve the current adhesive formulation.

PART II

DEVELOPMENT OF AN EPOXY BINDER

Development of Epoxy Materials for Highway Use

Part II

SCOPE

The purpose of this project was to develop an epoxy binder specifically for highway use. The binder was to be designed so that it could be mixed with selected aggregates to form a quick-setting, high strength, epoxy mortar or concrete which would develop a good bond with concrete or steel, and could be used mainly for:

1. Filling cracks and repairing pot holes or spalled areas on concrete roadways or structures.
2. Grouting steel dowel bars or bolts into place on concrete structures.

OBJECTIVES

The objective of this project was to develop from the basic raw materials an epoxy binder which would perform well in highway applications. The following specific requirements were placed on the proposed epoxy binder:

1. It must be capable of developing a good bond with concrete and steel.
2. The epoxy concrete or mortar resulting from mixing the binder with aggregates must be usable at temperatures of 60 to 105 degrees F.
3. The epoxy concrete or mortar resulting from mixing the binder with aggregates must set rapidly enough at 80 degrees F. that patches of the material will withstand traffic loads in 4 to 8 hours.
4. An epoxy binder should be of such a consistency that an epoxy mortar or concrete can be prepared and applied without difficulty.
5. The hardened epoxy concrete or mortar should be of such a nature that temperature changes will not develop detrimental stresses in the epoxy patch or the surrounding concrete.

CONCLUSIONS

The original developmental work on epoxy binders resulted in an unmodified epoxy-polysulfide binder for highway use. It was designated as Texas Highway Department Epoxy Binder B-100 and was used in limited quantities on an experimental basis.

As a result of weathering and aging tests which were performed on the epoxy materials, it appeared that the epoxy-modified amino-amide combinations would be more suitable for use as a binder than the epoxy-polysulfide systems. Also, it was difficult to mix the desired amount of aggregate with Epoxy Binder B-100. The resulting mortars did not handle as well as would be desired.

Additional investigation resulted in an epoxy binder utilizing a low viscosity unmodified epoxy resin and, as the curing agent, a modified amino-amide. This

formulation was designated as Epoxy Binder B-101 and 426 3/4 gallon units of this material were purchased for maintenance use. Field experience with Epoxy Binder B-101 indicated satisfactory performance with the possible exception that the mortars produced with this binder tended to set a little too rapidly in hot weather. Epoxy Binder B-101 was modified slightly to provide a little longer working time. The resulting formulation was designated as Epoxy Binder B-102 and is currently being used in maintenance work. Approximately 4100 gallons of this material have been purchased to date at an average cost of approximately $9.50 per gallon, including inspection and testing expenses. Thus far, this formulation appears to be performing quite satisfactorily in the field.

The following conclusions concerning an epoxy binder formulation specifically for highway use were reached:

1. The best epoxy resin among those tested is a low viscosity unmodified type.
2. Of the curing agents tested, a modified amino-amide or modified amino-amide-polyamide combination performs best.
3. If pigments, specifically titanium dioxide and carbon black, are used in epoxy-modified amino-amide systems, it is best to limit the amount to approximately 4 to 6 parts by weight per 100 parts by weight of epoxy resin. Larger amounts tend to lower the impact strength of the epoxy binder.

In addition to the development of a binder material, some work was done on the aggregates used with the epoxy binder to form a mortar. The following conclusions were reached regarding aggregates.

1. Rounded aggregates will generally result in a more workable, higher strength mortar than can be obtained with sharp, angular aggregates.
2. Aggregates from which all material finer than 50 mesh (U.S. Standard Screen) has been removed perform better than aggregates containing fine material.

MATERIALS

The specific materials obtained for possible use in an epoxy binder formulation are listed in the following paragraphs. Many of them, particularly the epoxy resins and curing agents, were originally considered for use in the adhesive, and their characteristics were discussed in Part I of this report.

Epoxy Resins

The epoxy resins considered were of the regular unmodified or low viscosity unmodified types. All of the resins considered were reaction products of bisphenol A and epichlorohydrin. The modified resins were not considered for several reasons. First of all, the modified resins react more slowly with the curing agents. Thus, if a modified resin were used, it should be

more difficult to produce a mortar with a sufficiently rapid set. Secondly, the modified resins result in formulations which tend to shrink somewhat on curing. There is almost no shrinkage problem with the unmodified resins. Thirdly, the modified resins generally will produce a formulation with a higher thermal coefficient, which is undesirable. The resins considered are shown below:

Table 1

Identification of Epoxy Resins

Resin Designation	Manufacturer	General Type
Araldite 6010	CIBA Chemical Co.	Regular Unmodified (10,000 - 16,000 cps. viscosity at 25 degrees C.)
Epon 828	Shell Chemical Co.	Regular Unmodified (10,000 - 16,000 cps. viscosity at 25 degrees C.)
Epotuf 6140	Reichhold Chemical Co.	Regular Unmodified (10,000 - 16,000 cps. viscosity at 25 degrees C.)
*Epi-Rez 509	Jones-Dabney Co.	Low Viscosity Unmodified (7,000 - 10,000 cps. viscosity at 25 degrees C.)

*This resin became available after initial evaluation of materials.

Of the above resins, the Araldite 6010, Epon 828, and Epotuf 6140 are equivalent materials and were used interchangeably throughout the work on the binder.

Curing Agents

The curing agents obtained for possible use in an epoxy binder are shown in Table 2.

Pigments

After choosing the resin and curing agent system for the epoxy binder, it was decided to add a small amount of pigment so that the epoxy mortar or concrete would more closely resemble portland cement concrete. The pigments which were combined to obtain a light gray color are shown in Table 3.

Thixotropic Agents

In the development of an epoxy adhesive, M-5 Cab-O-Sil colloidal silica, manufactured by Godfrey L. Cabot, Inc., was determined to be the best thixotropic agent for our use. This was the thixotropic agent considered for possible use in the binder formulation.

Table 2

Identification of Curing Agents

Curing Agent	Manufacturer	Type	Parts per 100 Parts of Epoxy Resin by Weight Generally Used
Epi-Cure 87	Jones-Dabney Co.	Modified Amine	20
Genamid 250	General Mills	Modified Amine	40 to 50
Lancast A	CIBA Chemical Co.	Modified Amine	40 to 50 plus 2 to 3 DMP-30
Epi-Cure 855	Jones-Dabney Co.	Modified Amino-Amide	40 to 60
*Epi-Cure 858	Jones-Dabney Co.	Modified Amino-Amide	50 to 200
**Epi-Cure 872	Jones-Dabney Co.	Modified Amino-Amide	35
*Genamid 2000	General Mills	Modified Amino-Amide	40 to 50
Versamid 140	General Mills	Polyamide	40 to 60
Thiokol LP-3	Thiokol Chemical Co.	Polysulfide	50 to 100 plus
DMP-30 (Tridimethyl Amino Methyl Phenol)	Rohm and Haas	Aromatic Amine	See Above

*These curing agents became available after initial evaluation of materials.
**The manufacturer has indicated that Epi-Cure 872 is actually a blend of Epi-Cure 87 and Epi-Cure 855.

Table 3

Identification of Pigments

Pigment	Manufacturer
R-610 Titanium Dioxide	DuPont Chemical Co.
*R-900 Titanium Dioxide	DuPont Chemical Co.
Excelsior Carbon Black	Columbian Carbon Co.

*This pigment became available after initial evaluation of materials.

PROCEDURE

An epoxy binder will consist basically of the epoxy resin and a curing agent. In some cases these are the only constituents present. Pigments may be added to produce a color similar to portland cement concrete. A thixotropic or gelling agent may be added to provide flow control so that mortars made up with the binder can be used on vertical or sloping surfaces. In some cases, a filler may be added to help fill the voids in the finished epoxy mortar or concrete.

In the development of an epoxy binder, we concentrated mainly on selection of the epoxy resin and the curing agent. Much of the information derived from the original experimental work on an epoxy adhesive for highway applications was utilized. Some of the same tests used in the adhesive development were used to evaluate experimental binder formulations. In order to determine the basic physical properties of epoxy resin-curing agent combinations without aggregate added, the previously described Adhesive Shear Strength, Cleavage Strength, Water Absorption, and Impact Tests were utilized. The Water Absorption test was performed on the mortar formed from mixing aggregate with the experimental binder as well as on the binder itself.

In order to determine the capabilities of bonding to concrete, another variation of ASTM C190-59 (Tensile Strength of Hydraulic Cement Mortars) was devised. Halves of broken briquettes, just as were used for testing Ability to Bond Plastic Portland Cement Mortar to Cured Mortar, were employed. The two components of the epoxy binder to be tested were mixed together and the broken surfaces of the dry, cured briquette halves were coated with the binder. The coated briquette halves were placed singly in briquette molds. Standard Ottawa sand as described in ASTM C190-59 then was mixed with the remaining binder in a ratio of 5 pbw sand to 1 pbw epoxy binder to form a mortar which was molded against the cement mortar briquette half to form a composite briquette. The resulting briquettes then were cured for one day in air at 70-80 degrees F., prior to testing.

The ease of mixing binder with aggregate and the workability of the resulting mixture were important considerations. In order to compare ease of mixing and workability of two or more binders, the same type and loading of aggregate must be used. For comparison purposes in the laboratory, the binders under test were mixed with standard Ottawa sand (20-30 mesh) in a ratio of 5 pbw sand to 1 pbw epoxy binder. The initial temperature of the materials and the ambient temperature was 75-80 degrees F. The ease of mixing and the workability were judged as good, fair, or poor. The approximate pot life at 75-80 degrees F. or time that the mortar was considered usable, was determined on this same mix. The pot life was considered to be the total time, taken from initiation of mixing of the two binder components together, until the mortar became too stiff to be usable.

One of the main requirements for an epoxy binder was that mortar or concrete made up with the binder should set rapidly enough at 80 degrees F. to support traffic loads within 4 to 8 hours. Those mortars which definitely were too slow in attaining an initial set were eliminated by observing the mix prepared

to determine ease of mixing and workability and pot life. The total amount of material prepared for this test was 500 grams contained in a #2 can. This mix was allowed to harden in the can and was probed at intervals with a steel rod to determine the point at which the mortar had attained an initial set. Those mixes which appeared to have attained an initial set within six hours were subjected to a more quantitative test. A mortar consisting of 5 pbw standard Ottawa sand (20-30 mesh) and 1 pbw of the binder being tested were formed into 2 inch cubes. The molds used were those specified in ASTM C109-58 (Compressive Strength of Hydraulic Cement Mortars). The mortar was prepared as follows: The two components of the binder were stirred together by hand for five minutes. The Ottawa sand was then gradually stirred into the binder. The sand and binder were mixed together for five minutes and then placed in the molds. Forming of the mortar into cubes was completed within 20 minutes after initiation of mixing of the two binder components together. The cubes were allowed to cure in the molds for 6 hours at 75 to 80 degrees F. prior to testing. Upon being removed from the molds, the cubes were subjected to compressive loading at the rate of 0.05 inch per minute. For purposes of comparison, the maximum load attained up to 0.05 inch deformation was recorded for each mortar. The wheel loads to which mortars used in bridge deck repair would be subjected would not be expected to be greater than 150 pounds per square inch. However, the effect of impact loading would augment the stresses to which the mortars would be subjected. It was felt that if epoxy mortar specimens prepared and tested as described above evidenced a strength on the order of 500 psi or greater, the mortars and concretes prepared for field use from the binder under consideration would have a sufficiently rapid set to perform satisfactorily.

In order to determine the comparative strength properties of mortars formed from the binders under consideration, two tests were used. One of these tests was another modification of ASTM C190-59 (Tensile Strength of Hydraulic Cement Mortars). The standard briquette molds were used to mold epoxy mortar specimens. Because the Riehle briquette tester has a maximum capacity of only 1000 pounds, a full size epoxy mortar briquette could not be taken to failure. Therefore, the molds were modified by reducing the depth to one-half inch. A one-half inch thick, shaped, wooden block was inserted into each mold space and the epoxy mortar molded in place on top of the wooden inserts. The resulting briquettes had a cross-sectional area at the mid point of one-half square inch. One of these briquettes is shown in Figure 1.

The second test employed was a compressive strength test. The molds used in making specimens for Compressive Strength of Hydraulic Cement Mortars (ASTM C190-58) were used to prepare 2 inch epoxy mortar cubes. These cubes were loaded in compression until failure occurred and the compressive modulus of elasticity as well as the ultimate compressive strength were determined. In order to calculate the compressive modulus, the deformation of the mortar cubes was obtained by determining the movement of the platen of the compression machine relative to the stationary head using a dial gage. Simultaneous readings of load and deformation were recorded. One of the mortar cubes in the process of being tested is shown in Figure 2.

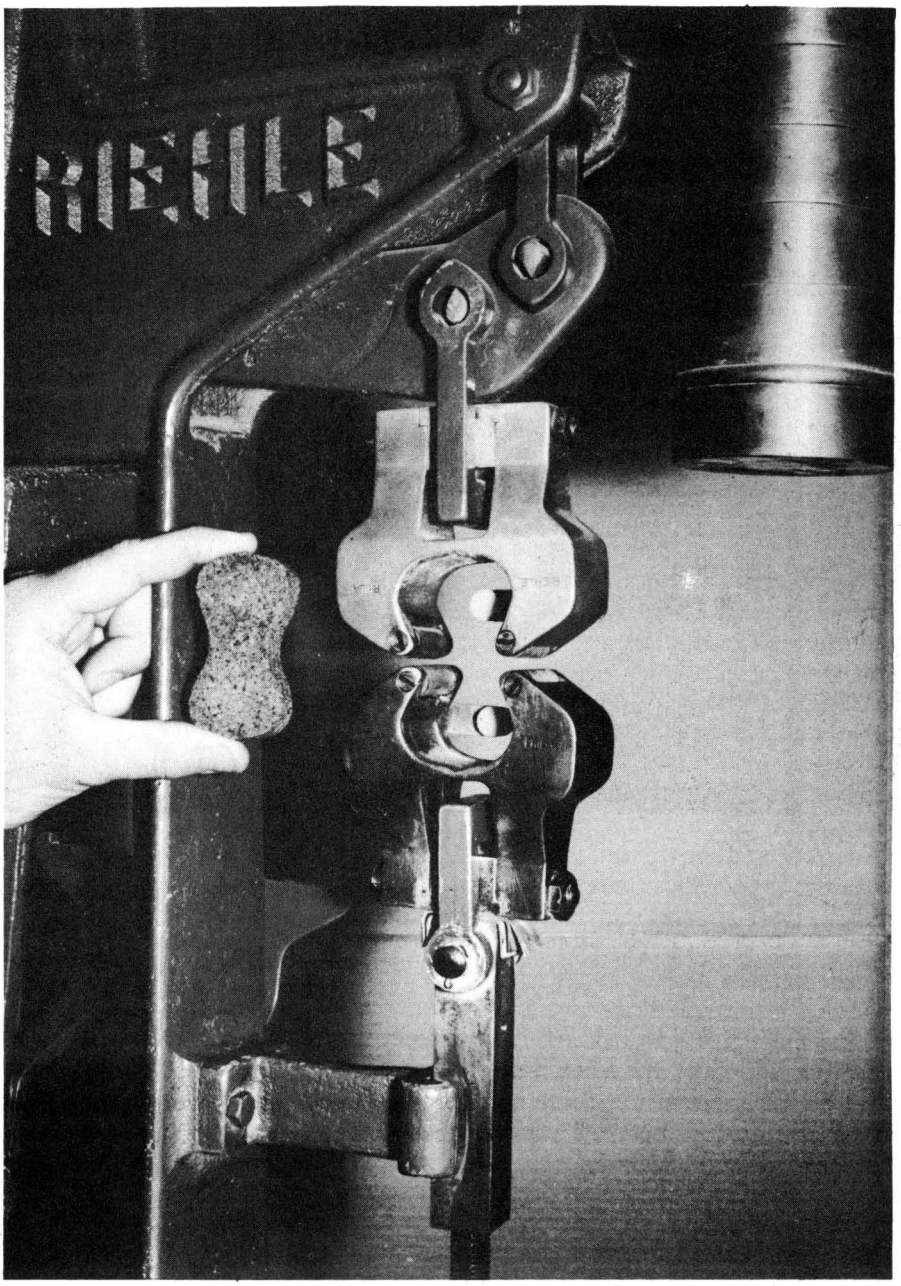

Figure 1.
Epoxy Mortar Tensile Specimen
About to be Placed in Riehle
Briquette Tester

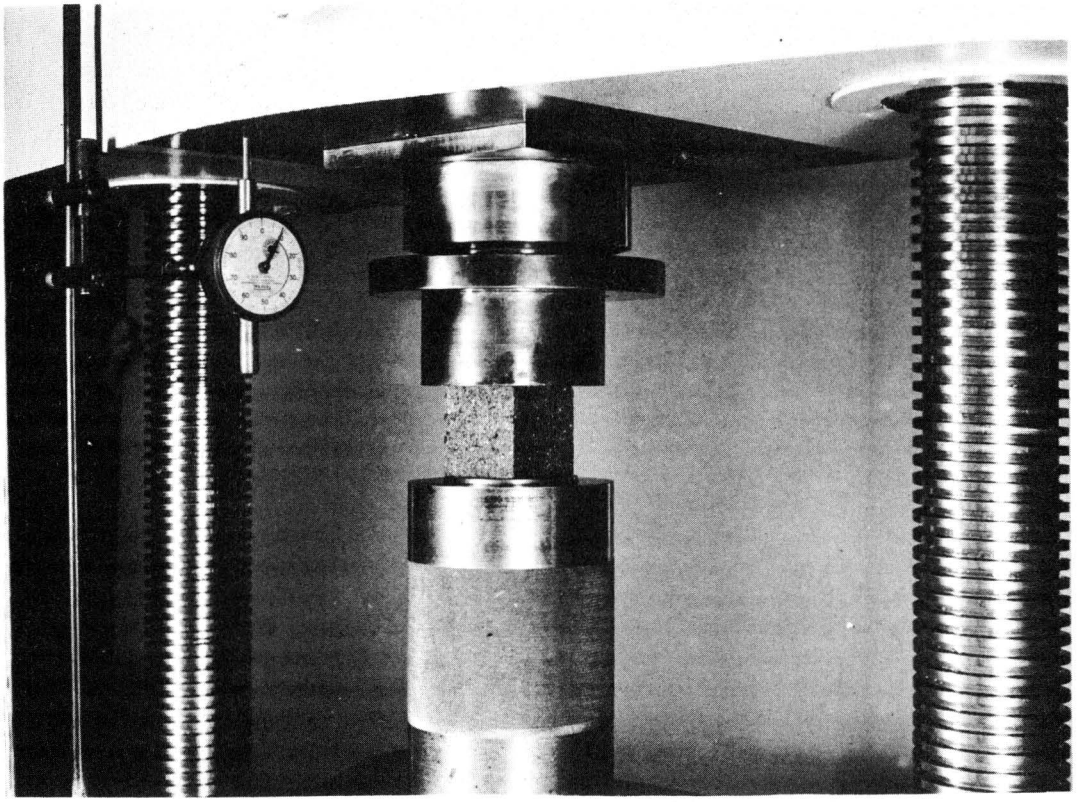

Figure 2.
Compressive Strength Test on Epoxy Mortars

It was found that the majority of the epoxy mortars did not perform as truly elastic materials in that the stress-strain relationship was not a straight-line function. The moduli given in this report are the slopes of chords drawn from 25% of ultimate strength to 75% of ultimate strength on the stress-strain curves. This method of determining the moduli was chosen for two reasons. First of all, the stress-strain relationship at low stress values was rather erratic for most of the mortars tested. Secondly, in almost all cases, the portion of the stress-strain curve between 25 and 75% of the ultimate strength most nearly approximated a straight line.

The final test used in evaluating epoxy binders was a determination of the thermal coefficients of the mortars produced from the various binders and aggregate. A bar of epoxy mortar 1" x 1" x 6¼" was cast and allowed to cure for seven days at 70-80 degrees F. This bar was then heated to 160 degrees F. in an oven. The hot bar was removed from the oven and mounted in one of two ways. Initially, the bars were placed with one end flush against a laboratory table top and between smooth, masonite covered faces of a vise. The vise was drawn up to the point that the faces would just touch the bar but would not

prevent its expansion. With the bar thus held in a vertical position, a dial gage capable of reading to 0.0001 inch was zeroed on the opposite end of the bar, which was then allowed to cool overnight to room temperature and the change in length determined by reading the gage. The method of mounting the bar was later changed. The bottom ¼ inch of the bar under test was clamped in the vise in a vertical position and the dial gage zeroed on the opposite end. This prevented any movement of the bar during the test. The results obtained by the two different methods of mounting the specimen did not differ appreciably. The change in length of the bar along with the change in temperature allowed us to calculate an average coefficient over the general range of 80 to 160 degrees F. The bar was then cooled to 0 degrees F. in a freezer and the same procedure repeated. This resulted in an average thermal coefficient over the range of 0 to 80 degrees F. A picture of this test in progress is shown below.

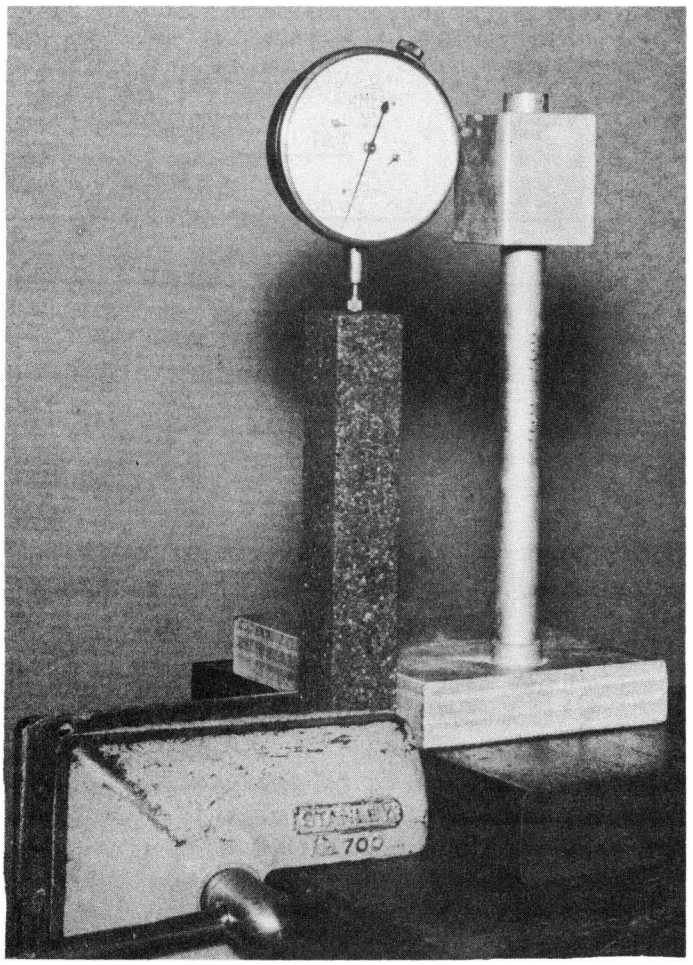

Figure 3.
Determination of Coefficients of
Thermal Expansion for Epoxy Mortars

For the Compressive and Tensile Strength tests and the Determination of Thermal Coefficients which were carried out on mortars, the procedure for preparation of the mortars was as follows:

The two components of the binder were weighed together in the proper proportions and mixed by hand for approximately 3 minutes. The aggregate was then added gradually to the binder and the mixture stirred well during the addition to insure that all the aggregate was uniformly wet with the epoxy binder. The resulting mortar mix was then formed into test specimens by essentially the same procedure used for cement mortars.

The aggregate used in preparing the specimens for these tests was prepared as follows: A sand meeting the requirements for Fine Aggregate under Item 403, "Concrete for Structures", Texas Highway Department 1951 Standard Specifications for Road and Bridge Construction, was separated into narrow gradations by screening. The screened material was dried in a 140 degree F. oven to make sure it was free from moisture. The various sizes of aggregate were combined to produce a fine aggregate of the gradation shown in Table 4.

Table 4

Gradation of Aggregate Used to Prepare Mortar Specimens

U.S. Standard Screen No.	Percent Retained, Cumulative
10	0
30	40
50	80
100	100

A ratio of 7 pbw aggregate to 1 pbw of each epoxy binder tested was used to make up the mortar.

A list of the tests which have been described and used in evaluating epoxy binder formulations follows.

Tests performed on the binder with no aggregate added: All of these tests are described in detail in the portion of this report dealing with development of the epoxy adhesive.

1. Adhesive Shear Test
2. Cleavage Test
3. Water Absorption Test
4. Falling Ball Impact Test

For all of these tests, the specimens were cured for 7 days at 70 to 80 degrees F. prior to testing.

Tests performed on epoxy mortar consisting of binder plus aggregate:

1. Ability to Bond to Cured Portland Cement Mortar. (The results will be reported as load at failure in pounds per square inch.)
2. Ease of Mixing and Workability. (Will be reported simply as good, fair, or poor.)
3. Ability to Support Traffic Loads after 6 Hours. (Will be reported as compressive strength after 6 hours at 75-80 degrees F.)
4. Tensile Strength. (Results will be reported in psi based on the minimum cross-sectional area of the test briquette.)
5. Compressive Strength. (Results will be reported in psi at failure.)
6. Modulus of Elasticity, Compressive.
7. Water Absorption Test. (The results will be reported as water gain, percent by wt.)
8. Thermal Coefficient. (Results will be reported as inches per inch per degree F. for two temperature ranges - 0 to 80 degrees F. and 80 to 160 degrees F.)

For tests 4 through 8, the specimens were cured for 7 days at 70-80 degrees F. prior to testing.

In summary, the adhesive shear and cleavage tests would indicate the ability of the various binder formulations to bond to steel and would give information regarding the ultimate strengths of various formulations. The water absorption tests on both the pure binder and the mortar specimens would indicate the completeness of reaction between the epoxy resin and the curing agent. The impact test was considered important because thin layers of epoxy grout or mortar would need to have good impact strength to perform satisfactorily. The test for ability to bond to cured concrete was included because this material would be used mainly to patch portland cement concrete structures. The ease of mixing and workability is an important factor in selecting an epoxy binder. Its usefulness is hampered considerably if it is difficult to mix with aggregate and difficult to place. The test to determine set time is important because one of the main advantages of using an epoxy mortar or concrete is its quick set. If the epoxy mortar will not set up rapidly enough at 80 degrees F. to support traffic loads within approximately six hours, it loses much of its usefulness. The tensile and compressive tests give an indication of the relative ultimate strengths which may be expected from epoxy mortars made up with the various binders. The compressive modulus and the thermal coefficient are both quite important in determining the relative merits of various epoxy mortars. The epoxies, along with other plastic materials, have a high coefficient of thermal expansion compared to that of concrete. The difference in thermal coefficients can be minimized by using a high ratio of aggregate to epoxy binder. However, the resulting epoxy mortar will still tend to expand and contract more than portland cement concrete with changes in temperature. Because of this, it is possible for stresses to be set up in a patched area due to changes in temperature. These stresses can become sufficiently great that, when combined with stress due to loading of the structure, the epoxy mortar patch will lose adhesion to the concrete or cause failure in the layer of concrete adjacent

to the patch. In order to offset the problem of difference in thermal coefficients, it is desirable first of all to obtain an epoxy material with a thermal coefficient as close to that of portland cement concrete as is practical. Secondly, it is desirable that the epoxy binder be a flexible rather than a rigid material; i.e., that it have a low modulus. The tests for compressive modulus and thermal coefficients would help in selecting a material with the best combination of these two properties.

In Table 5 below, data is presented on the repeatability of the tests performed on the various binder formulations and the binders in combination with aggregate.

Table 5

Repeatability of Tests Used in Developing Epoxy Binders

Test	Maximum Average Deviation for a Set of Two or More Specimens, Percent	Average of the Average Deviations for All Sets of Specimens, Percent
Tests performed on binder formulations:		
Adhesive Shear	9.7	5.7
Cleavage	5.0	3.6
Water Absorption	20.8	9.5
Impact	8.0	4.5
Tests performed on binder formulations plus aggregate:		
Tensile	9.6	4.5
Compressive	8.9	2.8
Water Absorption	23.5	8.6
Compressive Modulus	15.0	6.0
Thermal Coefficient	6.9	2.2

In a few instances deviations from average values greater than the maximums shown above were obtained. These results were discarded because of obvious errors in preparation of the binder formulations or the test specimens.

DISCUSSION

In developing an epoxy binder, the information regarding the basic constituents of epoxy formulations and the various combinations thereof obtained in

developing an epoxy adhesive was utilized. Initially, seven trial formulations were prepared and tested to determine their suitability as epoxy binders. These formulations are listed in Table 6. Note: Throughout this discussion, the abbreviations HVUR and LVUR will be used to designate High Viscosity Unmodified Epoxy Resin and Low Viscosity Unmodified Epoxy Resin respectively.

Table 6

Experimental Binder Formulations

Formulation 1	Formulation 2	Formulation 3
100 pbw HVUR	100 pbw HVUR	100 pbw HVUR
50 pbw Versamid 140	50 pbw Genamid 250	20 pbw Epi-Cure 855
		15 pbw Epi-Cure 87
Formulation 4	**Formulation 5**	**Formulation 6**
100 pbw HVUR	100 pbw HVUR	100 pbw HVUR
40 pbw Epi-Cure 855	50 pbw Lancast A	50 pbw Thiokol LP-3
10 pbw Epi-Cure 87	3 pbw DMP-30	10 pbw DMP-30
	Formulation 7	
	100 pbw HVUR	
	75 pbw Thiokol LP-3	
	10 pbw DMP-30	

The first step was to determine the ease of mixing and workability and the approximate usable pot life for these seven formulations. The results obtained are shown in Table 7.

Table 7

Handling Characteristics of Formulations 1 thru 7

Formulation	Ease of Mixing and Workability	Usable Pot Life at 75-80 Degrees F., Minutes
1	Fair to Poor	50
2	Good	60
3	Fair	45
4	Good	55
5	Good	55
6	Fair	45
7	Fair	45

The set time for these formulations was then considered. By probing the mortar which had been made up to determine ease of mixing and workability, it was ascertained that after 6 hours at 80 degrees F., formulations 1 and 2 were too soft to bear a load. For formulations 3 through 7, compression specimens were prepared to determine their load bearing capabilities after 6 hours cure at 75 to 80 degrees F. The results obtained are shown below:

Table 8

Determination of Curing Rate for Formulations 3 thru 7

Formulation	Compressive Strength After 6 Hours at 75-80 Degrees F., PSI
3	5000*
4	575
5	725
6	1500
7	325

*This was the maximum stress obtained, which occurred at 0.030 inch deflection.

All of these formulations with the exception of No. 7 set rapidly enough according to the criterion which we had established. Since Formulation 7 was of a flexible nature, the load obtained at 0.050 inch deformation represented a comparatively lower percentage of the ultimate load which the mortar could support. In view of this, Formulation 7 was considered along with Formulations 3 through 6 as a possible binder.

The physical properties of the above mentioned binders were determined. The results are tabulated in Tables 9 and 10.

Table 9

Results of Tests on Binders 3 thru 7 with No Aggregate Added

Property	3	4	5	6	7
Adhesive Shear Strength, PSI, Avg.	2450	2610	2810	3190	3105
Cleavage Strength, PSI, Avg.	1320	1395	1325	1365	1150
Water Gain, Percent by Wt., Avg.	0.12	0.07	0.17	0.19	0.35
Impact Strength, Ft.-Lbs., Avg.	5.5	7.0	5.5	7.5	9.0**

**Withstood limit of test equipment without failure occurring.

Table 10

Results of Tests on Mortars Produced From Binders 3 thru 7

Property	Formulation Number 3	4	5	6	7
Ability to Bond to Cured Portland Cement Mortar Load at Failure, PSI, Avg.	495	430	495	455	480
Type of Failure	100% in Portland Cement Mortar	100% in PCM	100% in PCM	100% in PCM	100% in PCM
Tensile Strength, PSI, Avg.	1395	1130	1040	1270	1700
Compressive Strength, PSI, Avg.	8020	7375	8900	7010	8225
Water Gain, Percent by Wt., Avg.	0.20	0.16	0.14	2.16	1.62
Modulus of Elasticity, Compressive x 10^6	1.10	0.91	0.90	0.62	0.31
Thermal Coefficient, In./In./Degree F. x 10^{-6} (0 to 80 Degrees F. Range)	10.1	9.2	10.2	10.5	13.3
Thermal Coefficient, In./In./Degree F. x 10^{-6} (80 to 160 Degrees F. Range)	14.4	15.2	14.0	18.8	19.2

The results of all the tests performed on these five binders indicated that Formulation 7 had somewhat better overall properties than the other formulations. Although Formulation 7 had the highest thermal coefficients, it also had the lowest modulus of elasticity. It was decided that Formulation 7 would be the basis of our binder formulation.

The information obtained on this type of formulation in the developmental work on epoxy adhesives indicated that a lower ratio of DMP-30 to epoxy resin might be desirable from the standpoint of physical properties of the cured material. Two trial formulations of the same composition as No. 7, but containing lesser amounts of DMP-30, were prepared and subjected to the set time test.

The results obtained are shown in Table 11.

Table 11

Effect of Lower Concentrations of DMP-30 on Set Time of Formulation 7

Pbw DMP-30 per 100 pbw Epoxy Resin	Compressive Strength After 6 Hours at 75-80 Degrees F., PSI
8	50
9	160

The above results indicated that a lower ratio of DMP-30 in Formulation 7 would result in too slow a set time, so it was decided that 10 pbw per 100 pbw epoxy resin would be used.

In order that the binder and the resulting epoxy mortar have a color more closely resembling that of concrete, a small amount of titanium dioxide and carbon black were added to produce a gray color. Two pbw DuPont R-610 titanium dioxide and 0.03 pbw Excelsior carbon black, manufactured by Columbian Carbon Co., were added to obtain the desired color.

To provide flow control, 4 pbw M-5 Cab-O-Sil was added per 100 pbw epoxy resin. To aid in gel formation, 1.0 pbw water per 100 pbw epoxy resin was also added. It was found that this amount of thixotropic agent would provide resistance to flow on sloping surfaces. Although the addition of the M-5 Cab-O-Sil did reduce the ease of mixing and the workability of the formulation somewhat, it was thought at the time that being able to use the mortar on sloping and vertical surfaces would outweigh these disadvantages. The pigments and the thixotropic agent were milled into the epoxy resin using a Cowles Dissolver. The finished epoxy binder formulation was as follows:

Resin Component:	100	pbw HVUR
	4	pbw M-5 Cab-O-Sil
	2	pbw DuPont R-610 titanium dioxide
	0.03	pbw Columbian Excelsior carbon black
	1	pbw water
Hardener Component:	75	pbw Thiokol LP-3 polysulfide
	10	pbw DMP-30

The mixing ratios for this formulation were as follows:

5 parts resin component by weight
4 parts hardener component by weight

or 7 parts resin component by volume
5 parts hardener component by volume

This material was designated as Texas Highway Department Epoxy Binder B-100. The packaging was such that mixing could be accomplished in one of the original containers. Four pounds of the resin component was packaged in a gallon container and 3.2 pounds of the hardener component was packaged in a one-half

gallon can with corrosion resistant lining. The hardener component was to be added to the resin component and mixing done in the resin component container. The two components when mixed together produced 3/4 gallon of finished binder. Fifty of these 3/4 gallon units of Epoxy Binder B-100 were purchased on a competitive bid basis in order that this material might be given a field trial. The price, including inspection and testing costs, was $8.55 per unit.

Since it was readily available, a concrete sand was used in most cases as the aggregate to make up mortars with Epoxy Binder B-100. Difficulty was encountered in mixing the desired amount of sand (6 to 7 pbw per 1 pbw epoxy binder) with B-100. Also, the workability of the resulting mixes was not as good as would be desired. Almost all of this material was being used in patching bridge decks or roadways (i.e., horizontal surfaces), and thus the thixotrophy or flow resistance was not needed.

In conjunction with the developmental work on epoxy adhesives and binders, weathering and aging tests were also performed, the results of which are discussed in Part III of this report. The results obtained indicated unsatisfactory performance on the part of epoxy-polysulfide mortars. The mortars prepared from binders cured with modified amines and modified amino-amides exhibited good aging and weathering properties. Work was initiated to develop a more satisfactory epoxy binder to replace B-100.

In developing a replacement for Epoxy Binder B-100, new resins and curing agents which had become available were considered. An unmodified epoxy resin of 7,000 to 10,000 cps viscosity was now available. This lower viscosity would aid considerably in developing a more workable formulation. Tests performed on this resin indicated that it was equivalent to the higher viscosity unmodified resins with respect to performance. (See Table 17, page 30, in section dealing with development of epoxy adhesive).

In the original work on epoxy binders, Formulation 4, which utilized a combination of Epi-Cure 87 and Epi-Cure 855 as a curing agent had performed fairly well. The Jones-Dabney Co. was now marketing a combination of these two curing agents as Epi-Cure 872. This curing agent was described by the manufacturer as containing a ratio of 2½ parts by weight Epi-Cure 855 to 1 part by weight Epi-Cure 87. Epi-Cure 872, in combination with the low viscosity unmodified epoxy resin was tested as a possible binder. The following two formulations were tried:

Formulation 8	Formulation 9
100 pbw LVUR	100 pbw LVUR
40 pbw Epi-Cure 872	45 pbw Epi-Cure 872

The ease of mixing and workability and the usable pot life were determined for these two formulations with the results shown in Table 12.

Table 12

Handling Characteristics of Formulations 8 and 9

Formulation	Ease of Mixing and Workability	Usable Pot Life at 75-80 Degrees F., Minutes
8	Good	55
9	Good	50

Compressive specimens were then prepared to determine the load bearing capabilities of these formulations after 6 hours at 75-80 degrees F. The results are given in Table 13.

Table 13

Determination of Curing Rate for Formulations 8 and 9

Formulation	Compressive Strength After 6 Hrs. at 75-80 Degrees F., PSI
8	520
9	630

The above results indicated that both of these formulations had satisfactory cure rates. A complete set of physical tests were performed on Formulations 8 and 9, the results of which are shown in Tables 14 and 15.

Table 14

Results of Tests on Binders 8 and 9 with No Aggregate Added

Property	Formulation No. 8	9
Adhesive Shear Strength, PSI, Avg.	2810	2746
Cleavage Strength, PSI, Avg.	1444	1470
Water Gain, Percent by Wt., Avg.	0.07	0.06
Impact Strength, Ft.-Lbs., Avg.	6.0	7.5

Table 15

Results of Tests on Mortars Produced From Binders 8 and 9

Property	Formulation No. 8	9
Ability to Bond to Cured Portland Cement Mortar (Load at Failure, PSI, Avg.)	515	480
Type of Failure	100% in Portland Cement Mortar	100% in Portland Cement Mortar
Tensile Strength, PSI, Avg.	1395	1300
Compressive Strength, PSI, Avg.	7710	7800
Water Gain, Percent by Wt., Avg.	0.17	0.19
Modulus of Elasticity, Compressive	1.02×10^{6}	0.89×10^{6}
Thermal Coefficient, In./In./Degree F. (0 to 80 Degrees F.)	9.2×10^{-6}	9.7×10^{-6}
Thermal Coefficient, In./In./Degree F. (80 to 160 Degrees F.)	14.6×10^{-6}	14.5×10^{-6}

The results of all the tests performed on Formulation 8 and 9 indicated fairly good overall properties. These binders did not evidence quite as high strengths as Epoxy Binder B-100 nor did they have as low a modulus of elasticity. However, these formulations had the advantages of good workability and ease of mixing. It would be possible to load them with a higher percentage of aggregate which would help considerably in bringing the thermal coefficients nearer to that of portland cement concrete. Also, we had information from the aging and weathering tests on a similar formulation which indicated that these formulations should age and weather quite well.

The only physical property in which there was any significant difference between Formulations 8 and 9 was the flexibility. Since Formulation 9 exhibited better impact resistance and a lower modulus of elasticity, we chose to use it as the basic formulation for our new epoxy binder. No thixotropic agent was added to this basic formulation. However, it was pigmented with titanium dioxide and carbon black which were milled into the epoxy resin with a Cowles Dissolver. A larger amount of titanium dioxide than was used in Binder B-100 was added in order to give the binder a little more coloring power. DuPont R-610 titanium dioxide was replaced

with DuPont R-900. The resulting color of this formulation was somewhat lighter than Epoxy Binder B-100. The finished formulation was as follows:

Resin Component:	100 pbw	LVUR
	6 pbw	DuPont R-900 titanium dioxide
	0.01 pbw	Columbian Excelsior carbon black
Hardener Component:	45 pbw	Epi-Cure 872

The mixing ratios for this formulation were as follows:

7 parts resin component by weight
3 parts hardener component by weight

or 2 parts resin component by volume
1 part hardener component by volume

This formulation was designated as Epoxy Binder B-101, and was packaged in 3/4 gallon units as described below.

One-half gallon of the resin component was packaged in a gallon can and one quart of hardener component was packaged in a quart can. The unit was designed so that the hardener could be added to the resin and mixing done in the gallon container prior to mixing the binder with aggregate to form a mortar. 426 units of this material were purchased for maintenance use on a competitive bid basis at a cost of $6.40 per unit, including inspection and testing. Reports from the field indicated that this material was performing satisfactorily. There was some indication that it was a little too fast setting in hot weather, which was a disadvantage.

After formulating Epoxy Binder B-101, additional work was done in the laboratory toward further improving the binder formulation. A part of this work was concerned with the pigment concentration present in the binder. Initially it was thought that the small amounts of pigment used to color the binder would have little or no effect upon its physical properties. However, in order to determine the actual effect of the pigments, tests were performed on the basic B-101 formulation (Formulation 9) into which were incorporated varying amounts of titanium dioxide and carbon black. The results are presented in Table 16. The pigment concentrations shown are in parts by weight per 100 parts by weight of epoxy resin.

The results indicate that for an epoxy-amino-amide system, the impact strength decreases as the pigment concentration reaches 8 to 10 pbw per 100 pbw of epoxy resin. The other physical properties did not seem to be affected. We decided that it would be best to limit the titanium dioxide content to 4 pbw per 100 pbw epoxy resin and the carbon black content to 0.005 pbw epoxy resin. This ratio of titanium dioxide to carbon black gave a satisfactory color.

Table 16

Results of Tests on Formulation 9 Plus Pigments Shown

Property	2 Pbw TiO_2 0.005 Pbw Carbon Black	4 Pbw TiO_2 0.005 Pbw Carbon Black	6 Pbw TiO_2 0.01 Pbw Carbon Black	8 Pbw TiO_2 0.01 Pbw Carbon Black	10 Pbw TiO_2 0.01 Pbw Carbon Black
Adhesive Shear Strength, PSI, Avg.	2655	2697	2730	2742	2630
Cleavage Strength, PSI, Avg.	1426	1510	1485	1490	1450
Water Absorption, Percent by Wt., Avg.	0.08	0.09	0.09	0.07	0.05
Impact Strength, Ft.-Lbs., Avg.	7.5	7.7	7.5	7.0	6.7

Some additional work also was done with the curing agents. Two new hardeners, Genamid 2000, manufactured by General Mills, and Epi-Cure 858, manufactured by Jones-Dabney were considered for possible use in the binder. Epi-Cure 858 had a viscosity of 20,000 to 30,000 cps at 75 degrees F., which is too high for use in a binder formulation. However, this hardener was considered in combination with Epi-Cure 872. Several formulations were prepared and tested for their suitability as a binder. The pigments were included in these test formulations. The resin component was the same for all the formulations and was composed of the following:

100 pbw LVUR
4 pbw TiO_2
0.005 pbw carbon black

The various hardener components used with the above resin component are listed below:

Formulation 10	Formulation 11	Formulation 12	Formulation 13
50 pbw Genamid 2000	40 pbw Epi-Cure 872 10 pbw Epi-Cure 858	40 pbw Epi-Cure 872 20 pbw Epi-Cure 858	30 pbw Genamid 2000 25 pbw Epi-Cure 855

Formulation 14

40 pbw Epi-Cure 872
15 pbw Versamid 140

The ease of mixing and workability and the usable pot life was determined for these five formulations. The results are given in Table 17.

Table 17

Handling Characteristics of Formulations 10 thru 14

Formulation	Ease of Mixing and Workability	Usable Pot Life at 75-80 Degrees F., Minutes
10	Fair	50
11	Fair	60
12	Fair	65
13	Good	65
14	Good	60

The usable pot life of Formulation 10 was approximately the same as that of Epoxy Binder B-101. This indicated that Formulation 10 would have too short a pot life when used during hot summer weather, so it was not considered further. Compression specimens were prepared for the other four formulations to determine their load bearing capabilities after 6 hours at 75-80 degrees F. The results obtained are given in Table 18.

Table 18

Determination of Curing Rate for Formulations 11 thru 14

Formulation	Compressive Strength After 6 Hrs. at 75-80 Degrees F., PSI
11	440
12	415
13	480
14	525

The results obtained indicated that Formulations 13 and 14 would be the most satisfactory with regard to set time. These two formulations were superior to Formulations 11 and 12 with regard to ease of mixing and workability, so Formulations 13 and 14 were chosen for further consideration as binders. The results of additional tests on these formulations are given in Tables 19 and 20.

Table 19

Results of Tests on Binders 13 and 14, No Aggregate Added

Property	Formulation No. 13	14
Adhesive Shear Strength, PSI, Avg.	2730	2985
Cleavage Strength, PSI, Avg.	1600	1723
Water Gain, Percent by Wt., Avg.	0.20	0.14
Impact Strength, Ft.-Lbs., Avg.	7.2	8.0

Table 20

Results of Tests on Mortars Produced From Binders 13 and 14

Property	Formulation No. 13	14
Ability to Bond to Cured Portland Cement Mortar (Load at Failure, PSI, Avg.)	495	480
Type of Failure	100% in portland cement mortar	100% in portland cement mortar
Tensile Strength, PSI, Avg.	835	1200
Compressive Strength, PSI, Avg.	6625	7990
Water Gain, Percent by Wt., Avg.	0.20	0.21
Modulus of Elasticity, Compressive	0.81×10^{6}	0.95×10^{6}
Thermal Coefficient, In./In./Degree F., (0 to 80 Degrees F.)	9.7×10^{-6}	8.7×10^{-6}
Thermal Coefficient, In./In./Degree F., (80 to 160 Degrees F.)	14.6×10^{-6}	14.5×10^{-6}

The above results indicated that Formulation 14 was superior to Formulation 13 with regard to physical properties. Since Formulation 14 had a longer pot life and exhibited just as good physical properties as Epoxy Binder B-101, it was chosen to replace B-101, and was designated as Epoxy Binder B-102. This is the

binder now being used by the Texas Highway Department in maintenance work. It is being packaged in 3/4 gallon and 2½ gallon units. The 3/4 gallon unit consists of 4.6 pounds (a little less than ½-gallon) of resin packaged in a gallon can, and 2.4 pounds (approximately 1/3 of a gallon) of hardener packaged in a ½-gallon can. For the 2½ gallon unit, 15.1 pounds (approximately 1½ gallons) of resin component is packaged in a 5-gallon container, and 8.0 pounds (one gallon) of hardener component is packaged in a 1-gallon can. Both of these units are designed so that the hardener can be added to the resin and mixing accomplished in the resin component container prior to combining the binder with aggregate to form a mortar.

Approximately 4100 gallons of Epoxy Binder B-102 have been purchased to date by the Texas Highway Department at an average cost of approximately $9.50 per gallon, including inspection and testing expenses. Reports from the field indicate that this material is performing satisfactorily.

In the appendix to this report is a copy of the specification which is currently being used to purchase this material on a competitive bid basis. Also included is a copy of the instruction pamphlet which is issued to the personnel engaged in the use of the binder.

Although this investigation was concerned primarily with the development of an epoxy binder, the aggregates used with the binder are of considerable importance in view of the fact that 80 to 90% by weight of an epoxy mortar or concrete consists of aggregate. A limited investigation of aggregates for use with the epoxy materials was done along with the developmental work on the epoxy binder.

The recommendations of the epoxy materials manufacturers, and specifications obtained from the various governmental agencies concerning aggregates for use with the epoxies, were fairly general. The main recommendations concerning aggregates were as follows:

1. The aggregates should be clean, dry, and non-porous.
2. The aggregates used should be igneous rather than sedimentary types.
3. The grading should be controlled to provide the best workability in the finished mortar.
4. The range of aggregate size should be varied with the depth of the patches to be placed.

It is desirable from the standpoint of field personnel that the aggregate be a low cost, readily available material. Considering this, our initial recommendation regarding aggregates was that sand meeting the requirements for Grade No. 1 Fine Aggregate as specified under Item 421, "Concrete for Structures", Texas Highway Department 1962 Standard Specifications for Road and Bridge Construction, be used in conjunction with the epoxy binder to produce a mortar. Experience in mixing small laboratory batches and field size batches of epoxy mortar indicated that 6 to 7 parts by weight of the above aggregate to 1 part by weight of epoxy binder produced the most workable and economical mix. Although the Grade No. 1 Fine Aggregate seemed to perform fairly well, there were quite possibly other aggregates which would perform better.

Five different aggregates were selected and tested in combination with Epoxy Binder B-102 to determine their relative suitability for use in epoxy mortars. These aggregates were as follows:

1. A silicious sand meeting 1962 Texas Highway Department Standard Specification for Fine Aggregate, Grade No. 1, Item 421, "Concrete for Structures".
2. The sand indicated above with all material passing the 50 mesh U.S. Standard Screen removed.
3. The same sand as No. 2, washed to remove any clay material and then dried in a 212 degree F. oven.
4. A rounded grain highly silicious washed sand produced by Pennsylvania Glass Sand Corporation, San Saba, Texas. It is reported to have the following chemical and physical properties:

   Mohs' hardness - - - - - 7 minimum
   Specific gravity - - - - 2.60 minimum
   Silica, percent by wt. - 99.5 minimum

   This sand is produced in narrow gradations. Several gradations were mixed together to produce the aggregate tested.
5. A composite sand made up from screened portions of a silicious concrete sand. The grading was exactly the same as that of Sand 4. This aggregate was included in order to eliminate the factor of grading when comparing a rounded sand such as No. 4 with a sharp, irregular sand. The screen analyses of these five aggregates are given in Table 21.

Table 21

Screen Analysis of Aggregates Tested

U.S. Standard Screen No.	Percent Retained on Each Screen, Cumulative				
	1	2	3	4	5
4	0				
8	13.7	17.1	Essentially the same as Aggregate No.2	0	Essentially the same as Aggregate No.4
10				3.0	
12				27.0	
16	33.3	41.7		47.6	
20	40.7	50.9		61.6	
30	51.2	64.1		89.8	
40	67.0	83.8		97.7	
50	79.9	100.0			
60	85.1				
80	93.4				
100	96.3				
200	98.7				

In comparing these five aggregates, a ratio of 7 parts by weight of aggregate to 1 part by weight of epoxy binder was used to make up the mortars which were tested. For all the tests involving cured specimens, the cure was 7 days at 70 to 80 degrees F. An additional test which was not used in evaluating mortar mixes prepared from different binders was run on these mortars. This was a variation of the impact test which was performed on the pure binders. The specimens were prepared by molding disks of the mortar 2-3/4 inches in diameter and a little over ¼ inch in thickness. After the mortar disks had attained a good initial set, they were ground on both sides to give plane parallel surfaces. The thickness after grinding was approximately 0.3 inch. The cured disks were tested by positioning them on a concrete floor and dropping a one pound steel ball onto the center of the disks from an initial height of one foot. The height of drop was increased in one foot increments until failure occurred. The height at which failure occurred was recorded as the impact strength in foot-pounds.

In comparing the workability of these five mortars, a number rating system was used. The mortar with the best working characteristics was rated No. 1 and the other mortars rated accordingly. The results obtained for the various tests are shown in Table 22.

Table 22

Results of Tests on Various Aggregates in Combination with Epoxy Binder B-102

Property	Aggregate Number 1	2	3	4	5
Comparative Workability	4	3	3	1	2
Tensile Strength, PSI, Avg.	1080	1035	980	1700	1205
Compressive Strength, PSI, Avg.	7010	7875	7575	10,500	7495
Water Gain, Percent by Wt., Avg.	0.20	0.18	0.24	0.17	0.23
Modulus of Elasticity, Compressive	0.56×10^{6}	0.70×10^{6}	0.56×10^{6}	0.97×10^{6}	1.90×10^{6}
Impact Strength, Ft.-Lbs., Avg.	1.0	1.0	1.0	3.0	3.0
Thermal Coefficient, In./In./Degree F. (0 to 80 Degrees F.)	9.1×10^{-6}	8.7×10^{-6}	8.7×10^{-6}	10.0×10^{-6}	9.8×10^{-6}
Thermal Coefficient, In./In./Degree F. (80 to 160 Degrees F.)	14.4×10^{-6}	14.1×10^{-6}	13.9×10^{-6}	13.8×10^{-6}	14.0×10^{-6}

These results indicate that the mortar containing Aggregate 4 had the best workability, while Aggregate 1 produced the least workable mortar. With regard to strength properties, the mortar which contained Aggregate 4, the rounded grain sand, was superior to the other mortars. There was no significant difference in the strength properties of the mortars produced from Aggregates 1, 2 and 3. Aggregate 5 produced a mortar with a little better strength properties than did Aggregates 1, 2 and 3.

The better workability achieved with Aggregate 4 apparently is due to the spherical shape of the aggregate particles and the small amount of fines present. A comparison of the strengths obtained with Aggregates 4 and 5 indicates that a rounded aggregate will give better performance than a sharp angular aggregate in epoxy mortars. The higher strengths obtained with the rounded aggregate could be due to a combination of several different factors. With the same degree of compaction, the mix containing the rounded aggregate will produce a denser mortar. The uniform composition of the aggregate particles probably contributes to the higher strengths. For a spherical aggregate, the ratio of surface area to volume is at a minimum. Therefore, for a given ratio of aggregate to epoxy binder, there is a thicker film of epoxy around each aggregate particle which apparently enhances the strength of the cured mortar. Also, the rounded grain sand is wet more uniformly than the conventional concrete sands due to the smooth, uniform surface.

One marked difference in the properties of the mortars made up from these five aggregates was the comparatively high compressive modulus of elasticity obtained with Aggregate 5. This was probably due to an interlocking of the aggregate particles under load.

As a result of the work on aggregates, the following points, in addition to those listed on page 24, should be considered in selecting an aggregate.

1. Better workability and physical properties can be obtained with a rounded aggregate as opposed to a sharp aggregate.
2. Workability and physical properties can be improved by limiting the amount of material finer than 50 to 60 mesh.

In view of the effect that variation in aggregate composition can have on the properties of the finished epoxy mortar, additional work on aggregates for use with epoxy binders should be quite beneficial.

Regarding the epoxy binder, the Materials and Tests Division desires to provide a material which will suit the needs of the maintenance forces as closely as possible. Their suggestions along with new developments in the field of epoxies may result in improvements in the current binder formulation.

PART III

AGING AND WEATHERING CHARACTERISTICS OF EPOXY ADHESIVES AND BINDERS

Development of Epoxy Materials for Highway Use

Part III

SCOPE

The purpose of this investigation was to determine the performance of various epoxy adhesive and binder formulations with regard to aging and weathering.

OBJECTIVES

The objective of this work was to obtain answers to the following specific questions.

1. Will the properties of the epoxy formulations used as adhesives and binders change with aging and/or weathering, and to what extent?
2. If the properties of these materials do change due to aging and/or weathering, will these changes adversely affect the performance of the epoxy formulations?
3. Are certain types of epoxy formulations less susceptible to changes brought about by aging and weathering than other types of epoxies?

CONCLUSIONS

The properties of all the epoxy formulations tested showed a change in properties as a result of aging and/or weathering. The magnitude of the change varied with the different types of epoxy formulations and the different test conditions. The changes in some of the epoxies as a result of aging and weathering did adversely affect their performance. It was found that generally, epoxies cured with modified amines or modified amine-polyamide combinations were less susceptible to weathering and aging changes than the epoxy-polysulfide systems. The change in properties of epoxy-polysulfide systems was sufficiently great in several cases to cause failure of these materials when used as concrete adhesives or as binders to produce epoxy mortar for patching concrete structures.

Specific conclusions reached as a result of the aging and weathering tests are as follows:

1. Epoxies cured with modified amines or modified amine-polyamide combinations should give longer service in highway applications, under the same conditions of exposure, than epoxy-polysulfide systems because of their greater resistance to weathering and aging changes.
2. Changes in the epoxy materials occur more rapidly at elevated temperatures (specifically 135 degrees F. or greater). The epoxy-polysulfide systems tested lost strength quite rapidly at elevated temperatures. The most noticeable change in the epoxies cured with modified amines or modified amine-polyamide combinations at elevated temperatures was a loss of

flexibility. The deterioration of these formulations occurred much less rapidly than the deterioration which occurred in the epoxy-polysulfide formulations.

Although somewhat limited in scope, the aging and weathering tests performed on various epoxy formulations have helped in selecting epoxy materials which will give the best overall service in highway applications.

PROCEDURE

Soon after development work was begun on an epoxy adhesive and binder for highway use, the aging and weathering investigation was initiated. The first phase consisted of preparing specimens for actual aging and weathering under conditions of normal exposure. Exposure specimens were prepared for some of the more promising initial adhesive formulations and for some commercially available epoxy adhesives. Also, exposure specimens were prepared for three of the more promising initial binder formulations. Test specimens for some of the binder formulations were retained in the laboratory to determine the effect of aging.

In addition to the normal weathering and aging investigation, some aging tests at elevated temperatures were conducted in the laboratory. The aging of thermoplastic materials, such as epoxies, and the resultant changes in properties usually occur more rapidly with increase in temperature. Any epoxy materials used in highway maintenance in Texas could be exposed to temperatures as high as 120 to 140 degrees F. during the summer months. Changes in some of the epoxy formulations at temperatures in this range were investigated. The same epoxy formulations were also subjected to a temperature of approximately 180 degrees F. in order to determine the effect on physical properties. The tests and types of specimens used for the outdoor weathering and aging tests are described in the following paragraphs.

In order to evaluate the weathering and aging characteristics of epoxy adhesives, composite briquette specimens prepared as described in Part I of this report for the test designated as Ability to Bond Plastic Portland Cement Mortar to Cured Mortar were utilized. A minimum of six briquettes were prepared for each adhesive formulation to be tested. All of the briquettes were cured for seven days according to the procedure set forth in ASTM C190-59. One-third of each set of briquettes were tested immediately following the curing period as a control. The remaining briquettes were allowed to cure in air in the laboratory for an additional two weeks. Half of the remaining briquettes were then subjected to four freeze-thaw-heating cycles prior to being tested in tension. Each cycle consisted of the following:

16 hours at 0 degrees F.
8 hours at 70-80 degrees F.
16 hours at 158 degrees F.
8 hours at 70-80 degrees F.

The remaining briquettes were placed on sign racks maintained in the Austin area by the Paint Section of Materials and Tests Division for exposure and aging. After a period of time varying from 1 to 3 years, these briquettes were tested in the Reihle briquette tester.

During the final phase of developing Epoxy Adhesive A-100, two 3' x 3' concrete slabs, 1½ inches thick, were cast and cured. One-half of the surface of Slab #1 was etched with 10% hydrochloric acid and the remainder washed with detergent. The complete surface of Slab #2 was detergent washed. Both slabs were rinsed thoroughly and allowed to dry. A coating of epoxy adhesive 20 to 30 mils thick was applied on the surface of the slabs. While the epoxy was still tacky, plastic portland cement concrete was applied to the coated slabs. A portland cement concrete overlay approximately 1½ inches thick was formed on top of each of the existing slabs to form composite slabs approximately 3 inches thick with the epoxy adhesives acting as the bonding agent. The temperature during the week following placement of the overlays varied from a low of 60 to a high of 90 degrees F., which allowed the epoxy adhesives to obtain a good cure.

The purpose of preparing these two composite slabs was to obtain some experience in working with the adhesives on a larger scale than was possible in the laboratory and to determine the effect, if any, that different types of surface preparation might have on the bond. These slabs (hereafter referred to as "composite slabs") would also give us an opportunity to observe the weathering and aging characteristics of the adhesives used. This would be accomplished by taking cores out of the slabs initially and then at later intervals and comparing their performance when tested in tension. Each time the slabs were cored, four cores were taken from each slab. On Slab #1, two were taken from the side where acid etching was used to prepare the surface and two from the side that was detergent washed. The cores were prepared for testing as described below.

The cores were ground on each end to remove laitance and to make the end areas parallel with the epoxy layer. After grinding, approximately 1¼ inches of concrete remained on each side of the epoxy layer. The cores had a diameter of approximately 2 inches. The ground end surfaces of the cores were acid etched, rinsed, and allowed to dry. Sections of I-Beams such as were used in the Adhesive Tensile Test which is described in Part I of this report were bonded onto each end of the cores with an epoxy adhesive. After the epoxy had cured for 24 hours at room temperature, the specimens were subjected to tensile loading until failure occurred. The method of loading was the same as was used in the Adhesive Tensile Test. Figure 1 shows one of these cores being tested.

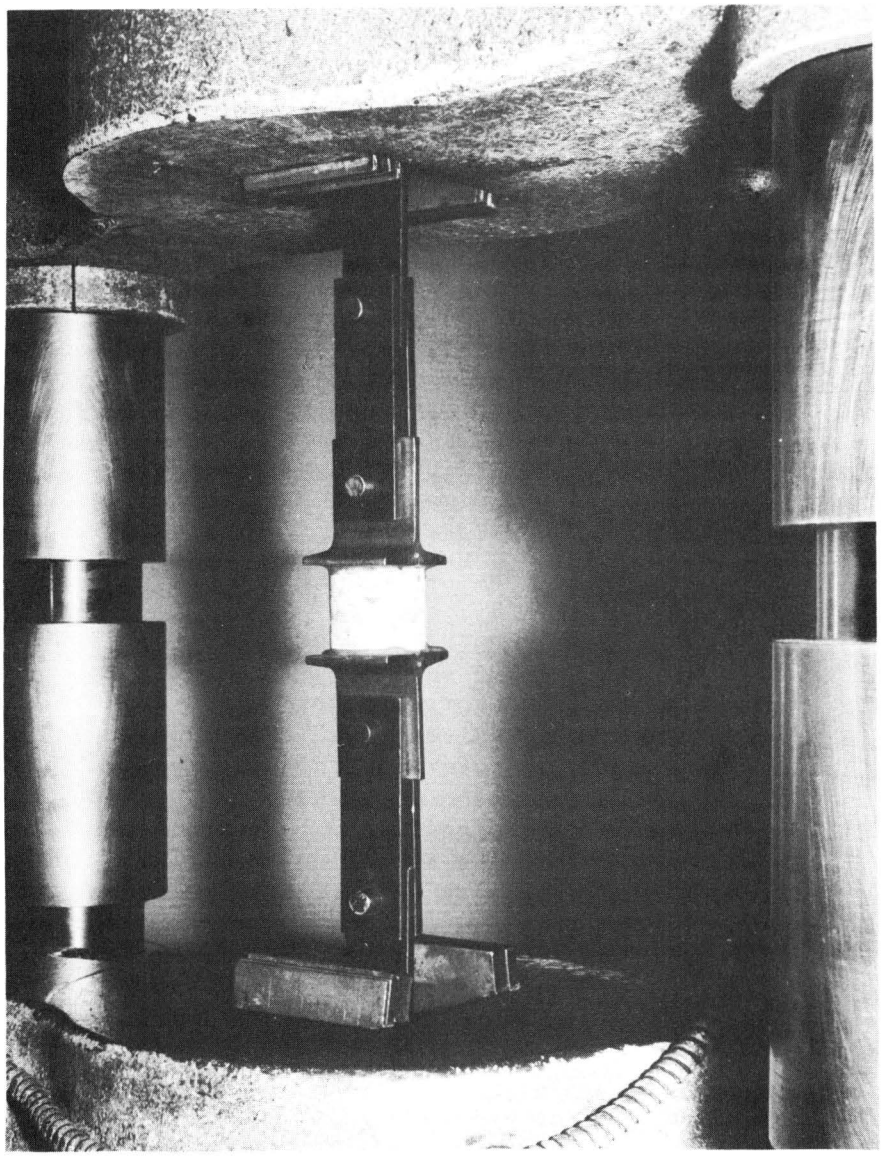

Figure 1
Core from Composite Slab
Being Loaded in Tension

In order to determine the aging and weathering characteristics of proposed epoxy binder formulations, two types of specimens were prepared. One of these specimens was in the form of a concrete block with a recessed area in the center, having ½ inch diameter reinforcing steel protruding from the concrete. The recessed area was filled with a mortar made from the epoxy

binder to be tested and a selected sand. A drawing of one of these specimens is shown below in Figure 2.

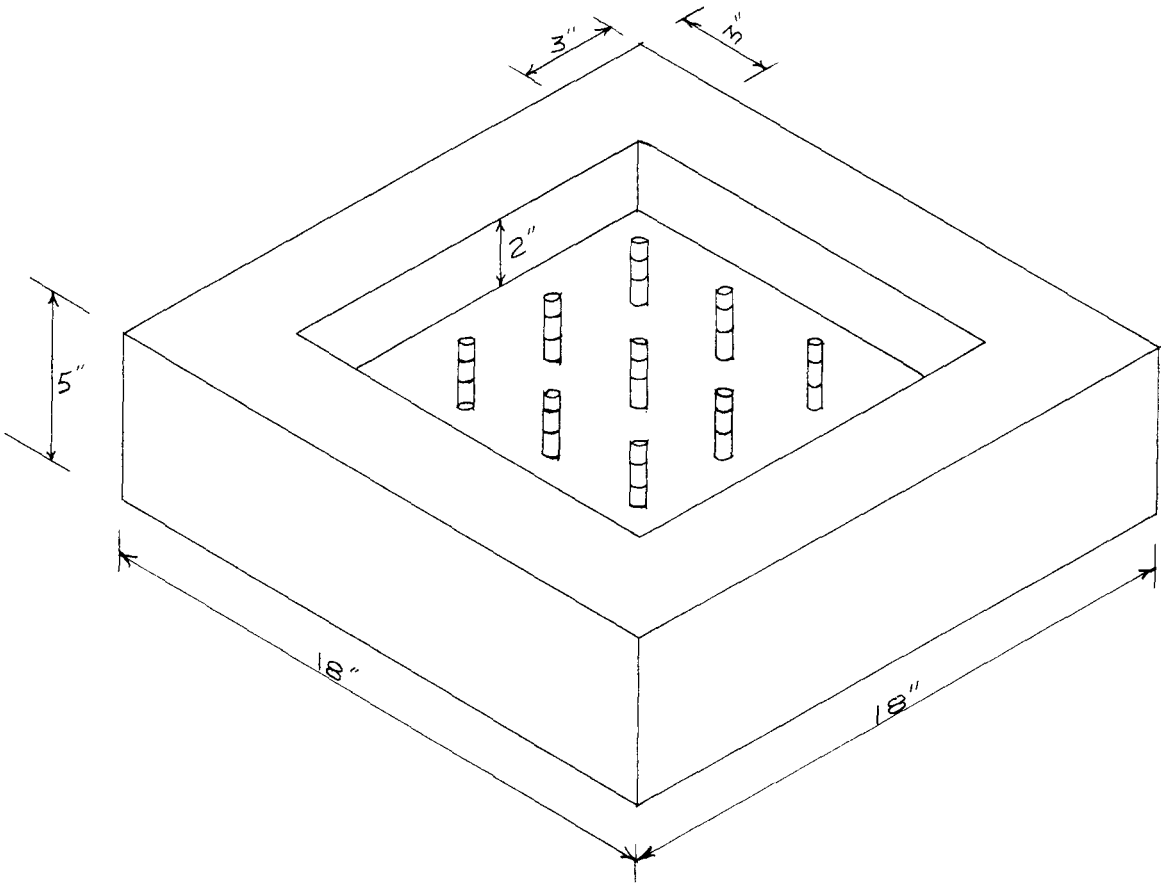

Figure 2
Schematic of Recessed Concrete Block
Used in Aging and Weathering Tests on
Epoxy Mortars

The specimen was prepared by pouring portland cement concrete mix into a rectangular form to a depth of approximately three inches. A two inch thick wooden block with nine pieces of ½ inch diameter steel reinforcing rod three inches in length fixed at intervals in the block was then placed on top of the concrete mix and the lengths of reinforcing steel forced down 1½ inches into the mix. After the block was placed, additional concrete mortar was added up to the top edge of the rectangular form.

After the concrete had attained a good initial set, the form and the block in the center were removed. The concrete specimens were kept moist for three days and then allowed to cure in air for approximately two weeks. At this time the recessed area and the exposed steel on each block were sandblasted. The concrete was sandblasted until all laitance was removed and the steel was sandblasted to white metal. Immediately following the sandblasting, the concrete and the exposed steel were primed with a portion of the epoxy binder under test. The remaining epoxy binder was mixed with aggregate to form an epoxy mortar. A ratio of seven parts by weight aggregate to one part by weight epoxy binder was used to make up each mortar. The aggregate was a silicious sand meeting the requirements of Texas Highway Department Standard Specification for Fine Aggregate, Grade No. 1, under Item 421, "Concrete for Structures". The grading was as follows:

U.S. Standard Screen No.	Percent Retained
4	0
8	5
16	30
50	89
100	97

The epoxy mortar was placed in the recessed area of the test specimens and compacted and troweled off to form the finished test specimen. The other type of specimen was prepared as follows:

Concrete blocks 12 inches square and 4 inches in thickness were cast and allowed to cure. Simulated pot holes were formed in these blocks by chipping our irregular areas up to one inch in depth and 4 to 8 inches in diameter. These simulated pot holes were patched with the same mortar that was placed in the recessed blocks. The surfaces of the pot holes were brushed out to remove any loose material and then were primed with the binder under test prior to placing the epoxy mortar. Placing of these epoxy mortars was done out of doors. The temperature at the time of placement was 80 to 90 degrees F. The temperature during the week following placement of the mortars ranged from a low of approximately 60 degrees F. to a high of approximately 90 degrees F. There was no precipitation during this week. Thus the epoxy mortars had a chance to cure under almost ideal weather conditions.

Both types of composite specimens were allowed to weather and age. They were examined at intervals to determine their condition.

As is discussed in Part II of this report, the coefficient of thermal expansion of an epoxy mortar is greater than that of portland cement concrete and therefore the epoxy mortar must have a fair degree of elasticity or flexibility in order to prevent the formation of undue stress as a result

of temperature changes in patched areas. An epoxy binder may initially have a satisfactory degree of flexibility or elasticity, but this property, along with the coefficient of thermal expansion, may change adversely with age. This could conceivably cause a mortar which initially performed satisfactorily to fail at a later date due to temperature stresses. In order to determine the effect of freeze-thaw cycling on epoxy mortars, composite specimens of portland cement mortar and epoxy mortar were prepared. The method of preparation was as follows:

Portland cement mortar blocks 12 inches long, 6 inches wide and 2 inches thick were obtained from the Bituminous Section of Materials and Tests. These blocks ordinarily are used in testing curing compounds. These blocks were etched with 10% hydrochloric acid on the top surface, rinsed thoroughly, and allowed to dry. A form which extended 3/4 inch above the top surface was placed around the blocks. The etched surfaces were then primed with a portion of the epoxy binder under test. The remainder of the binder was mixed with a silicious concrete sand having the following grading:

U.S. Standard Screen No.	Percent Retained
4	0
8	14
16	33
30	51
50	80
100	96

The ratio of aggregate to binder was seven to one by weight. The resulting epoxy mortar was placed on the surface of the mortar block, compacted and troweled off even with the top of the form. After the epoxy mortar had attained a good initial set, the forms were removed. The finished composite specimens consisted of a two inch thick portland cement mortar block with a 3/4 inch thick epoxy mortar overlay. These composite specimens were allowed to cure for a period of seven days at 70-80 degrees F. and were then subjected to a series of freeze-thaw and heat aging cycles. Figure 3 shows these blocks after the test was approximately two-thirds complete.
(See following page.)

Figure 3
Composite Blocks Used
in Freeze-Thaw Test

Note: Block designated as Epoxy #1 has overlay made up from Epoxy Binder B-102.
Block designated as Epoxy #3 has overlay made up from Formulation 10.

The tests used to determine the effect of elevated temperatures on the epoxies were as follows:

1. Adhesive Shear Test
2. Cleavage Test*
3. Water Absorption Test
4. Impact Test

*Cleavage test specimens were not prepared for the aging at 140 degrees F.

All four of these tests are described in Part I of this report. Epoxy Adhesives A-100, A-102, and A-103 and Epoxy Binders B-100, B-101, and B-102 were subjected to this test. Two complete sets of specimens for the above mentioned tests were prepared for each of these formulations. One set was used for aging at 140 degrees F. and the other set was used for aging at 180 degrees F. One-third of each set of specimens was tested after being cured for seven days at 70-80 degrees F. The remainder of the first set of specimens was placed in an oven maintained at 135-140 degrees F. Half of these specimens were tested after 30 days and the remainder after 60 days of heat aging. The second set of specimens was placed in an oven maintained at 175-185 degrees F. Half of these specimens were tested after 30 days aging and the remainder after 60 days of aging.

A presentation of the results obtained for the aging and weathering tests and the aging at elevated temperatures and a discussion of the results follows:

DISCUSSION

The epoxy adhesive formulations used to prepare composite briquettes to be subjected to aging and weathering are shown below. The abbreviation HVUR is used to designate high viscosity unmodified epoxy resin.

Formulation 1

100 pbw HVUR
50 pbw minus 325 mesh tabular alumina
5 pbw M-5 Cab-O-Sil
30 pbw Lancast A
3 pbw DMP-30

Formulation 2

100 pbw HVUR
75 pbw ASP 400 china clay
2.5 pbw M-5 Cab-O-Sil
50 pbw Lancast A
3 pbw DMP-30

Formulation 3

100 pbw HVUR
57 pbw minus 325 mesh tabular alumina
5 pbw titanium dioxide
5 pbw Bentone 27
75 pbw Thiokol LP-3
3.5 pbw DMP-30
3.5 pbw DMP-10

Formulation 4

Brand-name concrete adhesive-contained modified amine curing agent and a solvent

Formulation 5

Brand name concrete adhesive-probably cured with a modified amine

Formulation 6

Brand-name concrete adhesive-polyamine curing agent

Formulation 7

Epoxy Adhesive A-103

The results obtained for each of these formulations are presented in Table 1.

Table 1

Results of Weathering and Aging Tests on Composite Briquette Specimens

Formulation	Control Specimens		Specimens Subjected to Freeze-Thaw-Heat Cycling		Natural Weathering and Aging Specimens		
	Avg. Stress at Failure, PSI	Type of Failure	Avg. Stress at Failure, PSI	Type of Failure	Exposure Time, Months	Avg. Stress at Failure, PSI	Type of Failure
1	450	100% in mortar	415	100% in mortar	39	735	100% in mortar
2	440	100% in mortar	465	100% in mortar	39	725	100% in mortar
3	380	100% in mortar	510	100% in mortar	38	200	100% in mortar
4	260	100% in bond	315	50% in mortar	39	190	100% in bond
5	425	50% in mortar	545	75% in mortar	39	150	100% in mortar
6	375	100% in mortar	225	100% in bond	38	525	100% in bond
7	495	90% in mortar	480	100% in mortar	12	470	90% in mortar

The results obtained on the control specimens indicate that a good bond was obtained initially with all of these formulations except Formulation 4. This adhesive contained a considerable amount of solvent which might have interfered with the bond. The freeze-thaw-heat cycling did not adversely affect any of these formulations except No. 6. This material was fairly brittle and it is possible that the comparatively rapid temperature changes involved in the freeze-thaw-heat cycling caused a sufficiently rapid expansion and contraction in this material to cause it to pull away from the mortar. With respect to the aging and weathering specimens, Formulations 1, 2, 6 and 7 exhibited good retention of bond strength while Formulations 3, 4 and 5 exhibited bond failures. Formulations 1 and 2 were both cured with Lancast A, a modified amine curing agent. It is difficult to explain the high strengths of the aged mortar briquettes. Possibly the ratio of cement to sand was higher than the usual 1 to 3. Formulation 6 was a brand-name material the exact composition of which is unknown. According to the manufacturer, it

contained a polyamine (probably an aliphatic amine) as the curing agent. Formulation 3 was a polysulfide modified material. After the briquettes were broken the adhesive was examined and found to be rather soft and cheesy. These results indicated that the polysulfide modified epoxies might not have the aging and weathering characteristics which would be desirable. As has already been mentioned, Formulation 4 was a brand-name adhesive which contained a fairly large quantity of solvent. Both the control specimens and the aging and weathering specimens failed in the bond at approximately the same average loads. Formulation 5 had initially exhibited good bond strengths, but the aged specimens failed in the bond at low strengths indicating considerable deterioration. We had no specific information regarding the composition of this brand-name adhesive but it appeared to be an epoxy-modified amine system. These systems generally exhibited good weathering and aging properties. There was no explanation for the poor performance of this particular material. Soon after preparation of the original aging and weathering briquettes, the two composite slabs utilizing epoxy adhesives as the bonding agents were prepared. Formulation 8 was used on Slab #1 and Formulation 9 was used on Slab #2. The composition of these formulations was as follows:

Formulation 8*	Formulation 9
100 pbw modified epoxy resin	100 pbw modified epoxy resin
100 pbw minus 325 mesh tabular alumina	100 pbw minus 325 mesh tabular alumina
8 pbw M-5 Cab-O-Sil	5 pbw Bentone 27
1 pbw water	1 pbw 50% ethyl alcohol - 50% water
60 pbw Thiokol LP-3	60 pbw Thiokol LP-3
8 pbw DMP-30	8 pbw DMP-30

*The formulation used on Slab #1 was essentially the same as Epoxy Adhesive A-100.

Cores were taken from these slabs 10 days after placing the overlays to determine the initial bond obtained. In addition to these initial tests, cores were taken 2½ and 3½ years after placement of the overlays. The results of tests on these cores are presented in Table 2. For Slab #1, those cores taken from the acid etched portion are designated by "A" and those from the detergent washed portion are designated by "D".

Table 2

Results of Tensile Tests on Cores Taken from Epoxy-Bonded Composite Concrete Slabs

Slab from which Cores Taken	Age of Cores	Stress at Failure, PSI, Avg.	Type of Failure
1A	10 days	278	100% in concrete
1D	10 days	292	100% in concrete
2	10 days	341	100% in concrete
1A	2½ years	290	100% in concrete
1D	2½ years	306	100% in concrete
2	2½ years	368	80% in concrete
1A	3½ years	294	100% in concrete
1D	3½ years	267	100% in concrete
2	3½ years	371	100% in concrete

The test results on the initial set of cores indicated that a good bond was obtained between the cured and plastic portland cement concrete on both slabs. All the cores tested failed in the concrete. Unfortunately, the concrete in Composite Slab #1 did not have as high a tensile strength as would be desired. The strengths of the cores from Slab #2 more closely approximated the tensile strengths that would be expected for Class A concrete.

Of the total of 24 cores tested only one evidenced any failure in the bond. This core was taken from Slab #2 after 2½ years. The results indicate that after 3½ years, the bond between the layers of concrete on both slabs was at least as good as the tensile strength of the concrete itself. There was no evidence that the two types of surface preparation made any difference in the strength of the bond obtained.

The exposure of the briquettes had indicated that generally the modified amine cured epoxies had the best resistance to weathering and aging. The epoxy polysulfide adhesive exhibited considerable deterioration. The composite slabs involved only polysulfide systems, and cores taken from these slabs indicated that the epoxy was performing fairly well. The fact that the epoxy polysulfide systems used in preparing the composite slabs had not exhibited any extreme deterioration might be due to the difference in the severity of the tests. The

briquette test is the more severe. The edges of the one square inch bonded area are exposed to weathering and the epoxy bond is subjected to greater extremes of temperature. The epoxy bond in the cores taken from the composite slabs had not been subjected to any weathering on the edges of the bonded area. The comparatively large mass of the slabs provided an insulating effect thus reducing the temperature extremes and rate of temperature change to which the epoxy bond was subjected.

Referring now to the epoxy binders, outdoor exposure specimens were prepared from the following formulations.

Formulation 10	Formulation 11	Formulation 12
100 pbw HVUR	100 pbw HVUR	100 pbw HVUR
75 pbw Thiokol LP-3	50 pbw Lancast A	40 pbw Epi-Cure 855
10 pbw DMP-30	3 pbw DMP-30	10 pbw Epi-Cure 87

The portland cement concrete-epoxy mortar specimens were examined at intervals to determine if there was any apparent deterioration of the epoxy mortar. After slightly over a year, hairline separations between the epoxy mortar and the portland cement concrete were noted on those specimens prepared with Formulation 10 as the binder. The specimens prepared from Formulations 11 and 12 appeared sound.

After two years of exposure, these composite specimens were examined closely and then broken up with a sledge hammer. On those specimens prepared from Formulation 10, hairline cracks were present around all of the patches. When tapped with a hammer, the patches gave a hollow sound, indicating adhesion failure between the epoxy mortar and the concrete blocks. When the recessed blocks were broken up with the sledge hammer, the epoxy mortar separated from the concrete and the steel. Adhesion was very poor. The epoxy mortar appeared to contain moisture. The steel which had been imbedded in the epoxy mortar was corroded. The majority of the corrosion products were black, indicating that the corrosion was probably due to the polysulfide rather than to ordinary oxidation of the steel. When the concrete blocks with the artificially produced pot holes were broken up, the epoxy mortar patches came loose from the concrete in one piece indicating very poor adhesion.

Visual inspection of the specimens prepared from Formulations 11 and 12 indicated no hairline cracking at the edges of the epoxy mortar. Sounding with a hammer indicated that all the patches had maintained good adhesion to the concrete. The specimens were then broken up with a sledge hammer. The epoxy mortar adhered well to the concrete on all the specimens. The steel which had been imbedded in the epoxy mortar on the recessed specimens showed no evidence of corrosion. Adhesion of the mortar to the steel was very good.

These results indicated that modified amine cured epoxies such as Formulations 11 and 12 would perform better as binders than polysulfide systems. On the basis of these tests, use of Epoxy Binder B-100, which was basically the same composition as Formulation 10, was discontinued. The poor performance of the

epoxy-polysulfide mortar might have been due to several things. It was possible that the failure was due in part to the difference in thermal coefficients of the epoxy mortar and the concrete. The moisture which was present throughout the polysulfide mortar could have contributed to the failures.

In the original development of an epoxy binder, the mortars produced with polysulfide modified binders exhibited comparatively high water absorptions. Since the binders themselves did not have unusually high water gains, this is somewhat difficult to explain. It is possible that the epoxy-polysulfide binders did not wet the aggregates as well as the epoxy-modified amine systems. The tendency of the epoxy-polysulfide mortars to absorb moisture was not originally thought to be detrimental. However, the results of these tests indicate that although the epoxy-polysulfide mortars have many good properties initially, their ability to perform satisfactorily should have been questioned on the basis of their tendency to absorb moisture. In addition to the weathering and exposure specimens which were prepared, composite blocks of epoxy mortar and portland cement mortar were prepared as described on page 7. Specimens were prepared for Epoxy Binder B-102 and Formulation 10. These two formulations were used in order to compare the performance of an epoxy-modified amine system and an epoxy-polysulfide system. The composite blocks were subjected to 40 freeze-thaw cycles which consisted of 16 hours at 0 degrees F. and 8 hours at 75-80 degrees F. At the end of 40 cycles, there was no evidence of distress on either of the composite blocks. After being subjected to the freeze-thaw cycling, the blocks were aged at 135-140 degrees F. for 30 days. The aged blocks were then subjected to 20 additional freeze-thaw cycles. There was still no apparent degradation or loss of bond on either block. The blocks were then completely submerged in distilled water at 70-75 degrees F. for 24 hours, then placed in a freezer maintained at 0 degrees F. for 8 hours. The blocks were then removed from the freezer and submerged in 70-75 degree water for 8 hours. The specimens were subjected to 14 additional cycles of freezing for 4 to 8 hours and thawing in water for 8 hours. Hairline cracks began to appear in the portland cement mortar portion of both of these composite blocks after about 5 cycles. These cracks grew until they extended almost completely through the cement mortar portion of the blocks. The cement mortar also exhibited some scaling on the corners and sides. The epoxy mortar on both blocks did not appear to be affected by the wet freezing and thawing.

After completion of the 15 wet freeze-thaw cycles, each composite block was subjected to beam type loading until failure occurred. The blocks were supported on each end with the epoxy portion up and a load was applied directly to the center of the epoxy overlay. After the blocks had broken, they were examined. Epoxy Mortar #1, which was made up from Epoxy Binder B-102, did not appear to have been damaged by the freeze-thaw cycling and heat aging. The adhesion of the epoxy mortar to the cement mortar was very good. The epoxy mortar was perhaps a little more brittle than it had been originally. When the composite block made up with Epoxy Mortar #3 (Formulation 10 as the binder) was broken by flexural loading, the epoxy overlay partly separated

from the cement mortar, indicating some loss of adhesion. The epoxy mortar appeared to have softened and lost quite a bit of its strength. There was some moisture present in the epoxy mortar, although not as much as had been present in the same type of mortar on the outdoor exposure test specimens. The loss of adhesion between Epoxy Mortar #3 and the cement mortar block was not as pronounced as it was for the polysulfide mortar on the outdoor exposure test.

The results of this test indicate better performance by the epoxy-modified amine system tested compared to the epoxy-polysulfide system. The comparatively poor performance of the polysulfide modified system appeared to be due mainly to the change in physical properties with aging and possibly to the moisture absorbed by the mortar.

A limited number of mortar specimens prepared in the original evaluation of epoxy binders (Part II of this report) were retained in the laboratory at 70-80 degrees F. for a little over four years and then tested. A comparison of the results obtained is presented in Table 3. The original value obtained for each property is given first and the values obtained on the aged specimens presented on the second line. The formulation numbers are those used in Part II of this report. The composition of these formulations is given in Table 6 on page 13 of Part II.

Table 3

Comparison of Original With Aged Properties of Epoxy Mortars

Property	Formulation Number 4	5	6	7
Tensile Strength, PSI	1130	1040	1270	1700
		1170	875	945
Compressive Strength, PSI	7375	8900	7010	8225
	8210		8210	6740
Modulus of Elasticity, Compressive, x 10^6	0.84	0.90	0.62	0.31
	1.00		1.91	0.68
Thermal Coefficient, In./In./Degree F. x 10^{-6} (0 to 80 Degrees F. Range)	9.3	10.2	10.5	13.3
	10.5	10.4	10.7	15.7
Thermal Coefficient, In./In./Degree F. x 10^{-6} (80 to 160 Degrees F. Range)	15.2	14.0	18.8	19.2
	15.9	15.1	21.2	22.6

Note: Upper number in each case is original test value.

The greatest change in properties were exhibited by Formulations 6 and 7, both polysulfide modified materials. Formulation 6 had over a 300 per cent increase in the modulus of elasticity, which indicates considerable loss of flexibility. This formulation also showed a decrease in tensile strength and an increase in compressive strength. The thermal coefficients for this formulation increased, which, in combination with the loss of flexibility, could result in considerable thermal stresses in patches on concrete pavement or bridge decks. Formulation 7 decreased considerably in tensile and compressive strength. The compressive modulus was greater on the aged mortar, which was the opposite of what was expected in view of the fact that the aged tensile specimens exhibited considerable elongation before failure occurred. The thermal coefficients for Formulation 7 showed an increase over both temperature ranges.

The results of the tests performed on these aged specimens indicate that all of these formulations change in properties with age. The changes in the formulations containing the polysulfide were the most significant. After aging, the performance of the mortars prepared from Formulations 6 and 7 was much less satisfactory than the performance of the mortars prepared from Formulations 4 and 5.

The results of the heat aging tests described in the procedure are given in Tables 4 and 5. Table 4 presents the results obtained for the specimens aged at 135-140 degrees F. The first values given for each test are the results obtained for the control specimens. The second values are the results after 30 days of aging and the third values are the results after 60 days of aging. Table 5 presents the results obtained for the specimens aged at 175-185 degrees F. The first values given for each test are the results obtained for the control specimens. The second and third values are the results after 30 and 60 days of aging respectively.

Table 4

Effect of Aging at 135-140 Degrees F. on Epoxy Formulations

	Formulation					
Property	Epoxy Adhesive A-100	Epoxy Adhesive A-102	Epoxy Adhesive A-103	Epoxy Binder B-100	Epoxy Binder B-101	Epoxy Binder B-102
Adhesive Shear	3172	3354	3452	3397	2596	2504
Strength,	2144	3332	3399	2042	2728	2673
PSI, Avg.	1676	2952	3261	1676	2292	2812
Water Gain,	0.18	0.08	0.09	0.26	0.07	0.07
Percent by Wt.,	0.14	0.11	0.13	0.20	0.06	0.08
Avg.	0.12	0.06	0.07	0.17	0.06	0.06
Impact Strength,	9.0*	5.8	6.0	9.0*	6.5	6.0
Ft.-Lbs., Avg.	9.0*	8.5	6.2	9.0*	7.8	6.5
	9.0*	5.5	5.5	9.0*	6.0	5.2

*Limit of test equipment without failure occurring.

Table 5

Effect of Aging at 175-185 Degrees F. on Epoxy Formulations

Property	Formulation: Epoxy Adhesive A-100	Epoxy Adhesive A-102	Epoxy Adhesive A-103	Epoxy Binder B-100	Epoxy Binder B-101	Epoxy Binder B-102
Adhesive Shear Strength, PSI, Avg.	3143	2882	3010	3376	2546	2636
	1615	3312	3400	1673	3470	3502
	1704	3354	3726	1702	3178	3618
Cleavage Strength, PSI, Avg.	1350	1740	1710	1230	1465	1590
	760	1810	1910	1220	1705	1645
	885	1870	1835	1215	1765	1675
Water Gain, Percent by Wt., Avg.	0.22	0.20	0.14	0.30	0.08	0.14
	0.16	0.14	0.16	0.18	0.10	0.16
	0.14	0.20	0.15	0.18	0.08	0.18
Impact Strength, Ft.-Lbs., Avg.	9.0	6.2	5.8	9.0*	6.5	6.5
	7.5	7.0	6.8	7.2	7.2	6.2
	6.5	5.5	5.8	5.8	5.8	5.8

*Limit of test equipment without failure occurring.

The results presented in Tables 4 and 5 indicate that definite changes in properties of these epoxy formulations occur at elevated temperatures. At both 140 and 180 degrees F., the epoxy-polysulfide formulations (Epoxy Adhesive A-100 and Epoxy Binder B-100) showed a rapid drop in strengths after 30 days. Additional aging at 140 degrees F. resulted in little additional change in properties. Additional aging at 180 degrees F. produced no significant change in any property except the impact strength, which continued to decrease. Epoxy Binder B-100 was unique in that its cleavage strength did not vary upon aging at 180 degrees F. The water gain values for these two formulations decreased as a result of the aging at elevated temperatures.

The epoxy formulations cured with modified amines or combinations of modified amines and polyamides (Epoxy Adhesives A-102 and A-103 and Epoxy Binders B-101 and B-102) reacted similarly to the aging at elevated temperatures. There was little change in the properties of these four materials after 30 and 60 days at 140 degrees F. The impact strength followed a pattern of slight improvement after 30 days and then a decline to less than the original impact strength after 60 days. This indicated some embrittlement was taking

place. When aged at 180 degrees F., the adhesive shear and cleavage strengths of these four formulations tended to increase. The water gains did not change significantly. The impact strengths followed the same pattern set at 140 degrees F.

In view of the fact that concrete roadways and bridge decks approach temperatures of 135-140 degrees F. in Texas during the summer months, the changes noted at this temperature in the laboratory could be expected to take place in actual use. The aging at 180 degrees F. is of interest and could possibly be regarded as an indication of resistance to actual aging. There is apparently little information available regarding aging of epoxies and correlation of accelerated aging with actual aging. No attempt was made to derive a direct correlation between aging at 180 degrees F. and the actual aging of these epoxies.

The performance of Epoxy Adhesive A-100 and Binder B-100 indicates that considerable change in physical properties could be expected when these materials are exposed to temperatures of 135 degrees F. or greater. The loss in strengths is sufficiently large that the performance of these polysulfide formulations could easily be affected. The performance of Epoxy Adhesives A-102 and A-103 and Epoxy Binder B-101 and B-102 in this test indicates that some change in properties, particularly loss of flexibility, could be expected as a result of prolonged exposure to temperatures of 135 degrees F. or greater. These changes occur at a comparatively slow rate which would indicate that these materials should have a longer service life than the epoxy-polysulfide systems.

This investigation of the weathering and aging properties of epoxy materials was rather limited, but we feel that the information derived from these tests has helped us to provide better materials for highway use. The performance of these epoxy materials in actual use over a period of time will provide the best indication of their ability to resist aging and weathering changes.

APPENDIX

SPECIFICATION FOR EPOXY ADHESIVE A-103

The following specification item governs the basic materials and the composition, manufacture, and testing of the finished product for Epoxy Adhesive A-103 which is designed mainly for the following uses:

1. Bonding fresh portland cement concrete to existing portland cement concrete.

2. Bonding cured concrete to existing concrete structures.

3. Bonding steel to fresh or hardened concrete.

Raw Materials

The basic raw materials to be incorporated into this binder are listed on the following pages along with the specific requirements for each material. Prior to manufacture of this material, the contractor shall inform the Highway Department as to the exact brands of raw materials which he proposes to use. The final decision as to equality of materials shall be made by the Highway Department. After the Highway Department has approved the brand names of raw materials proposed by the contractor, no substitution will be allowed during the manufacture without prior approval of the Highway Department.

Epoxy Resin

The basic epoxy resin used in this formulation shall be an unmodified liquid resin conforming to the following chemical and physical requirements:

Viscosity at 25.0 $\pm$ 0.1 degrees C., cps. - - - - - - 7,000 to 10,000

Weight per epoxy equivalent, gms. per gm.-mole - - - 175 to 195

Color (Gardner Number), Max. - - - - - - - - - - - - - 5

Hydrolyzable chlorine, Max. per cent by weight - - - 0.2

Specific gravity, 25/25 degrees C. - - - - - - - - - - 1.14 to 1.18

Test methods to be used in determining these qualities are listed below:

1. Viscosity - Method of Test for Kinematic Viscosity (ASTM D445-60).

2. Weight per Epoxy Equivalent - Method of Test for Epoxy Content of Epoxy Resins (ASTM Designation D1652-59T).

3. Color - Method of Test for Color of Transparent Liquids (Gardner Color Scale) (ASTM Designation D1544-58T).

4. Hydrolyzable Chlorine - Method of Test for Hydrolyzable Chlorine Content of Liquid Epoxy Resins (ASTM Designation D1726-60T).

5. Specific Gravity - Method of Test for Density of Paint, Varnish, Lacquer, and Related Products (ASTM Designation D1475-57T).

Curing Agents or Hardeners

Modified Amido-Amine Curing Agent

This curing agent shall be Genamid 2000, marketed by the Chemical Division of General Mills, Inc. The detailed requirements for this curing agent are as follows:

Viscosity at 77 degrees F., cps. - - - - - - - - - - - 1500 to 2100

Specific Gravity, 20/20 degrees C. - - - - - - - - - - 0.97 to 0.99

Amine Value - 575 to 625

Test methods to be used in determining these qualities are listed below:

1. Viscosity - Tests for Varnishes (ASTM Designation D154-58)

2. Specific Gravity - Method of Test for Density of Paint, Varnish, Lacquer, and Related Products (ASTM Designation D1475-57T).

3. Amine Value - Method of Test for Total Amine Values of Fatty Amines by Referee Potentiometric Method (ASTM D2073-62T).

Amido-Amine Curing Agent

This curing agent shall be one of the following:

Araldite 9130, marketed by CIBA Products Corp.
Epi-Cure 855, marketed by Jones-Dabney Co., Division of Devoe & Raynolds Co., Inc.

The detailed requirements for this curing agent are as follows:

Viscosity at 77 degrees F., cps. - - - - - - - - - - 150 to 400

Specific Gravity, 20/20 degrees C. - - - - - - - - - 0.94 to 0.96

Flash Point, degrees F.,
Cleveland Open Cup - - - - - - - - - - - - - - - - - - 440 to 460

Test methods to be used in determining these qualities are listed below:

1. Viscosity - Tests for Varnishes (ASTM Designation D154-58).

2. Specific Gravity - Method of Test for Density of Paint, Varnish, Lacquer, and Related Products (ASTM Designation D1475-57T).

3. Flash Point - Flash and Fire Points by Cleveland Open Cup (ASTM Designation D92-57).

Mineral Filler

The mineral filler used in this formulation shall be a minus 325 mesh silica flour equivalent to that produced by Pennsylvania Glass Sand Corporation. Specific requirements are as follows:

SiO_2 Content	99.0% by Wt. Minimum
Particle Size	95% by Wt. Minus 325 Mesh (Tyler Standard Screen)

Gelling Agent

The gelling or thixotropic agent used in this formulation shall be Cab-O-Sil, grade M-5 colloidal silica, manufactured by Godfrey L. Cabot, Inc. The colloidal silica shall exhibit the following chemical and physical properties:

Silica Content (Moisture-free basis)	99.0% by wt. Minimum
Free Moisture (1 hr. at 105 degrees C.)	1.5% by wt. Maximum
Ignition Loss (Dry basis - ½ hr. at 1000 degrees C.)	1.0% by wt. Maximum
Particle Size Range	0.015 - 0.020 Micron
Surface Area (Nitrogen absorption)	190 ± 15 Sq. Meters/Gm.
pH(10% aqueous dispersion)	3.6 - 4.2
Oil Absorption (Gardner Method) ASTM Designation D1483-57T	150 lbs. oil/100 lbs. pigment
Bulking Value	0.057 Gal./lb.
Apparent Bulk Density	4.5 lb./cu. ft.

Compounding of the Components of Epoxy Adhesive A-103

The components of the epoxy adhesive shall be compounded from the materials previously specified according to the following formulas.

Resin Component

The resin component shall consist of epoxy resin only packaged according to specification requirements.

Hardener Component

The hardener component of the epoxy adhesive shall contain the following materials in the ratio shown:

37 parts by weight Genamid 2000

18 parts by weight Epi-Cure 855 or Araldite 9130

51 parts by weight minus 325 mesh silica flour

4 parts by weight Cab-O-Sil

The method of mixing these materials to form the hardener component shall be as desired by the manufacturer so long as the finished product complies with the physical requirements stated in this specification. However, the following point should be considered:

The temperature of the hardener mixture should not be allowed above approximately 140 degrees F. for any length of time during mixing as this would result in decomposition and vaporization of the curing agents with a resultant change in the physical properties of the finished product.

Packaging

Shipment shall be made in suitable, strong, well sealed containers which not only meet specification and ICC requirements but also are sufficiently sturdy to withstand the normal handling to which shipments are subjected in transit. The size and type of containers and the amount of material to be placed in each container shall be as specified on the bid invitation and material requisition.

Labeling

The finished epoxy adhesive containers and cases shall be plainly and securely labeled. The hardener component shall have the following instructions on the label:

Texas Highway Department Epoxy Adhesive A-103

This material is intended to be used mainly for bonding fresh

portland cement concrete to existing portland cement concrete, bonding cured concrete to existing concrete structures, and bonding steel to fresh or hardened concrete. Note: For detailed instructions see D-9 pamphlet entitled Instructions Regarding the Use of Epoxy Adhesive A-103.

Add the resin component to the can containing the hardener component and mix thoroughly, preferably with a small mechanical stirrer. If material is to be mixed by hand, gradually add the resin component to the hardener component while stirring. If less than entire unit is needed, the two components may be mixed in the following ratio:

1 part resin component by volume
1 part hardener component by volume
or
1.0 part resin component by weight
1.1 parts hardener component by weight

Caution - Do not attempt to thin epoxy adhesive with any type of solvent. Use toluene or bead binder thinner for clean-up of equipment. Do not store at temperatures below 60 degrees F. or above 100 degrees F.

WARNING

May cause skin irritation. Avoid contact with skin, eyes and clothing. In case of contact with skin or clothing, wash skin immediately and thoroughly with soap and water. Remove and wash clothing before re-use. For eyes, flush with plenty of water; get medical attention.

Each container of hardener component shall also have the following information on the label:

HARDENER COMPONENT
Batch Number
Order Number
Date of manufacture
Gross weight
Net weight
Name of manufacturer

The resin component shall have the following information on the label:

Texas Highway Department Epoxy Adhesive A-103
RESIN COMPONENT
Batch Number
Order Number
Date of manufacture
Gross weight
Net weight
Name of manufacturer

Each container of resin component also shall have the following storage and handling precautions on the label:

Do not store at temperatures below 60 degrees F. or above 100 degrees F.

WARNING

May cause skin irritation. Avoid contact with skin, eyes and clothing. In case of contact with skin or clothing, wash skin immediately and thoroughly with soap and water. Remove and wash clothing before re-use. For eyes, flush with plenty of water; get medical attention.

Physical Requirements for Epoxy Adhesive A-103

Resin - Hardener Mixture

The resin-hardener mixture shall consist of one part by weight resin component and 1.1 parts by weight hardener component. The mixture must meet the following requirements:

Pot Life at 77 Degrees F. - 40 Minutes Minimum
54 Minutes Maximum

Samples of each of the adhesive components shall be brought to 77 $\pm$ 2 degrees F. Then 47 grams of the resin component and 52 grams of the hardener component shall be weighed into a 9 ounce unwaxed paper cup. The time shall be recorded and the two components mixed for three minutes, taking care to periodically scrape the walls and bottom of the cup. The cup shall then be set on a wooden bench top and probed every two minutes with a stirring rod, starting 30 minutes from the time of mixing. The time at which gelled material first forms in the container is recorded as the pot life.

Thixotrophy - The degree of thixotrophy shall be determined as described below. (The ambient temperature and the temperature of the materials used in this test shall be 77 $\pm$ 2 degrees F.)

The two components of the epoxy adhesive shall be stirred together for approximately 5 minutes and then applied to a smooth clean steel plate to form a panel of epoxy material 2 inches wide, 4 inches in length, and 0.10 inch (100 mils) in thickness. A removable form of the proper dimensions may be used in placing the epoxy on the steel plate. The epoxy may be poured into the form and the excess struck off level with the top edge and then the form removed. Immediately after forming the epoxy material, the steel panel shall be placed in a vertical position, the 4 inch dimension of the epoxy panel perpendicular to the horizontal. Not more than 7 minutes shall elapse between the initiation of mixing and the placing of the steel panel in the vertical position.

Requirements - The adhesive must be sufficiently resistant to flow that an average thickness of 0.050 inch (50 mils) of cured material will remain on the test panel.

Physical Requirements for the Cured Adhesive*

Adhesive Shear Strength - Steel to Steel - (ASTM -D1002-53T) - 2800 PSI Minimum

The surfaces of the test specimens used in the adhesive shear strength test shall be prepared by blasting to white metal. The blasted surfaces shall be washed with methyl ethyl ketone and allowed to dry before applying the adhesive. The surface of the test specimens shall have a prepared surface of equivalent "anchor pattern" to that which would be obtained by abrasive blasting the surfaces of the test specimens with a gun pressure of 50 to 75 psi using a ¼ inch diameter nozzle and employing Garnet Blasting Abrasive "Gem Blast", 60 mesh (No. 45 to No. 74 U.S. Standard Screens), as marketed by Clemtex, Inc., of P. O. Box 15214, Houston 20, Texas.

Cleavage Strength - Steel to Steel (ASTM-D1062-51) - 1200 PSI Minimum.

Surface preparation shall be as outlined in Adhesive Shear Strength.

Water Gain - 24 hour immersion at 23 degrees C. - (ASTM D-570-57T) - 0.20 per cent by weight Maximum

Ability to Bond Fresh Portland Cement Concrete to Cured Portland Cement Concrete

For this test, the equipment and materials used as specified in ASTM C 190-59 (Tensile Strength of Hydraulic Cement Mortars)

The procedure is as follows:

Ordinary tensile briquettes shall be prepared according to the method prescribed in the above mentioned ASTM procedure. These briquettes shall be prepared using a ratio of 3 parts sand to 1 part high-early strength cement by weight. These briquettes shall be cured for a minimum of 7 days and then broken, using a Riehle briquette tester. Any of the briquettes which break at a load of less than 450 pounds shall be rejected. The remaining broken halves shall be allowed to dry and may then be used in preparing the test specimens involving the epoxy adhesive. The sections of broken briquettes are coated on the broken area with the epoxy adhesive. After this material has become tacky, new concrete shall be molded up against it to form a complete briquette. The resulting briquettes are then cured according to the method set forth in ASTM C 190-59. A minimum of six briquettes shall be prepared - three to be tested after three days cure time and three to be tested after seven days cure time.

*The adhesive shear strength, cleavage strength, and water gain are to be determined on material that has cured for seven days at 70 - 80 degrees F.

Requirements - None of the briquettes tested shall evidence separation in the bonding material. Three-day briquettes shall show an average strength of 350 pounds minimum. Seven-day briquettes shall show an average strength of 400 pounds minimum. If the briquettes tested break in the concrete at average values below those required, additional test specimens shall be prepared.

SAMPLING AND TESTING

A. Materials to be tested. The epoxy adhesive purchased by the Highway Department under this specification shall be tested for conformance to said specification.

B. Agency. All tests on finished products and raw materials, as well as inspection during manufacture will be made by the Texas Highway Department or by a commercial laboratory designated by the Highway Department.

C. Inspection and Testing Expenses. The cost of inspection and testing of this material will be borne by the Highway Department. The manufacturer will be expected to bear the actual cost of the materials and finished adhesive taken as samples at the time of manufacture. The size of the samples taken will be no larger than is necessary for testing purposes.

D. Methods.

1. Sequence of Inspection

a. Immediately after the contract has been awarded, the supplier will contact the Materials and Tests Engineer, Texas Highway Department, Austin, Texas, regarding brand names and characteristics of all raw materials which the contractor proposes to use; and to make arrangements for inspection during production.

b. Manufacture shall be witnessed in whole or in part depending upon the discretion of the testing agency. Production will not begin prior to the arrival of the Highway Department inspector unless prior specific approval for such starting has been obtained. Samples of raw materials actually used in production and samples of the adhesive will be taken during production. The manufacturer shall accord the inspector free access to those parts of the plant wherein the adhesive is being manufactured or raw materials are being stored, and in all other ways shall facilitate the inspection and sampling process. Any questions regarding inspection or testing should be addressed to the Materials and Tests Engineer, Texas Highway Department, Austin, Texas.

2. Basis for Rejection

The finished epoxy adhesive and any of the materials used in the adhesive which fail to meet any or all requirements of this specification shall be subject to rejection. Final acceptance or rejection shall be based on results of tests on samples of raw materials, adhesive components, and the mixture of the two components taken during production and upon tests made on the adhesive components and the combination of the two components after the adhesive has arrived at the shipping destination. Approval of materials, as a result of preliminary testing prior to manufacture into the finished adhesive, shall not be binding upon final approval or rejection. Because of the possibility of contamination and volatile losses, it shall be agreed that the resin and hardener reference standard currently in the possession of the Texas Highway Department or its authorized testing agencies, shall constitute standards for final comparison involving acceptance or rejection. Samples of these reference standards are available to the manufacturer. The judgment of the Highway Department's Materials and Tests Engineer shall be final in all questions relative to conformance with the provisions of this specification.

INSTRUCTIONS FOR USE OF TEXAS HIGHWAY DEPARTMENT EPOXY ADHESIVE A-103

Materials and Tests Division

Epoxy Adhesive A-103 has been designed to be as near as possible a general epoxy adhesive for highway use. It may be used for bonding fresh portland cement concrete to existing concrete, bonding cured concrete to existing concrete, bonding steel to fresh or hardened concrete, and in some cases, bonding steel to steel. It may be used either on vertical or horizontal surfaces. This adhesive has been designed to be applied by brush, squeegee, roller, or some similar means of application. It is currently available in 3/4 gallon and 1½ gallon units.

Instructions concerning the use of this material are given on the following pages. It should be kept in mind that this material will perform satisfactorily only if it is used properly.

General Instructions

Handling Precautions

The epoxy materials are capable of causing irritation or other physiological reaction in some individuals. For this reason, care should be exercised in handling this material. If a workman should develop a skin rash or similar reaction while working with the epoxy, it would be best for him to discontinue handling this material, as continued contact with the epoxy will result in greater irritation or reaction.

Mixing

The two components of this material are to be mixed in the following ratio:

1 part resin component by volume
1 part hardener component by volume

or

1 part resin component by weight
1.1 part hardener component by weight

The two components must be thoroughly mixed together, preferably with a small mechanical mixer. If a mechanical mixer is not available, the adhesive may be mixed by hand. The resin component should be added in small amounts to the hardener component and the mixture stirred well after each addition. Adequate mixing will require 5 to 10 minutes. No more than approximately 1½ gallons of finished adhesive should be prepared in a single batch. The reaction of the resin and hardener components generates heat and in a large batch this heat is trapped and cannot be given off as rapidly as it is generated. This results in a build-up of heat which causes the reaction to proceed at a more rapid rate thus shortening the working time.

Working Time

After the two components have been mixed, one unit of the adhesive will retain good working properties for about 20 minutes. The adhesive will then begin to thicken. This time is based on the initial temperature of the two components and the ambient temperature being approximately 80 degrees F. If the initial temperature of the two components or the ambient temperature is less than 80 degrees F., the working time will be somewhat longer. If the initial temperature of the two components or the ambient temperature is greater than 80 degrees F., the working time will be somewhat shorter than that shown. This material should not be used if the ambient temperature is below 60 degrees F.

The adhesive will remain workable for a longer period of time if it is removed from the container and spread over the surface to be coated immediately after mixing. If the material is allowed to remain in the container after mixing, the heat from the reaction between the hardener and the resin will build up and cause the adhesive to thicken more rapidly.

Coverage

For best results, enough Epoxy Adhesive A-103 should be applied to the surfaces to be bonded at least 0.02 inch (20 mils) thick. One 3/4 gallon unit of this material will cover approximately 50 square feet if applied to this thickness. If the adhesive is being applied to a rough surface, a heavier coat may be necessary.

Clean-up

Any equipment used in applying the adhesive must be cleaned before the adhesive hardens. Toluene is recommended as a solvent for removing the epoxy material. BBT-9 Bead Binder Thinner may also be used for this purpose if it is more readily available. Both of these solvents are stocked by the Highway Department regional warehouses in 5 gallon containers. The stock numbers are as follows:

307700 - - - - Thinner, BBT-9 Bead Binder
307800 - - - - Toluene

It should be kept in mind that these solvents are quite flammable and should be used with caution.

Specific Applications

I. Bonding fresh portland cement concrete to existing portland cement concrete.

A. Surface Preparation
The existing concrete surface should be free of any loose or unsound concrete. After this has been assured, the surface may be prepared in one of several ways. These methods of surface preparation are described below.

1. Sandblasting
The area to be coated should be blasted sufficiently to remove any

laitence or road film present and provide a roughened surface. Before applying the adhesive, see that the surface is free from loose fines.

2. Acid etching
Acid etching is generally used as an alternate to sandblasting for removing laitence and road film and providing a roughened surface. The concrete to which the adhesive is to be applied may be treated with a solution of 2 parts water and 1 part 20 degree Baume' muriatic (commercial hydrochloric) acid by volume. The amount of acid required is approximately one gallon per square yards. The acid should be spread over the surface and scrubbed in with a broom or brush. After the reaction between the acid and the concrete has taken place, the surface should be thoroughly washed to remove the salts formed in the reaction. In most cases, acid etching will provide a surface to which the epoxy will adhere quite well. However, there are several problems involved in the use of acid. The personnel engaged in working with the acid must exercise care in order to prevent burns and damage to clothing. Goggles and rubber gloves should be supplied the personnel applying the acid. Hydrochloric acid gives off noxious fumes which in some cases may present a hazard to the personnel. Any equipment used in the operation must be protected from the hydrochloric acid due to its extreme corrosiveness.

3. Detergent Washing
If the concrete to which the adhesive is to be applied does not have an extreme amount of laitence or road film present, washing with a detergent followed by rinsing with plain water may be sufficient surface preparation. The concrete surface should be scrubbed by broom or brush with the detergent solution and then thoroughly rinsed.

All three of the above methods of surface preparation have been used successfully. In some cases, where the adhesive is to be applied to a concrete surface which has been jackhammered to expose sound relatively clean concrete, all that may be needed in the way of surface preparation is pressure washing with water. In all cases where the concrete has been washed, the epoxy adhesive should not be applied until the surface is reasonably dry.

B. Application
After the existing concrete has been prepared, the mixed adhesive may be applied. The adhesive should be allowed to become tacky before pouring the fresh concrete against it. This is essential to obtaining a good bond. The time required, after mixing, for the adhesive to become tacky will vary with temperature. The following chart gives, for several different ambient temperatures, the minimum and maximum time which may elapse between mixing the epoxy adhesive and pouring the fresh concrete against the epoxy.

Temperature, Degrees F.	Time Between Mixing of Epoxy and Pouring of Fresh Concrete Minimum	Maximum
60	45 Minutes	3 Hours
70	30 "	2½ "
80	15 "	2 "
90	15 "	1½ "
100	15 "	1 Hour

The fresh concrete which is to be bonded to existing concrete should be a slow slump workable mix. The presence of excessive water in the fresh concrete will hinder the development of a good bond. The fresh concrete may be worked in the usual manner after it is poured in place.

II. Bonding cured concrete to existing concrete.
In this case, both of the surfaces to be bonded should be prepared in one of the ways previously described. A thin coating of the adhesive (approximately 10 mils) may be applied to each surface and the coated surfaces then joined together.

III. Bonding steel to concrete.
Once again, the concrete surface should be prepared either by sandblasting, acid etching or detergent washing. For best results, the surface of the steel which is to be bonded should be sandblasted to white metal in order to obtain a clean rough surface which will provide maximum adhesion. The steel surface must be free of any oil or grease. The steel should be degreased with toluene if there is any evidence of oil or grease present.

IV. Bonding steel to steel.
In cases where there is no need for high structural strengths, Epoxy Adhesive A-103 may be used to join steel to steel. All surfaces should be sandblasted and brushed free of dust and fines and then coated with approximately 10 mils of the adhesive. All surfaces to be joined must be free of oil or grease. If any oil or grease is present, degrease the surfaces with toluene.

Specification for Epoxy Binder B-102

The following specification item governs the basic materials and the composition, manufacture, and testing of the finished product for Epoxy Binder B-102. This material is to be mixed with various types of aggregate to form an epoxy mortar or concrete to be used in the repair of portland cement concrete structures. This type of material is intended to be used where high strength, quick set, and a good bond to the existing concrete is desired.

Raw Materials

The basic raw materials to be incorporated into this binder are listed on the following pages along with the specific requirements for each material. Prior to manufacture of this material, the contractor shall inform the Highway Department as to the exact brands of raw materials which he proposes to use. The final decision as to equality of materials shall be made by the Highway Department. After the Highway Department has approved the brand names of raw materials proposed by the contractor, no substitution will be allowed during the manufacture without prior approval of the Highway Department.

Epoxy Resin

The basic epoxy resin used in this formulation shall be an unmodified liquid resin conforming to the following chemical and physical requirements:

Viscosity at 25.0 $\pm$ 0.1 degrees C., cps. - - - - - - - - 7,000 to 10,000

Weight per epoxy equivalent, gms. per gm. - mole - - - 175 to 195

Color (Gardner Number), Max. - - - - - - - - - - - - - - - 5

Hydrolyzable chlorine, max. per cent by weight - - - - 0.2

Specific gravity, 25/25 degrees C. - - - - - - - - - - - 1.14 to 1.18

Test methods to be used in determining these qualities are listed below:

1. Viscosity - Method of Test for Kinematic Viscosity (ASTM D445-60).

2. Weight per Epoxy Equivalent - Method of Test for Epoxy Content of Epoxy Resins (ASTM Designation D1652-59T).

3. Color Method of Test for Color of Transparent Liquids (Gardner Color Scale) (ASTM Designation D1544-58T).

4. Hydrolyzable Chlorine - Method of Test for Hydrolyzable Chlorine Content of Liquid Epoxy Resins (ASTM Designation D1726-60T).

5. Specific Gravity - Method of Test for Density of Paint, Varnish, Lacquer, and Related Products (ASTM Designation D1475-57T).

Curing Agents or Hardeners

Modified Amido-Amine Curing Agent

This curing agent shall be one of the following:

Araldite H-955, marketed by CIBA Products Company
Epi-Cure 872, marketed by Jones-Dabney Co., Division of Devoe & Reynolds Co.,Inc.

The detailed requirements for this curing agent are as follows:

Viscosity at 77 degrees F., cps. - - - - - - 500 to 900

Specific Gravity, 20/20 degrees C. - - - - - 0.97 to 0.99

Flash Point, degrees F.,
Cleveland Open Cup - - - - - - - - - - - - - 290 to 300

Test methods to be used in determining these qualities are listed below:

1. Viscosity - Tests for Varnishes (ASTM Designation D154-53)

2. Specific Gravity - Tests for Varnishes (ASTM Designation D154-53)

3. Flash Point - Flash and Fire Points by Cleveland Open Cup (ASTM Designation D92-57)

Polyamide Curing Agent

This curing agent shall be one of the following:

DEH 14, marketed by Dow Chemical Co.
Epon V-40, marketed by Shell Chemical Co.
Pentamid 840, marketed by CIBA Products Co.
Versamid 140, marketed by General Mills, Inc.

The detailed requirements for this curing agent are as follows:

Viscosity at 25 degrees C., cps - - - - - - - 12,500 to 17,500

Specific Gravity, 20/20 degrees C.- - - - - - 0.96 to 0.98

Amine Value - - - - - - - - - - - - - - - - - 350 to 400

Test methods to be used in determining these qualities are listed below:

1. Viscosity - Method of Test for Kinematic Viscosity (ASTM D445-60)

2. Specific Gravity - Method of Test for Density of Paint, Varnish, Lacquer, and Related Products (ASTM Designation D1475-57T)

3. Amine Value - Method of Test for Total Amine Values of Fatty Amines by Referee Potentiometric Method (ASTM D2073-62T)

Pigments

1. Titanium Dioxide

   The titanium dioxide used in this formulation shall be equivalent to DuPont R-900. This shall be a pure, non-chalking, Rutile titanium dioxide meeting the requirements of Federal Specification TT - T - 425a, Type III.

2. Carbon Black

   The carbon black used in this formulation shall be equivalent to Excelsior, manufactured by Columbian Carbon. The carbon black used shall conform to Federal Specification TT - C - 120.

Compounding of the Components of Epoxy Binder B-102

The components of the epoxy binder shall be compounded from the materials previously specified according to the following formulas.

Resin Component

The resin component of the epoxy binder shall contain the following materials in the ratio shown:

100 parts by weight epoxy resin

4 parts by weight Titanium Dioxide

0.005 part by weight carbon black

The resin component, after compounding, shall have a grind of 5 minimum.

The method of mixing these materials to form the resin component shall be as desired by the manufacturer so long as the finished product complies with the physical requirements stated in this specification.

Hardener Component

The hardener component of the epoxy binder shall contain the following materials in the ratio shown:

40 parts by weight modified amido-amine curing agent

15 parts by weight polyamide curing agent

These materials shall be thoroughly mixed and packaged as the hardener component.

Packaging

Shipment shall be made in suitable, strong, well sealed containers which not only meet specification and ICC requirements but also are sufficiently sturdy to withstand the normal handling to which shipments are subjected in transit. The size and type of containers and the amount of material to be placed in each container shall be as specified on the bid invitation and material requisition.

Labeling

The finished epoxy binder containers and cases shall be plainly and securely labeled. The resin component shall have the following instructions on the label:

Texas Highway Department Epoxy Binder B-102

This material is intended to be mixed with various types of aggregate to form an epoxy mortar or concrete to be used in the repair of portland cement concrete structures.
Note: For detailed use instructions, see D-9 pamphlet entitled Instructions Regarding the Use of Epoxy Binder B-102.

Add the hardener component to the can containing the resin component and mix thoroughly, preferably with a small mechanical stirrer. If material is to be mixed by hand, gradually add the hardener component to the resin component while stirring. If less than the entire unit is needed, the two components may be mixed in the following ratio:

3 parts resin component by volume
2 parts hardener component by volume
or
1.9 parts resin component by weight
1.0 parts hardener component by weight

Caution - Do not attempt to thin epoxy binder with any type of solvent. Use toluene or bead binder thinner for clean-up of equipment. Do not store at temperatures below 60 degrees F. or above 100 degrees F.

WARNING

May cause skin irritation. Avoid contact with skin, eyes and clothing. In case of contact with skin or clothing, wash skin immediately and thoroughly with soap and water. Remove and wash clothing before re-use. For eyes, flush with plenty of water; get medical attention.

Each can of resin component shall also have the following information on the label:

RESIN COMPONENT
Batch Number
Order Number
Date of manufacture
Gross weight
Net weight
Name of manufacturer

The hardener component shall have the following information on the label:

Texas Highway Department Epoxy Binder B-102

HARDENER COMPONENT
Batch Number
Order Number
Date of manufacture
Gross weight
Net weight
Name of manufacturer

Each container of hardener component shall also have the following storage and handling precautions on the label:

Do not store at temperatures below 60 degrees F. or above 100 degrees F.

WARNING

May cause skin irritation. Avoid contact with skin, eyes and clothing. In case of contact with skin or clothing, wash skin immediately and thoroughly with soap and water. Remove and wash clothing before re-use. For eyes, flush with plenty of water; get medical attention.

Physical Requirements for Epoxy Binder B-102

Resin-Hardener Mixture

The epoxy binder shall consist of 1.9 parts by weight resin component and 1.0 part by weight hardener component. The mixture of the two binder components shall meet the following requirements:

Pot Life at 77 Degrees F. - 36 minutes minimum
50 minutes maximum

Samples of each of the binder components shall be brought to 77 $\pm$ 2 degrees F. Then 62.5 grams of the resin component and 33 grams of the hardener component shall be weighed into a 9 ounce unwaxed paper cup. The time shall be recorded and the two components mixed for three minutes, taking care to periodically scrape the walls and bottom of the cup. The cup shall then be set on a wooden bench top and probed every two minutes with a stirring rod, starting 20 minutes from the initiation of mixing. The time at which gelled material first forms in the container is recorded as the pot life.

Physical Requirements for the Cured Binder*

Adhesive Shear Strength - Steel to Steel - (ASTM-D1002-53T) - 2400 PSI Minimum.

The surfaces of the test specimens used in the adhesive shear strength test shall be prepared by blasting to white metal. The blasted surfaces shall be washed with methyl ethyl ketone and allowed to dry before applying the adhesive. The surface of the test specimens shall have a prepared surface of equivalent "anchor pattern" to that which would be obtained by abrasive blasting the surfaces of the test specimens with a gun pressure of 50 to 75 psi using a ¼ inch diameter nozzle and employing Garnet Blasting Abrasive "Gem Blast", 60 mesh (No. 45 to No. 74 U.S. Standard Screens), as marketed by Clemtex, Inc., of P. O. Box 15214, Houston, Texas.

Cleavage Strength - Steel to Steel - (ASTM-D1062-51) - 1000 PSI Minimum
Surface preparation shall be as outlined in Adhesive Shear Strength.

Water Gain - 24 hour immersion at 23 degrees C. - (ASTM-D570-57T) - 0.20 by weight maximum.

Sampling and Testing

A. Materials to be tested. The epoxy binder purchased by the Highway Department under this specification shall be tested for conformance to said specification.

B. Agency. All tests on finished products and raw materials, as well as inspection during manufacture will be made by the Texas Highway Department, or by a commercial laboratory designated by the Highway Department.

C. Inspection and Testing Expenses. The cost of inspection and testing of this material will be borne by the Highway Department. The manufacturer will be expected to bear the actual cost of the materials and finished binder taken as samples at the time of manufacture. The size of samples taken will be no larger than is absolutely necessary for testing purposes.

D. Methods.

1. Sequence of Inspection.

a. Immediately after the contract has been awarded, the supplier will contact the Materials and Tests Engineer, Texas Highway Department, Austin, Texas, regarding brand names and characteristics of all raw materials which the contractor proposes to use; and to make arrangements for inspection during production.

*The adhesive shear strength, cleavage strength, and water gain are to be determined on material that has cured for seven days at 70 - 80 degrees F.

b. Manufacture shall be witnessed in whole or in part depending upon the discretion of the testing agency. Production shall not begin prior to the arrival of the Highway Department Inspector unless prior specific approval for such starting has been obtained. Samples of raw materials actually used in production and samples of the binder shall be taken during production. The manufacturer shall afford the inspector free access to those parts of the plant wherein the binder is being manufactured or raw materials are being stored, and in all other ways shall facilitate the inspection and sampling process. Any questions regarding the inspection or testing shall be addressed to the Materials and Tests Engineer, Texas Highway Department, Austin, Texas.

2. Basis for Rejection. The finished epoxy binder and any of the materials used in the binder which fail to meet any or all of the requirements of this specification shall be subject to rejection. Final acceptance or rejection shall be based on results of tests on samples of raw materials, binder components, and the mixture of the two components taken during production and upon tests made on the binder components and the combination of the two components after the binder has arrived at the shipping destination. Approval of materials, as a result of preliminary testing prior to manufacture into the finished binder, shall not be binding upon final approval or rejection. Because of the possibility of contamination and volatile losses, it shall be agreed that the resin and hardener reference standards currently in the possession of the Highway Department or its authorized testing agencies, shall constitute standards for final comparison involving acceptance or rejection. Samples of these reference standards are available to the manufacturer. The judgment of the Highway Department's Materials and Tests Engineer shall be final in all questions relative to conformance with the provisions of these specifications.

INSTRUCTIONS FOR USE OF TEXAS HIGHWAY DEPARTMENT EPOXY BINDER B-102

Materials and Tests Division

Epoxy Binder B-102 is intended to be mixed with selected aggregates to form a quick-setting epoxy mortar or concrete. The resulting epoxy mortar or concrete may be used to fill cracks and to repair pot holes or spalled areas on concrete structures. This material may be used when the concrete temperature is between 60 and 120 degrees F. and the ambient temperature between 60 and 105 degrees F. Epoxy Binder B-102 should not be used outside these temperature limits. This material is currently available in 3/4 gallon and 2½ gallon units. Instructions concerning its use are given on the following pages. It should be kept in mind that in order to obtain the best results with this material, it must be properly used.

Aggregates to be Used in Preparing Epoxy Concretes and Mortars

The aggregates to be used with Epoxy Binder B-102 should meet the quality requirements specified under Item 421, "Concrete for Structures," THD 1962 Standard Specifications for Road and Bridge Construction. The aggregates to be used should be dry. The maximum size of the aggregate to be used will depend upon the dimensions of the fill. A good rule to follow is that the maximum size aggregate should not exceed one-fourth of the smallest dimensions of the fill. For an epoxy concrete to be used where a large volume of fill is required, gravel or crushed stone, 3/4 to 1/2 inch maximum size, uniformly graded from coarse to fine may be used. For most work, an epoxy mortar prepared using an aggregate with a grading approximating that of THD Grade No. 1 Fine Aggregate would be desirable. The grading limits for this material are as follows:

Sieve Size		Per Cent Retained, Cumulative
No. 4	(4.76 mm)	0-5
No. 8	(2.38 mm)	0-20
No. 16	(1.19 mm)	15-50
No. 30	(595 microns)	40-75
No. 50	(297 microns)	70-90
No. 100	(149 microns)	90-100

THD Grade No. 1 Fine Aggregate (Concrete Sand) may be used in preparing epoxy mortars. However, somewhat higher strengths and better workability may be obtained if all material passing the No. 50 sieve is screened out before use. Even better results may be obtained by using a rounded grain sand free of fines. A specification for this type of sand is given below.

> The sand to meet this specification shall be a rounded grain washed sand free of fines. It shall comply with the following physical and chemical requirements:
>
> Moh Hardness - - - - 7 Minimum
> Specific Gravity - - 2.60 Minimum
> Per Cent Silica - - 99.5 Minimum by Wt.
> Grading - To be specified

Note: A sand meeting these requirements is produced by Pennsylvania Glass Sand Corporation, San Saba, Texas. The following gradings of this sand are packaged in 100 pound sacks:

8 - 12 mesh
10 - 20 mesh
16 - 30 mesh
20 - 40 mesh

Because of the availability of this sand, the Materials & Tests Division used it in experimental work involving aggregates. The best results were obtained using a combination of the gradings shown above. The composition of a typical mix prepared with special sand and 3/4 gallon of Epoxy Binder B-102 is shown below:

24 pounds 8 - 12 mesh sand
6 " 10 - 20 mesh sand
6 " 16 - 30 mesh sand
12 " 20 - 40 mesh sand
7 " B-102 Epoxy Binder

or by Volume:

1½ gallons 8 - 12 mesh sand
½ " 10 - 20 mesh sand
½ " 16 - 30 mesh sand
3/4 " 20 - 40 mesh sand
3/4 " B-102 Epoxy Binder

In some cases where it is desired to fill shallow spalled areas or relatively small cracks, a relatively fine sand of uniform grading may be used. A 30 mesh sand has been used successfully as the aggregate for this type of application.

General Instructions

Handling Precautions

The epoxy materials are capable of causing irritation or other physiological reaction in some individuals. For this reason, care should be exercised in handling this material. If a workman should develop a skin rash or similar reaction while working with the epoxy, it would be best for him to discontinue handling this material, as continued contact with the epoxy may result in greater irritation or reaction.

Mixing

The two components of Epoxy Binder B-102 must be thoroughly mixed together, preferably with a small mechanical mixer. If a mechanical mixer is not available, the binder may be mixed by hand. If an entire unit is to be mixed and used at one time, the hardener component should be added to the resin component and mixing done in the resin component container. If less than a full unit of binder

is needed for a particular application, the two components may be mixed in the following ratios:

3 parts resin component by volume
2 parts hardener component by volume

or

1.9 parts resin component by weight
1.0 part hardener component by weight

After the two components of the binder have been mixed together, the binder is ready to add to the aggregate which has been selected. The ratio of aggregate to the epoxy binder will usually range from 5 to 10 parts aggregate by weight to 1 part binder by weight. The ratio will depend upon the type of aggregate used and the working characteristics desired. In order to determine for any given aggregate the ratio of aggregate to binder that will give the desired finishing and placing characteristics, it is a good practice to make up small trial batches of the epoxy mortar. Batches of material made up experimentally should contain at least 2 pounds total weight of aggregate and binder.

The simplest method of mixing the aggregate and binder is by hand in metal pans. This simplifies clean-up of the equipment following the mixing of the epoxy mortar. The aggregate to be used may be placed in a metal pan of suitable size and the binder added gradually, with mixing, to the aggregate. Note: The binder should be added to the aggregate immediately after the resin and hardener components have been mixed together. A small amount of the binder should be saved rather than added to the aggregate. This amount of binder should be used to prime the concrete surfaces against which the epoxy mortar is to be placed. A thin coat of the pure binder should be brushed onto all the surfaces to which the epoxy mortar is to be bonded.

The size of the batch of epoxy mortar made up using 3/4 gallon of binder will vary from about 40 to 80 pounds. The average time required to mix the binder and aggregate by hand with a hoe or trowel will be about 10 minutes. The working time after the mortar is ready for use will vary from about 20 to 50 minutes depending upon the ratio of aggregate to binder, the size of the batch, and the ambient temperature. The working time can be extended somewhat by spreading the mix out thin prior to placing it so that there will not be a heat build-up due to the reaction which takes place between the epoxy resin and the hardener.

Placing of the Epoxy Mortar

Prior to placing the epoxy mortar, the surfaces to which it is to bond must be properly prepared. Any loose or unsound concrete must be removed. Areas to be patched should be chipped out so that the edges will be essentially perpendicular to the top surface of the finished patch. This will eliminate thin featheredging. Layers of epoxy mortar less than ¼ inch in thickness are susceptible to chipping and for this reason it is generally best to avoid featheredging patches. The surfaces to which the epoxy mortar is to be bonded must be clean. Pressure washing with water followed by drying with compressed air is adequate in many cases. In cases where the concrete surface is soiled with oil, grease, or

other foreign matter which cannot be removed by water washing, sandblasting is desirable. The sandblasted area should be washed and dried prior to placing the epoxy mortar. In some cases, acid etching of the concrete may be feasible. A solution of 2 parts water and 1 part 20 degree Baume' muriatic (Commercial hydrochloric) acid by volume may be used for this purpose. The amount of acid required is approximately one gallon per four square yards. The acid is spreas over the surface and allowed to react with the concrete. This must be followed by a thorough washing with water. The concrete should then be dried before applying the epoxy mortar. It should be kept in mind that if acid etching is used as a method of surface preparation, precautions must be taken to protect both personnel and equipment from the acid and its fumes. Personnel applying the acid should be supplied with goggles and rubber gloves.

After the concrete surfaces have been prepared, the binder to be used as a primer should be brushed on. The epoxy mortar may then be placed. Working of the epoxy mortar while putting it in place should be kept to a minimum to prevent the binder portion from being worked up to the surface of the mortar. If it is necessary to use forms in conjunction with placing the epoxy mortar, the surfaces of the forms may be greased in order to prevent adherence of the mortar to the forms. Polyethylene sheeting may also be used between the epoxy mortar and the form in order to prevent adhesion.

After being put in place, the epoxy mortar will require about 6 hours at 80 degrees F. to obtain a good initial set. If the ambient temperature and the temperature of the concrete is 90 degrees F. or higher, it would be possible, if necessary, to turn traffic on the repaired areas 3 to 4 hours after placement of the epoxy mortar. However, it is best to wait 6 hours if conditions permit.

Clean-up

Any equipment used in mixing and applying the epoxy binder and mortar should be cleaned before the material hardens. Toluene is recommended as a solvent for approxing the epoxy material. BBT-9 Bead Binder Thinner may also be used for this purpose if it is more readily available. Both of these solvents are stocked by the Highway Department regional warehouses in 5 gallon containers. The stock numbers are as follows:

307700 - - - - Thinner, BBT-9 Bead Binder
307800 - - - - Toluene

It should be kept in mind that these solvents are quite flammable and should be used with caution.