



TWR-16417

EVALUATION OF A METERING, MIXING, AND DISPENSING
SYSTEM FOR MIXING POLYSULFIDE ADHESIVE

FINAL REPORT

JUNE 1989

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**EVALUATION OF A METERING, MIXING, AND DISPENSING
SYSTEM FOR MIXING POLYSULFIDE ADHESIVES**

FINAL REPORT

JUNE 1989

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References: 1. Work Request No. I89028

2. ETP-0406, "Evaluation of a Metering, Mixing, and Dispensing System for Mixing PR-1221 Polysulfide Adhesive"

3. ETP-0493, "Polysulfide Mix Ratio and Lot-to-Lot Variation Study"

Process Engineering Technical Report Categories

| | |
|-----------|----------|
| Adhesives | Mixing |
| Mixers | Sealants |

1.0 INTRODUCTION

Presently, a 4-gallon Ross mixer (Model No. LDM4) is used to mix PR-1221 polysulfide adhesive (STW4-3311) which is used as a sealant in the RSRM nozzle/case joint. This mixer is inefficient in that it does not mix the adhesive completely. A scraper arm is not attached on the mixer blades and the adhesive is viscous enough to make a thorough manual scrapedown of the mix bowl interior very difficult. Consequently, unmixed base and catalyst remain on the mix bowl interior and mixer blades upon completion of the mix cycle. Also, this adhesive is vacuum loaded into Semco cartridges which allows for air bubbles in the material.

Tests were performed at Liquid Control Corporation (LCC) to evaluate the efficiency of a fixed-ratio meter mixer which has been built to meter, mix, and dispense polysulfide adhesives, PR-1221, PR-1860, and PS-875 manufactured by Products Research Company (PRC). (Adhesives PR-1860 and PS-875 are being qualified as replacements for PR-1221 which will be discontinued in the near future.)

The mechanical (tensile adhesion, 180 degree T-peel, and Shore A hardness) and physical (specific gravity of mixed adhesive) property data show no difference in static and batch (hand) mixing. In fact, a test of the null hypothesis (student t distribution) which was performed on the average tensile adhesion and peel strengths justifies the statement that with 99 percent confidence the differences in average tensile adhesion and average maximum peel strengths of static-mixed and hand (control)-mixed polysulfide adhesives are not statistically significant.

Visual tests, called the butterfly tests, showed no evidence of unmixed material. (These data are tabulated in Table I.) It is important to note that these data are useful only for comparing the static and batch mixing methods. Because these samples were disturbed and exposed to temperature extremes during transport from the LCC lab to the plant, it is scientifically unsound to compare these data to the existing database. Also, the existing database (tensile adhesion and peel) is based upon specimens which were prepared from a 10:1 base-to-catalyst weight ratio. As is explained in the next paragraph, the samples of these tests were prepared from a different component weight ratio for each adhesive.

The meter mixer system was also checked out for its metering accuracy and consistency. This checkout was accomplished simply by taking weight ratio measurements. Ideally, one stroke of each pump should volumetrically meter ten parts by weight of base to one part of catalyst. However, weight ratios of 9.45:1, 8.94:1, and 12.53:1 for PR-1221, PR-1860, and PS-875, respectively, were measured at the pump exits. These ratios were repeated on each measurement.

The metering consistency of the pumps verifies that the discrepant ratios were not caused by a pump design defect. The differing weight ratios could be due to one or both of the following reasons:

- o Variation in component specific gravity
- o Incorrect volumetric ratios given by PRC to which the metering pumps were sized

This issue will be resolved by further testing as outlined in ETP-0493.

In general, the meter mixer performed adequately with minor exceptions. The maximum flow rates at maximum pump pressure of PR-1221 and PR-1860 were measured to be 0.31 and 0.23 pounds per minute which are lower than the vendor guaranteed 0.5 pounds per minute. Obviously, lower flow rates mean more time required to apply the adhesive. However, this increase in application time is not significant. Also, the higher pump pressure (90 psi) caused micro-balloon breakage in the PS-875 base material. This problem is not of concern since the base metering pump can be modified to eliminate breakage. Otherwise, upon resolution of the discrepant weight ratio problem, the metering, mixing, and dispensing system is satisfactory for production qualification testing.

2.0 TEST OBJECTIVE

To evaluate whether a metered mixing system can mix PR-1221 polysulfide adhesive as well as or better than batch-mixed adhesive. Also, to evaluate the quality of meter-mixed PR-1860 and PS-875 polysulfide adhesives. These adhesives are candidate replacements for PR-1221 which will not be manufactured in the future. The following material properties were evaluated:

- o Peel strength
- o Specific gravity and adhesive components of mixed adhesives

- o Shore A hardness
- o Tensile adhesion strength
- o Flow rate

Finally, a visual test called the "butterfly test" was performed to observe for bubbles and unmixed adhesive.

3.0 CONCLUSIONS

1. The present polysulfide (PR-1221) adhesive batch mixing and application method results in unmixed and aerated material.
2. A metering, mixing, and dispensing system is advantageous to the polysulfide mixing and application process because it:
 - o Can be located at the application site
 - o Reduces material wastes
 - o Allows more time for application
 - o Dispenses adhesive directly on the part
 - o Yields an air-free material
 - o Completely mixes the adhesive
3. In terms of tensile adhesion and peel strength, Shore A hardness, and mixed material specific gravity, there is no difference between statically-mixed and the control (hand)-mixed PR-1221, PR-1860, and PS-875 polysulfide adhesives. Also, it can be said with 99 percent confidence that the difference in average tensile adhesion and maximum peel strengths of statically- and hand-mixed PR-1221, PR-1860, and PS-875 adhesives is not statistically significant.
4. Butterfly tests showed no striations of unmixed adhesive after statically mixing these adhesives through a series of two 1/2-in. and one 3/4-in. static mixers (48 elements total).

5. The meter mixing system produced maximum flow rates of 0.31, 0.23, and 0.96 pounds per minute for the PR-1221, PR-1860, and PS-875, respectively. At these flow rates, it would take about 35, 48, and 11 minutes to apply the approximate amount of adhesive (11 pounds) necessary for the nozzle-to-case joint. Two of these flow rates are low compared to the 0.50 pounds per minute promised by the vendor in the purchase contract. However, flow rate is not a critical criterion of the polysulfide application.
6. The average (three measurements) base-to-catalyst weight ratios of PR-1221, PR-1860, and PS-875 were 9.41:1, 8.94:1, and 12.53:1, respectively. These ratios (measured at the sampling ports just downstream from the pumps) are discrepant to the vendor-recommended 10:1 ratio. The cause of the differing ratios is due to incorrect base/catalyst volumetric ratio supplied by PRC to LCC and/or lot-to-lot variations in the specific gravities of the base and catalyst components.
7. Specific gravities of most components of these adhesives measured at LCC were significantly different from the values provided by PRC.
8. Repeating weight ratios of all three adhesives verify that the metering pumps consistently meter the same volume of components after each stroke. Also, this repeatability was verified at the end of the 25-ft hoses.
9. A pump pressure of 90 psi caused significant breakage of the micro-balloons in the PS-875 base material. The specific gravity of the base increased by 20 percent after being pumped.
10. The following modifications and repairs of the metering, mixing, and dispensing system are necessary before shipment:
 - o Faulty valve replacement in the control panel
 - o Base recycle system
 - o Catalyst recycle system for catalyst side drainage capability
 - o Clean and repaint equipment

4.0 RECOMMENDATIONS

It is recommended that:

1. Tests be performed to determine the effect of varying the weight ratio of PR-1221, PR-1860, and PS-875 on the characteristics of the adhesives. Also, tests be conducted to check for any variation in the properties of each adhesive on a lot-to-lot basis. These tests should include tensile adhesion strength, T-peel, viscosity versus time, Shore A hardness, and specific gravities of the components and the mixed adhesives.
2. Pending positive outcome of the PR-1221 phase of Recommendation No. 1, the meter-mixing system be shipped to plant for further full-scale (NJAD) testing. However, the previously identified improvements and repairs (Conclusion 10) must be made prior to shipment.

5.0 DISCUSSION

A fixed-ratio airless metering, mixing, and dispensing system is recommended to efficiently mix polysulfide adhesives. Basically, the recommended system consists of automatic proportioning pumps which meter the base and catalyst flow according to the desired base-to-catalyst weight ratio. (Figure 1 illustrates the basic design of a fixed-ratio metering, mixing, and dispensing system.)

The adhesive components are mixed just prior to being dispensed through the nozzle in a static mixer (Figure 2). A static mixer divides and combines materials by two raised to the nth power; n being the number of elements in the mixing chamber.

The mixed adhesive can be directly dispensed in Semco tubes or onto the part by a gun. This metered mixing system has the following advantages:

- o A portable system, located in the facility where the application is performed, would eliminate the need to transport the polysulfide adhesive from one facility to another.
- o A reduction (about 45 percent) in the amount of adhesive material wasted during the operation could be realized.
- o Mixing the adhesive in the mixing chamber of the system eliminates concerns that the pot life would be exceeded before the adhesive can be applied. Pot life constraints sometimes result in hasty and sloppy work.

Space Operations

- o The mixed material would be delivered to the part air-free as long as the raw materials are packaged air-free.
- o There would be no unmixed base or catalyst; hence, adhesive with optimum material properties would be delivered to the part.

The following list helps to more specifically describe the model built for polysulfide application:

- (1) The catalyst is gravity fed from the component reservoir to the pump cylinder.
- (2) Because of its high viscosity, the base is fed from the component reservoir to the pump cylinder by pressure applied to a follower plate.
- (3) Catalyst and base sampling ports are supplied for weight ratio checks immediately after metering and for pump phasing.
- (4) Prior to mixing, the components flow separately through 25 feet of stainless steel braided, Teflon-lined, lightweight hose.
- (5) Initial mixing is accomplished by two ½-in. by 12-element stainless steel static mixers (24 elements total). The catalyst is introduced into the mixer downstream from the base. This sequence prevents catalyst from being lost on the mixer interior because of the high volume base flow.
- (6) Approximately the last three feet of hose are flexible hose for ease of application.
- (7) Attached to the hand-held dispensing gun is a 3/8-in. to 3/4-in. by 24-element disposable static mixer which was added when it was discovered that 24 elements did not completely mix the components.
- (8) Upon completion of the operation, the catalyst can easily be recycled to its reservoir while the system is purged of mixed material by the base.
- (9) The system has two dispensing modes; continuous and single shot/stroke.
- (10) The catalyst reservoir tank is supplied with an agitator to blend materials which may settle out after periods of inactivity.

- (11) Both component pumps are single acting. A single acting pump discharges material on the down stroke and reloads material on the up stroke. To prevent cavitation, which is a problem when pumping material of high viscosity, the pressure-applied follower plate was added to the base reservoir. Also, cavitation will be prevented by keeping the component reservoirs full and the component feed lines unrestricted.

In addition, Liquid Control agreed to make the following modifications/improvements to the machine before delivery:

- o Replace a faulty valve in the control panel.
- o Provide a base recycle system to minimize base material waste while the system is being charged with catalyst.
- o Provide capability to completely drain the catalyst side by pumping unused catalyst into the reservoir tank. This option eliminates the possibility of using unagitated catalyst after periods of inactivity.
- o Clean and repaint the equipment.

The specific gravity, flow rate, peel strength, tensile adhesion, Shore A hardness, and butterfly tests were performed as outlined in the Test Implementation section. In addition to these tests, base-to-catalyst weight ratios and base and catalyst specific gravities were measured. As instructed by the test plan (ETP-0406), tack-free time tests were performed; however, the results are invalid since the samples were exposed to a low temperature (about 55°F) overnight. This type of prolonged exposure significantly overextended the tack-free time. Specific gravities of the components and mixed adhesives were conducted with a pycnometer which differs from the method specified in the test plan. However, use of a pycnometer is adequate for these tests. Shore A hardness measurements were taken at 8, 18, and 23 days. All other tests were conducted as originally outlined in the test plan with the exception of ~~the~~ the adhesive cure cycles which reflect the fact that samples were transported from the LCC lab (Canton, Ohio) to the Morton Thiokol lab. Weight ratio tests were performed by a simple procedure which is:

- (a) Tare out two sample cups.
- (b) Engage the metering pumps for a single stroke.
- (c) Allow the components to fill the tared cups at the sampling ports.

- (d) Weigh the filled cups.
- (e) Calculate the weight ratio by dividing the weight of the catalyst into the weight of the base.
- (f) Repeat Steps 1 through 5 until three data points are obtained.

All data are tabulated in Tables I and II. The tensile adhesion data show little difference between tensile adhesion strengths of hand-mixed and meter-mixed PR-1221, PR-1860, and PS-875. Test of the null hypothesis (student distribution t test) confirms that with 99 percent confidence, the difference between the average tensile adhesion strengths of each adhesive mixed by the different methods is not statistically significant. The overall failure modes of each meter-mixed and hand-mixed adhesive are similar. The high coefficients of variation are due to the erratic cure cycles experienced by the specimens. Particularly, the PS-875 samples which were never exposed to an elevated temperature cure (the oven at the LCC lab was mistakenly turned off). The adhesive was tacky to the touch even after the specimens were tested. Most PR-1221, PR-1860, and PS-875 specimens were not allowed enough time to cure. Nevertheless, the tensile adhesion data clearly show that static mixing is just as efficient as hand mixing.

The peel data reflect the same conclusion about static and hand mixing as the tensile adhesion data. Again, statistical analysis verifies that with 99 percent confidence, the differences in average maximum peel strengths is not statistically significant. Of some concern is the adhesive failure displayed by the PR-1860 meter mixed samples (23 percent). Because the peel strength is high and there were considerable voids, this high degree of undesirable failure is probably due to the method of preparation of the peel specimen.

It is important to emphasize that the data compare mixing ~~methods~~ (static and hand) only. Comparison of the meter ~~mixing~~ data with an existing database would be unscientific, because these samples were cured at different conditions, formulated by different weight ratios, and physically disturbed during transfer from lab to lab.

Also of importance is the difference between hand and machine batch mixing. Although hand mixing is a batch mixing method, it is only somewhat representative of the present production batch mixing process in the Ross mixer simply because small amounts of adhesive are hand mixed. This distinction means that hand-mixed batches are easier and more thoroughly mixed than a machine-mixed adhesive. By visual observation, these control mixes were completely mixed. Theoretically, the control sample data reflect the optimum adhesive properties relative to the cure cycle used in testing. This issue is relevant because it further validates static mixing in that the mechanical properties of the statically mixed sample are equivalent to the controls. Hence, static mixing yields an adhesive of excellent mechanical properties. Furthermore, a premise for conversion from batch mixing to static mixing was the incomplete mixing common to mixing polysulfides in a Ross mixer. Static mixing satisfies this premise.

All other data listed in Table I (i.e., butterfly tests, and specific gravities of the mixed adhesive) support the conclusion that static mixing produces polysulfide adhesive equivalent to the control mixed adhesive. The butterfly test is a visual method for checking the mix for evidence of unmixed material. The initial sequence of static mixers included two 12-element mixers. Butterfly tests showed striations of catalyst in the base after mixing with this configuration. To improve the mix, a 24-element mixer was added prior to the dispensing gun and each subsequent butterfly test passed.

Table I lists the average of four and maximum flow rate measurements for each adhesive. The vendor was contracted to build a machine which generates a minimum flow rates of 0.5 pounds per minute. Even at the highest pump pressure (90 psi), the maximum flow rates for PR-1221 and PR-1860 were low. At these flow rates, the actual time for application of the adhesives to the part will be 35, 48, and 11 minutes for the PR-1221, PR-1860, and PS-875, respectively. (Each production application averages 11 pounds of PR-1221.) Present processing time for application of adhesive to a part is 20 to 25 minutes. The low PR-1221 flow rate is not of concern since processing time is increased by only 10 to 15 minutes. However, it is of significance when applying the PR-1860 and modifications to the system may be necessary.

Tests were conducted to check out the metering efficiency of the meter mixing system. (The discussion in the following paragraphs details the sequence of events as these tests were performed on PR-1221. However, this discussion is relevant to all three adhesives.) The base to catalyst weight ratio out of the sampling ports repeatedly measured 9.41:1 (measured three times) and the vendor, Products Research and Chemical Corporation (PRC), recommended ratio is 10:1. The metering ratio was also checked at the end of the 25-ft hoses and consistently measured to be 9.45:1. The repeating ratios verified that once phased (stroking at the same time), the metering pumps were consistently metering the same volume of components. However, the 9.4:1 and 10:1 ratio discrepancy is cause for concern. Since the pumps were accurately metering, the cause of the discrepant ratios is due to incorrect base/catalyst volumetric ratio given by PRC and/or lot-to-lot variations in the specific gravities of the components.

Theoretically, the metering cylinders of the pumps can be volumetrically sized to achieve the proper weight ratio according to the formula:

$$V = W \times \frac{SGB}{SGA}$$

Where:

- V - Volumetric ratio of base to catalyst
- W - Weight ratio of base to catalyst
- SGB - Specific gravity of the catalyst
- SGA - Specific gravity of the base

Obviously, if the components' specific gravities vary from lot-to-lot; then, the volumetric ratio must vary to maintain a constant weight ratio. It is not possible to vary the volumetric ratio since the metering system is fixed ratio and a fixed ratio machine is preferred over a variable ratio. Therefore, the weight ratio will vary with specific gravities. According to the polysulfide adhesive specification (STW4-3311), the base-to-catalyst weight ratio can vary from 8.9:1 to 11.3:1; hence, the measured meter mixer weight ratios were within specification limits.

To investigate the issue further, the specific gravities of the base and catalyst were measured to be 1.4 and 3.2, respectively. The PRC-provided literature values are 1.4 (base) and 2.65 (catalyst). When contacted, PRC said that polysulfide component specific gravities can vary from batch-to-batch; particularly, the catalyst which contains lead which frequently settles out. The tested catalyst (specific gravity 3.2) had not been agitated prior to testing.

The PR-1221 metering cylinders were sized and built by LCC based upon the volumetric ratio given by PRC (20.20:1). However, when the vendor-specified specific gravities (1.4 and 2.65) and weight ratio (10:1) are plugged into the above formula, the volumetric ratio is 18.93:1. Finally, when the measured specific gravities (1.404 and 3.217) and weight ratio (9.41:1) are used, the volumetric ratio is 21.56:1. This confusion for all three adhesives is summarized in Table II.

The bottom line questions that must be answered by future testing to clear the confusion are:

1. Did PRC provide incorrect volumetric ratios and base and catalyst specific gravities?
2. Is there a wide lot-to-lot variation in specific gravities of the individual components?

If Number 1 is determined to be the cause; then, the problem can be resolved by adjusting the meter mixer to the new volumetric ratio. However, if Number 2 is the problem; then, it must be determined if an acceptable mix can be consistently produced over the entire specific gravity tolerance band. These tests will be performed as outlined in ETP-0493.

The final item that this TWR needs to document is a problem that may be encountered if PS-875 is used as the nozzle-to-case joint adhesive. The PS-875 base is filled with micro-balloons. Micro-balloons burst when under high pressures. Of course, this bursting effect may diminish the mechanical properties of the adhesive.

Therefore, the specific gravity of the base before and after it was pumped was checked. A significant increase in the specific gravity of the pumped material would indicate breakdown of the micro-balloons. The increase when the pump pressure was 40 psi was not significant (four percent) as the specific gravity went from 0.99 to 1.029. However, when the pump pressure was increased to 90 psi, the specific gravity of the base increased by 20 percent from 0.99 to 1.18. (The popping of the micro-balloons could actually be heard.) Therefore, if PS-875 is chosen as a replacement, it will not be possible to pump the base material at higher pressures unless special modifications are made to the pump.

To summarize, pending the outcome of the tests conducted through ETP-0493, this metering, mixing, and dispensing system should be shipped to the plant for large-scale qualification tests (NJAD).

6.0 TEST IMPLEMENTATION

The following test procedures were implemented for batch-mixed and continuous-metered mixed adhesive. Also, the tests were repeated for all three adhesives. Specimens were prepared at the meter mixer manufacturer's facility and transported back to MTI for testing. Control specimens were prepared at the manufacturers facility by hand mixing an adhesive batch for each adhesive.

6.1 Specific Gravity

Specific gravities on the adhesive components and mixed adhesive (uncured) of all three adhesives were performed by a pycnometer. Three specific gravity measurements were taken on all materials.

6.2 Flow Rate Tests

- a. Tare at least three containers in which adhesive can be dispensed.
- b. ~~Dispense~~ Dispense adhesive into a tared container for at least 30 seconds.
- c. Repeat for the other two tared containers.
- d. Weigh the adhesive in the three containers.

- e. Calculate flow rates as follows:

$$\text{Flow Rate} = \frac{W}{T}$$

Where W = Weight of the adhesive to each container
(final weight - tare weight)

T = Time in minutes the adhesive was
dispensed.

- f. Record the average, maximum, and minimum flow rates.

6.3 Tack-Free Time

- a. Mix adhesive.
- b. Apply the mixed adhesive to a Plexiglas plate, or other suitable flat surface, to form six specimens measuring approximately two inches in diameter by 1/8-in. thick.
- c. At the end of 24 hours, measured from the beginning of the mixing period, place a polyethylene film, approximately 1-in. wide by 6-in. long by 0.004±0.002-in. thick, on each of the specimens.
- d. Hold the film in place with a pressure of at least two ounces per square inch of adhesive specimen for at least two minutes.
- e. Remove the film slowly at a right angle to the surface of the specimens.
- f. Inspect to determine if the film has come away clean and free of adhesive.

6.4 Peel Strength Tests

- a. Prepare two T-peel test panels having a minimum of five specimens per panel as detailed in ASTM-D-1876 and use 0.300-in. thick vulcanized NBR.
- b. Bond the panels with mixed adhesive by wetting both panel bonding surfaces.
- c. Apply an adhesive layer of approximately 1/8-in. on each bonding surface. (See Figure 3 for the specimen configuration.)

- d. Cure the adhesive for 24 hours at $120^{\circ}\pm 5^{\circ}\text{F}$, then for 168 hours minimum at $75^{\circ}\pm 5^{\circ}\text{F}$.
- e. Cut each panel into separate one-inch wide specimens.
- f. Pull the specimens at 10 in. per minute and record the peel strength in pounds per linear inch and the failure mode.

6.5 Tensile Adhesion Tests

The specimen configuration for these tests is illustrated in Figure 4.

- a. Obtain 20 D6 steel tensile adhesion buttons which have been vapor degreased and grit blasted. Remove grit with a nitrogen blast within four hours of bonding or vulcanizing.
- b. Apply a coat of Chemlok 205 to 10 buttons. Allow 10 minutes to dry.
- c. Apply a coat of Chemlok 233 to the same 10 buttons used in Step 8.5 (b). Allow 30 minutes to dry.
- d. Lay up 0.10 in. of NBR (STW4-2621) on the 10 buttons used in Step 8.5 (c).
- e. Cure 90 - 120 minutes at $300^{\circ}\pm 10^{\circ}\text{F}$ and 100 psi.
- f. Obtain 10 CP discs which have been cut out with the carbon fibers parallel to the bonding surfaces.
- g. TCA wipe the CP discs. Allow 30 minutes to dry.
- h. Abrade the CP discs with 180 - 220 grit abrasive material.
- i. TCA wipe the CP discs. Allow 30 minutes to dry.
- j. Bond the CP discs to 10 D6 buttons with a minimum bondline of EA 934NA. Secure with shrink tape.
- k. Cure for a minimum of 180 minutes at 170°F .
- l. TCA wipe both the CP and NBR bonding surfaces. Allow 30 minutes to dry.
- m. Abrade both the CP and NBR bonding surfaces with 180-220 grit abrasive material.

- n. TCA wipe both the CP and NBR bonding surfaces. Allow 30 minutes to dry.
- o. Bond 10 D6/CP buttons to 10 D6/NBR buttons with a 0.060-in. bondline for mixed adhesive. Secure with shrink tape.
- p. Cure for 24 hours at $120^{\circ}\pm 5^{\circ}\text{F}$, then for 168 hours minimum at $75^{\circ}\pm 5^{\circ}\text{F}$.
- q. Condition Set B at $75^{\circ}\pm 5^{\circ}\text{F}$ for two hours minimum before pulling. The crosshead rate shall be 0.5 in./min.
- r. Record the tensile adhesion strength and failure mode of each specimen. Also, examine for voids and note other anomalies.

6.6 Shore A Hardness Tests

- a. Pour mixed adhesive into five aluminum weighing dishes.
- b. Cure the adhesive for 24 hours at $120^{\circ}\pm 5^{\circ}\text{F}$, then for 168 hours minimum at $75^{\circ}\pm 5^{\circ}\text{F}$.
- c. Measure Shore A hardness upon completion of the cure cycle.
- d. Measure Shore A hardness three days after cure.
- e. Measure Shore A hardness eight days after cure.
- f. Measure Shore A hardness 13 days after cure.
- g. Measure Shore A hardness 18 days after cure.
- h. Measure Shore A hardness 23 days after cure.

6.7 "Butterfly" Tests

- ~~a.~~ Fold a heavy 12- by 12-in. piece of kraft paper in half.
- b. Apply a bead of adhesive in the crease of the kraft paper.
- c. Fold the kraft paper in several layers so that the adhesive is sandwiched.
- d. Thoroughly smear the adhesive.
- e. Unfold the kraft paper and observe for unmixed adhesive.

Table I. Meter- and Hand-Mixed Data

MM 1221 HM 1221 MM 1860 HM 1860 Q MM 875 QHM 875

TENSILE ADHESION DATA

AVG (10 SAMPLES) STRENGTH (PSI)

AVG FAILURE MODE:

- % COHESIVE IN POLYSULFIDE
- % ADHESIVE INSULATION/POLY
- % ADHESIVE COMPOSITE/POLY
- % VOIDS

COEFFICIENT OF VARIATION (%)

PEEL DATA

AVG (5 SAMPLES) MAXIMUM STRENGTH (PLI)

AVG FAILURE MODE:

- % COHESIVE IN POLYSULFIDE
- % ADHESIVE INSULATION/POLY
- % VOIDS
- COEFFICIENT OF VARIATION (%)

SHORE A HARDNESS DATA - AVG OF 3 SAMPLES AND 3 READINGS PER SAMPLE

- 8 DAYS
- 18 DAYS
- 23 DAYS

| | | | | | |
|-----|-----|-----|-----|-----|-----|
| 68 | 67 | 204 | 196 | 40 | 57 |
| 99 | 99 | 12 | 44 | 100 | 98 |
| | | 39 | 33 | | |
| | | 49 | 23 | | |
| 1 | 1 | | | | 2 |
| 10 | 14 | 6 | 17 | 40 | 21 |
| 26 | 25 | 24 | 25 | 20 | 18 |
| 100 | 100 | 68 | 90 | 100 | 100 |
| | | 23 | 10 | | |
| | | 9 | 8 | 2 | 5 |
| 5 | 6 | 10 | | | |
| 20 | 18 | 24 | 19 | 19 | 18 |
| 18 | 21 | 28 | 25 | 26 | 26 |
| 19 | 21 | 30 | 32 | 39 | 36 |

Table I. Meter- and Hand-Mixed Data (Continued)

| | <u>MM 1221</u> | <u>HM 1221</u> | <u>MM 1860</u> | <u>HM 1860</u> | <u>MM 875</u> |
|---|----------------|----------------|----------------|----------------|---------------|
| BUTTERFLY TESTS | PASSED | PASSED | PASSED | PASSED | PASSED |
| FLOW RATE (LB/MIN) - AVG OF FOUR MEASUREMENTS @ 90 PSI | 0.22 | 0.22 | 0.2 | 0.91 | 0.91 |
| MAXIMUM FLOW RATE (LB/MIN) @ 90 PSI | 0.31 | 0.31 | 0.23 | 0.96 | 0.96 |
| AVG SPECIFIC GRAVITY MIXED POLY - AVERAGE OF THREE MEASUREMENTS | 1.51 | 1.5 | 1.4 | 1.41 | 1.02 |

NOTATION:

- MM = METER MIXED ADHESIVE
- HM = HAND MIXED ADHESIVE
- 1221 = PR-1221 POLYSULFIDE ADHESIVE
- 1860 = PR-1860 POLYSULFIDE ADHESIVE
- 875 = PS-875 POLYSULFIDE ADHESIVE

@ THE TENSILE AND PEEL SAMPLES OF THE PS-875 WERE NOT EXPOSED TO ELEVATED TEMPERATURE CURE BECAUSE THE CURE OVEN WAS MISTAKENLY TURNED OFF

Table II. Data From Metering Checkout

| | <u>SG BASE (A)</u> | <u>SG CATALYST (B)</u> | <u>A:B WEIGHT</u> | <u>A:B VOLUMETRIC</u> |
|--|--------------------|------------------------|-------------------|-----------------------|
| <u>PR-1221</u> | | | | |
| @ VOLUMETRIC RATIO CALCULATED FROM MEASUREMENTS TAKEN AT LCC | 1.404 | 3.217 | 9.41 | 21.56 |
| # VOLUMETRIC RATIO CALCULATED FROM PRC GIVEN DATA | 1.4 | 2.65 | 10 | 18.93 |
| + ACTUAL VOLUMETRIC RATIO USED IN THE DESIGN OF THE MACHINE | | | | 20.2 |
| <u>PR-1860</u> | | | | |
| @ VOLUMETRIC RATIO CALCULATED FROM MEASUREMENTS TAKEN AT LCC | 1.37 | 1.9 | 8.94 | 12.4 |
| # VOLUMETRIC RATIO CALCULATED FROM PRC GIVEN DATA | 1.32 | 1.95 | 10 | 14.77 |
| + ACTUAL VOLUMETRIC RATIO USED IN THE DESIGN OF THE MACHINE | | | | 12.27 |

Table II. Data From Metering Checkout (Continued)

| | <u>SG BASE (A)</u> | <u>SG CATALYST (B)</u> | <u>A:B WEIGHT</u> | <u>A:B VOLUMETRIC</u> |
|--|--------------------|------------------------|-------------------|-----------------------|
| PS-875 | | | | |
| @ VOLUMETRIC RATIO CALCULATED FROM MEASUREMENTS TAKEN AT LCC | 0.99 | 1.57 | 12.53 | 19.87 |
| # VOLUMETRIC RATIO CALCULATED FROM PRC GIVEN DATA | 0.94 | 1.8 | 10 | 19.15 |
| + ACTUAL VOLUMETRIC RATIO USED IN THE DESIGN OF THE MACHINE | | | | 19.12 |

@ VOLUMETRIC RATIO = (SG CATALYST * SG BASE)/WEIGHT RATIO

WHERE: SPECIFIC GRAVITIES AND WEIGHT RATIO WERE MEASURED AT LIQUID CONTROL CORP

VOLUMETRIC RATIO = (SG CATALYST * SG BASE)/WEIGHT RATIO

WHERE: SPECIFIC GRAVITIES AND WEIGHT RATIO WERE PROVIDED BY PRODUCTS RESEARCH COMPANY

+ THIS VALUE IS THE VOLUMETRIC RATIO THAT PRC PROVIDED LCC FOR THE DESIGN OF THE MACHINE

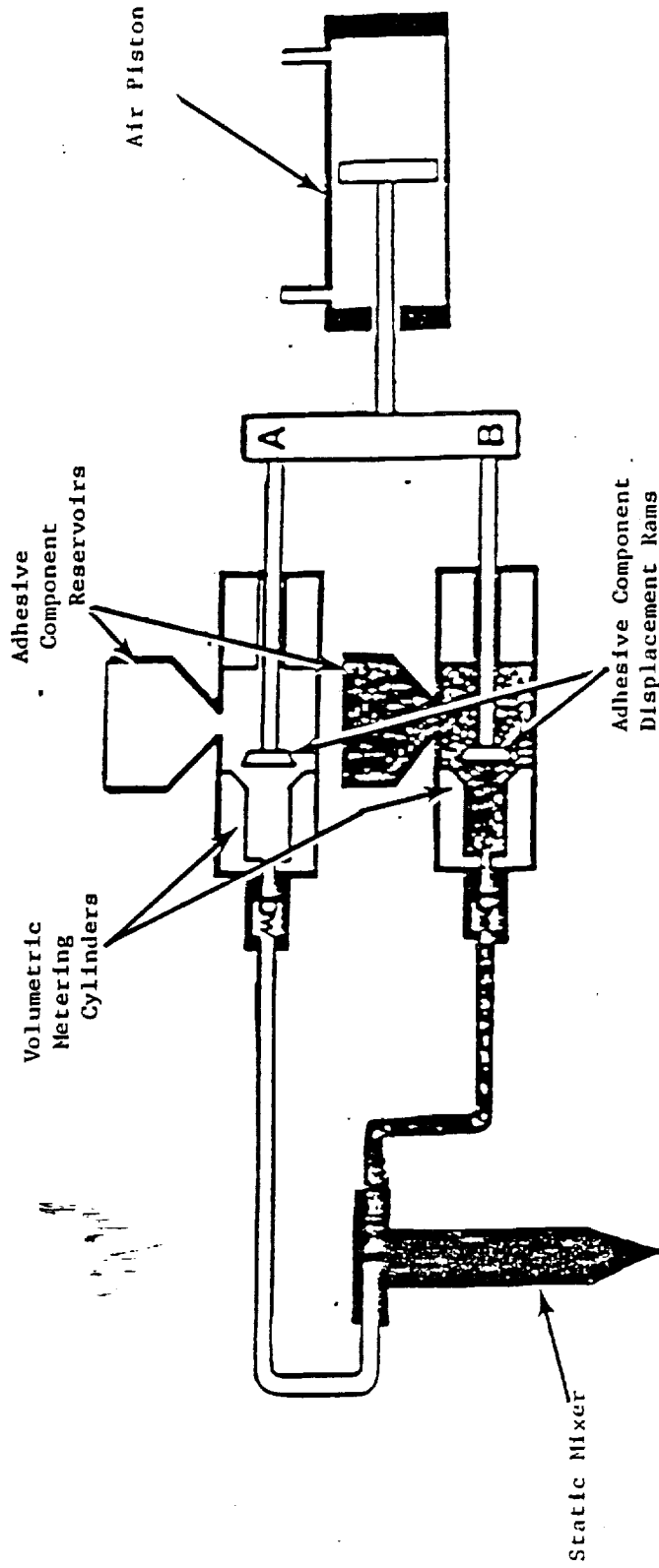


Figure 1. Typical Metering, Mixing, and Dispensing Machine (Fixed Ratio)

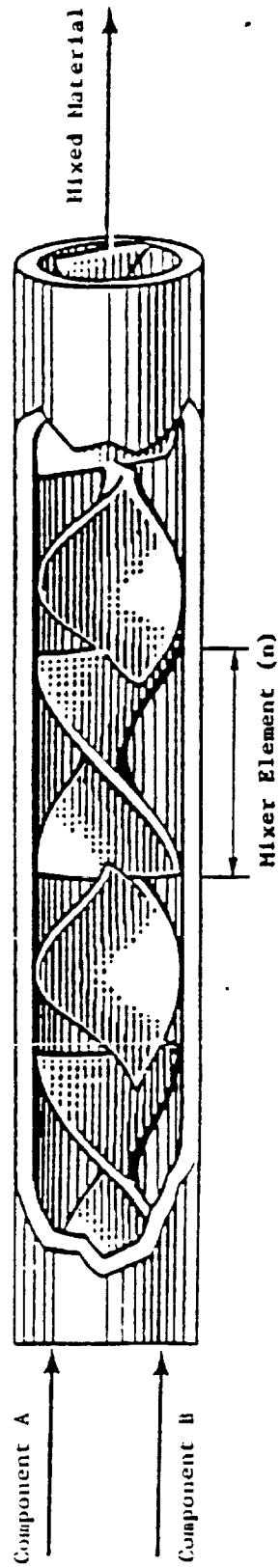


Figure 2. Static Mixer (Provided By The CHEM-MIXX Corporation)

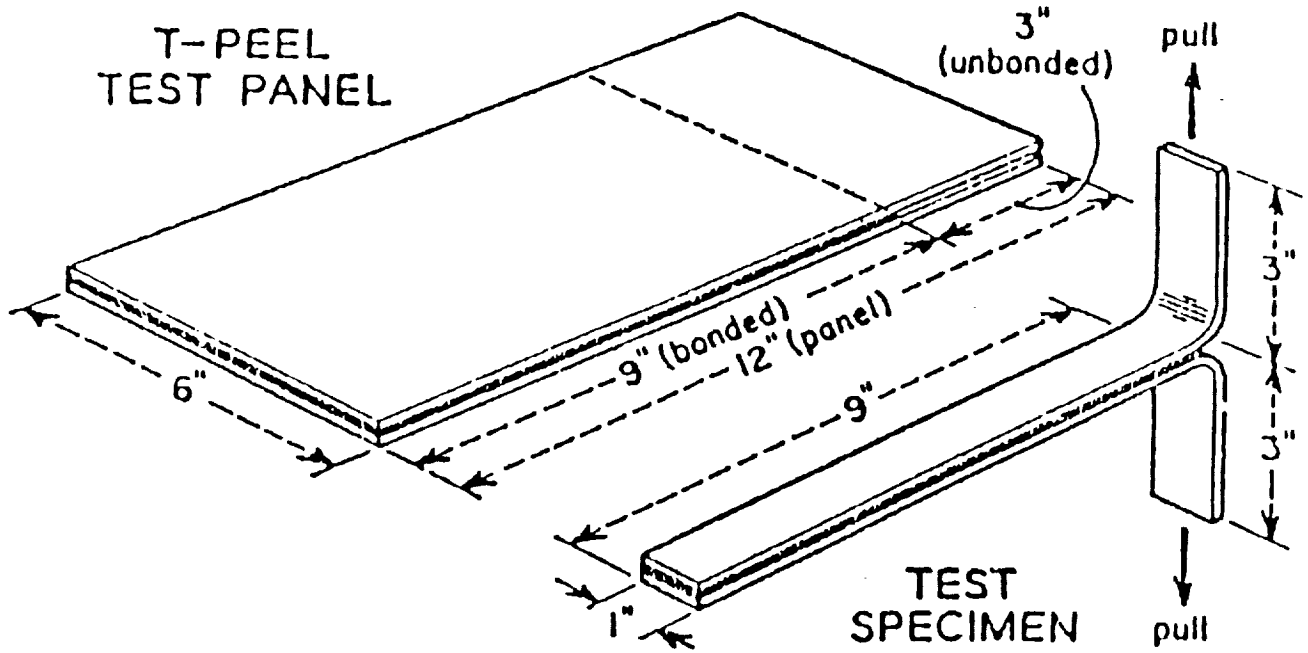


Figure 3. Peel Strength Specimen

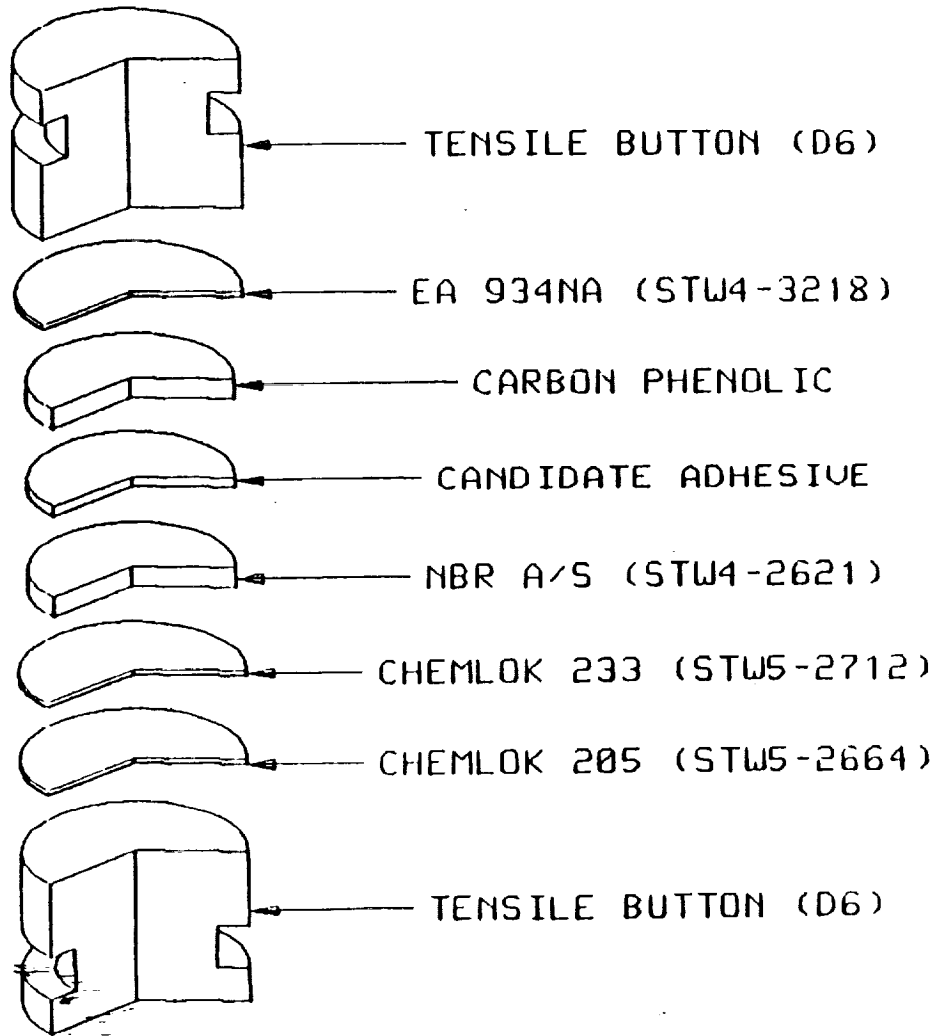


Figure 4. Tensile Button Configuration

REVISION _____

FORM TC 7994-310 (REV 2-88)