

NBS  
Publi-  
cations

Reference

NBSIR 82-2535

NAT'L INST. OF STAND & TECH



A11106 259631

# Development of A Test Apparatus and Method for Measuring Adhesion of Protective Coatings

---

U.S. DEPARTMENT OF COMMERCE  
National Bureau of Standards  
National Engineering Laboratory  
Center for Building Technology  
Building Materials Division  
Washington, DC 20234

July 1982

QC

100

.U56

82-2535

1982

Prepared for:

Tri-Services Building Materials Investigation Program Committee



JUL 27 1982

Not acc. Ref

QC100

.456

NO. 82-2535

1982

NBSIR 82-2535

**DEVELOPMENT OF A TEST APPARATUS  
AND METHOD FOR MEASURING  
ADHESION OF PROTECTIVE COATINGS**

James F. Seiler  
Mary E. McKnight  
Larry W. Masters

U.S. DEPARTMENT OF COMMERCE  
National Bureau of Standards  
National Engineering Laboratory  
Center for Building Technology  
Building Materials Division  
Washington, DC 20234

July 1982

Prepared for:  
Tri-Services Building Materials Investigation Program Committee



---

**U.S. DEPARTMENT OF COMMERCE, Malcolm Baldrige, *Secretary***  
**NATIONAL BUREAU OF STANDARDS, Ernest Ambler, *Director***



## ABSTRACT

A pneumatic test apparatus and associated test method for measuring the adhesion of coatings have been developed with particular emphasis on: 1) overcoming some of the shortcomings of existing tests; and 2) providing a method which can provide quantitative information for both laboratory and field applications.

The test apparatus utilizes compressed air to apply a constant rate of load to a stainless steel loading fixture (button) which is bonded with an adhesive to the surface of a protective coating. The tensile force required to remove the loading fixture from the coating is measured. Assuming the level of adhesion of the adhesive to the coating is greater than that of the coating to the substrate, the tensile force at break provides a measure of the coating adhesion. Laboratory studies with two coating materials have been performed to assess the method.

This report describes the test apparatus and associated test method and presents test data obtained to date, proposed modifications to the initial test apparatus design, and additional research needs. An Instruction Manual for use of the test apparatus is included in the Appendix.

## ACKNOWLEDGMENT

The authors gratefully acknowledge: 1) the support of the Tri-Services Building Materials Investigation Program Committee in carrying out this research; 2) the assistance of the staff of the NBS Shops in fabricating portions of the test apparatus; and 3) the technical assistance provided by Mr. Jack Lee in obtaining test data.

## TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT .....	iii
ACKNOWLEDGMENT .....	iv
LIST OF FIGURES .....	vi
LIST OF TABLES .....	vi
1. INTRODUCTION .....	1
1.1 Background .....	1
1.2 Objectives .....	2
2. TEST APPARATUS .....	2
2.1 Piston Assembly .....	2
2.2 Loading Fixture .....	3
2.3 Rate of Load Device .....	3
2.4 Air Handling System .....	3
2.5 Method for Using Test Apparatus .....	6
3. EXPERIMENTAL PROCEDURES .....	8
3.1 Surface Preparation .....	8
3.2 Laboratory Tensile Testing Machine .....	8
3.2.1 Bonding the Loading Fixture .....	8
3.2.2 Effect of Adhesive Thickness .....	8
3.2.3 Effect of Substrate Thickness .....	10
3.2.4 Calibration of the Pneumatic Test Apparatus .....	10
3.2.5 Coating Tests .....	10
3.3 Pneumatic Tester .....	10
4. RESULTS AND DISCUSSION .....	12
4.1 Effect of Adhesive Thickness .....	12
4.2 Effect of Substrate Thickness .....	12
4.3 Calibration of Pneumatic Tester .....	12
4.4 Coating Adhesion Tests .....	12
5. CONCLUSIONS .....	17
6. REFERENCES .....	18
APPENDIX - INSTRUCTION MANUAL FOR THE USE OF THE PNEUMATIC FIELD ADHESION TESTER	



LIST OF FIGURES

	<u>Page</u>
Figure 1. Field adhesion tester in carrying case .....	4
Figure 2. Schematic drawing of the pneumatic test apparatus .....	5
Figure 3. Rate of load mechanism .....	7
Figure 4. Schematic drawing illustrating procedure used for measuring adhesion with a laboratory tensile testing machine .....	9
Figure 5. Apparatus used to calibrate pneumatic tester .....	11
Figure 6. Thickness of epoxy adhesive vs stress at rupture .....	13
Figure 7. Substrate thickness vs stress at rupture for epoxy adhesive ..	14
Figure 8. Calibration curve for pneumatic tester .....	15

LIST OF TABLES

	<u>Page</u>
Table 1. Results of Coating Adhesion Tests Using both Laboratory Tensile Machine and Pneumatic Test Apparatus .....	16



## 1. INTRODUCTION

### 1.1 BACKGROUND

The primary purposes of coatings used on the exterior surfaces of buildings and structures are to provide a protective barrier between the substrate and the environment, and to provide a decorative finish. One of the most important performance requirements of protective coatings is that they adhere to the substrate, both when initially applied and during exposure for extended periods of time in the environment. Loss of adhesion leads to delamination (peeling) of the coating and, therefore, to the inability of the coating to provide a protective barrier to the substrate from the environment.

Because coating adhesion is an essential attribute in achieving an effective protective barrier, it must be measured in generating: 1) the performance and durability data needed for effective selection criteria of protective coatings; and 2) the data needed for decision-making pertaining to repair and replacement of existing coatings on buildings and structures. Therefore, coating adhesion measurements are needed in the laboratory and field.

Tests have been developed to aid in assessing adhesion of coatings. These include pull-off by tape, scratch-resistance, and resistance to cracking after bending the substrate over a mandrel. While these tests are useful as rapid screening tools, they do not provide adequate quantitative data on coatings adhesion. Another test, frequently termed the "pull-off test," was developed to aid in achieving improved quantitative data. The test consists of using an adhesive to bond a metallic loading fixture (button) to the surface of the coating. After the adhesive cures, the loading fixture is pulled from the substrate. Assuming adhesion to the coating is greater than that of the coating to the substrate, coating adhesion can be measured. Two standard test methods, ISO 4624 and DIN 53 232 [1,2], describe procedures for performing laboratory pull-off tensile tests. Previous work [3,4] shows that repeatability of pull-off tests ranges from 5-30 percent, depending on the procedure used.

Often it is necessary to measure adhesion in the field and commercial devices are available using a pull-off method. However, several problems have been identified which may affect the accuracy and reproducibility of these measurements. These include:

1. The force needed to remove the loading fixture is not applied at a constant rate.
2. The calibrations of the testers are not always linear [5].
3. Calibration of a tester may change with time.
4. The tensile force is not necessarily coaxial with the loading fixture which may cause paint peeling.

In addition, other factors which could affect test results from the pull-off method have not been fully addressed. These include: 1) type, thickness, and cure of adhesive used for bonding the loading fixture to the coating; and 2) effect of substrate composition and thickness.

The need for reliable measurements of coating adhesion, combined with the shortcomings of existing test methods, requires research to develop an improved field test method.

## 1.2 OBJECTIVES

The objectives of this study were to:

1. Develop a new and improved field test apparatus and associated test method for use in measuring adhesion of protective coatings,
2. Obtain preliminary laboratory data to assess the effectiveness of the test apparatus,
3. Prepare an Instruction Manual for use of the test apparatus in measuring adhesion of coatings in the field, and
4. Identify the limitations of the apparatus and/or test method, and recommend research needed to overcome the limitations.

## 2. TEST APPARATUS

Because of its ability to provide quantitative data on the tensile force required to remove coatings from their substrates, the pull-off method was selected for use in this study. The study focused initially on devising a test apparatus that could overcome the problems observed with existing devices which utilize the pull-off method. Other criteria for the test apparatus were that it be portable and reliable.

A pneumatic adhesion tester, suitable for use in field applications, was designed and built to measure the tensile force required to pull off a stainless steel loading fixture bonded to a coating surface. A picture of the instrument as it appears in the carrying case is shown in figure 1. The primary elements of the test apparatus are a piston assembly, a loading fixture, a rate of load device, and an air handling system. Figure 2 is a schematic drawing of the test apparatus.

### 2.1 PISTON ASSEMBLY

The piston assembly [6] permits the load to be applied to the loading fixture. It includes a pulling cap and a piston.

The pulling cap is a 50.8 mm (2 in) diameter 31.8 mm (1.25 in) high, stainless steel cylinder with a smooth top face, a groove cut out around the sides, and a 9.5 mm (0.375 in) threaded hole tapped in the center of the bottom. A

concentrically located ring protrudes beyond the bottom 1.6 mm (0.0625 in) and has an outside diameter of less than 28.6 mm (1.125 in) with an inside diameter of more than 19 mm (0.75 in). This ring centers the piston with the pulling cap. The pulling cap is attached with screw threads to the loading fixture.

The piston consists of a 146 mm (5.75 in) diameter, 4.8 mm (.1875 in) thick, stainless steel plate, with an air inlet on the top side, a mechanically attached membrane on the bottom side, and a 28.6 mm (1.125 in) hole through the center. Compressed air, which is forced between the piston plate and the piston membrane, provides the load needed to lift off the loading fixture.

## 2.2 LOADING FIXTURE

The loading fixture is a specially machined stainless steel "button". The cylindrical section of the loading fixture has a diameter of 19 mm (0.75 in) and is 6.4 mm (0.25 in) thick. The bottom face of the cylinder has four spacers or "feet" machined to a specified length and symmetrically located around the perimeter. The area of the face is  $285 \text{ mm}^2$  ( $0.442 \text{ in}^2$ ) and the total area of the four feet is less than  $1.3 \text{ mm}^2$  ( $0.002 \text{ in}^2$ ). A concentrically located, threaded stud, 9.5 mm (0.375 in) in diameter and height is machined into the top of the cylinder. The feet ensure that the loading fixture will set normal to the substrate and that the adhesive between the test surface and the loading fixture face will be uniform in thickness. The threaded stud allows the pulling cap to be screwed into place.

## 2.3 RATE OF LOAD DEVICE

The rate of load mechanism is shown in figure 3. It consists of a precision air pressure gauge with a circular dial readout (recording dial), a throttle, and a rigid, transparent pacer wheel which is attached coaxially to the recording dial. The wheel is driven by a gear motor-pulley assembly. Large changes in speed of the pacer wheel are made by replacing pulley wheels, and small changes are made by adjusting the motor voltage. A constant rate of load is provided by manually controlling the rate of increase of air pressure so that the indicating pointer on the recording dial follows a mark near the perimeter of the moving pacer wheel. The maximum air pressure obtained during a test is measured by a pointer on the recording dial.

## 2.4 AIR HANDLING SYSTEM

A schematic of the compressed air handling system is included in figure 2. It consists of a regulated supply of compressed air, air pressure gauges, throttle hose, quick disconnects, and a valve to protect the precision air pressure recording dial. The umbilical hose is 7.6 m (25 ft) long so that tests can be performed at a distance from the controls.



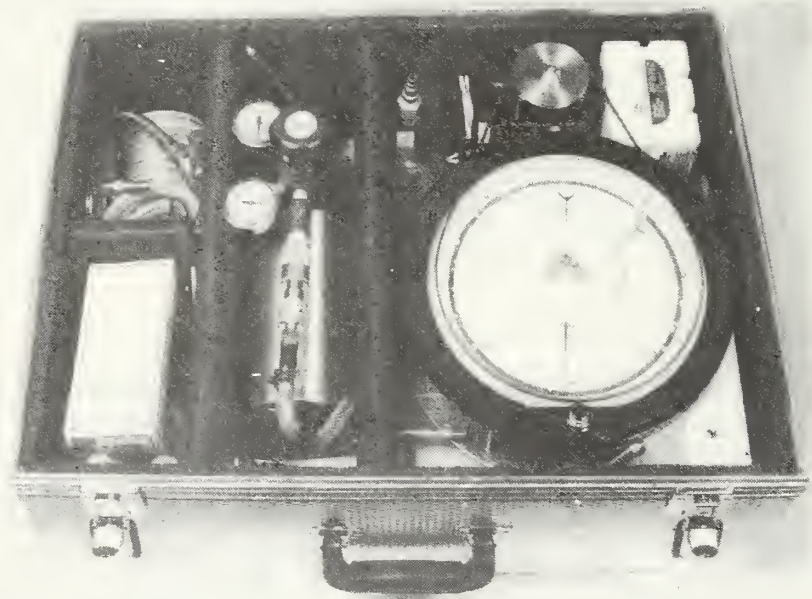


Figure 1. Field adhesion tester in carrying case

# ADHESION TESTER SCHEMATIC

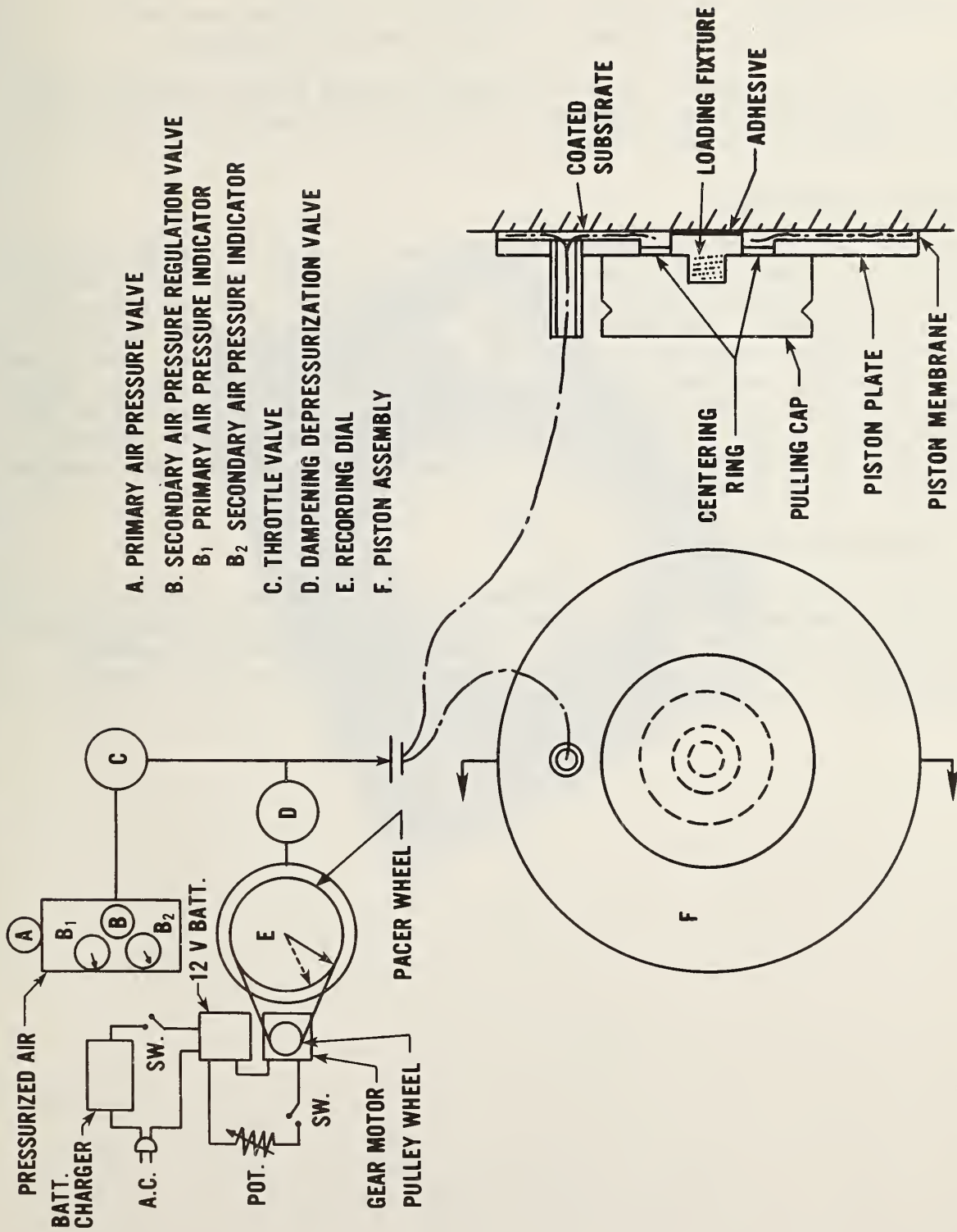


Figure 2. Schematic drawing of the pneumatic test apparatus

to the loading fixture, the steadily increasing air pressure eventually ruptures the adhesive bond between the coating and the substrate.

Assemblies can be designed to span the range of forces needed to measure the adhesion strength of various coatings by changing the diameter of either or both the loading fixture and piston.

### 2.3 RATE OF LOAD DEVICE

A picture of the rate of load mechanism is shown in figure 3. It consists of a precision air pressure gauge with a circular dial readout (recording dial), a throttle, and a rigid, transparent pacer wheel which is attached coaxially to the recording dial. The wheel is driven by a gear motor-pulley assembly. Large speed changes to the pacer wheel are made by replacing pulley wheels, and small speed changes are made by adjusting the motor voltage. A constant rate of load is provided by controlling the air pressure indicating pointer on the recording dial, so that it follows a mark near the perimeter of the moving pacer wheel. The maximum air pressure obtained during a test is also measured by a pointer on the recording dial.

### 2.4 AIR HANDLING SYSTEM

A schematic of the compressed air handling system is included in figure 2. It consists of a regulated supply of compressed air, air pressure gauges, throttle hose, quick disconnects, and a valve to protect the precision air pressure recording dial. The umbilical hose is 7.6 m (25 ft) long so that tests can be performed at a distance from the controls.



Figure 3. Rate of load mechanism



### 3. EXPERIMENTAL PROCEDURES

Laboratory tests were performed with a laboratory tensile testing machine to 1) develop a uniform method for bonding the loading fixture, 2) assess the effect of adhesive thickness on tensile strength at rupture, 3) assess the effect of substrate thickness on tensile strength at rupture, and 4) calibrate the pneumatic test apparatus.

Pull-off tests were carried out on two coating systems using both the tensile testing machine and the pneumatic test apparatus to assess the accuracy and reproducibility of the pneumatic apparatus. The procedures used and the method of surface preparation are described below.

#### 3.1 SURFACE PREPARATION

The surfaces of the steel substrates were prepared by sandblasting one group and by roughening the others with sandpaper. Acetone was used to remove oil and other surface debris. Also, the faces of the loading fixtures were sandblasted and wiped with acetone. The surfaces of the coatings were lightly sandpapered and cleaned with alcohol.

#### 3.2 LABORATORY TENSILE TESTING MACHINE

A laboratory tensile testing machine, adapted for coating adhesion pull-off tests (see figure 4), was used to refine the method of determining the bond strength of coatings. A loading fixture was designed which, when bonded in place, sat normal to the substrate and retained a uniform adhesive thickness and area.

##### 3.2.1 Bonding the Loading Fixture

A systematic procedure was developed for bonding a loading fixture to a substrate and involved the use of a sleeve. In these experiments, the adhesive used to bond the loading fixture to the substrate was a structural strength amine epoxy which cures in 24 hours at room temperature or in 40 minutes at 70°C (158°F) with a 1 hour cool down time. A polytetrafluoroethylene sleeve was used to minimize the effect of adhesive spill-over. The detailed procedure used to bond the loading fixture to a substrate is described in the Appendix.

##### 3.2.2 Effect of Adhesive Thickness

The effect of epoxy adhesive thickness on the test results was determined using the laboratory tensile testing machine. Loading fixtures having a 19 mm (0.75 in) diameter were constructed. The feet (or spacers) on the loading fixtures were varied to span a range of adhesive thicknesses. The procedure described in the Appendix was used to bond the loading fixtures to a large plate of 6.35 mm (0.25 in) thick steel. The cross-head speed of the tensile testing machine was 1 mm/min (0.04 in/min), which corresponds to a rate of stress of approximately 0.39 MPa/s (56 psi/s). The load at rupture was measured and recorded.

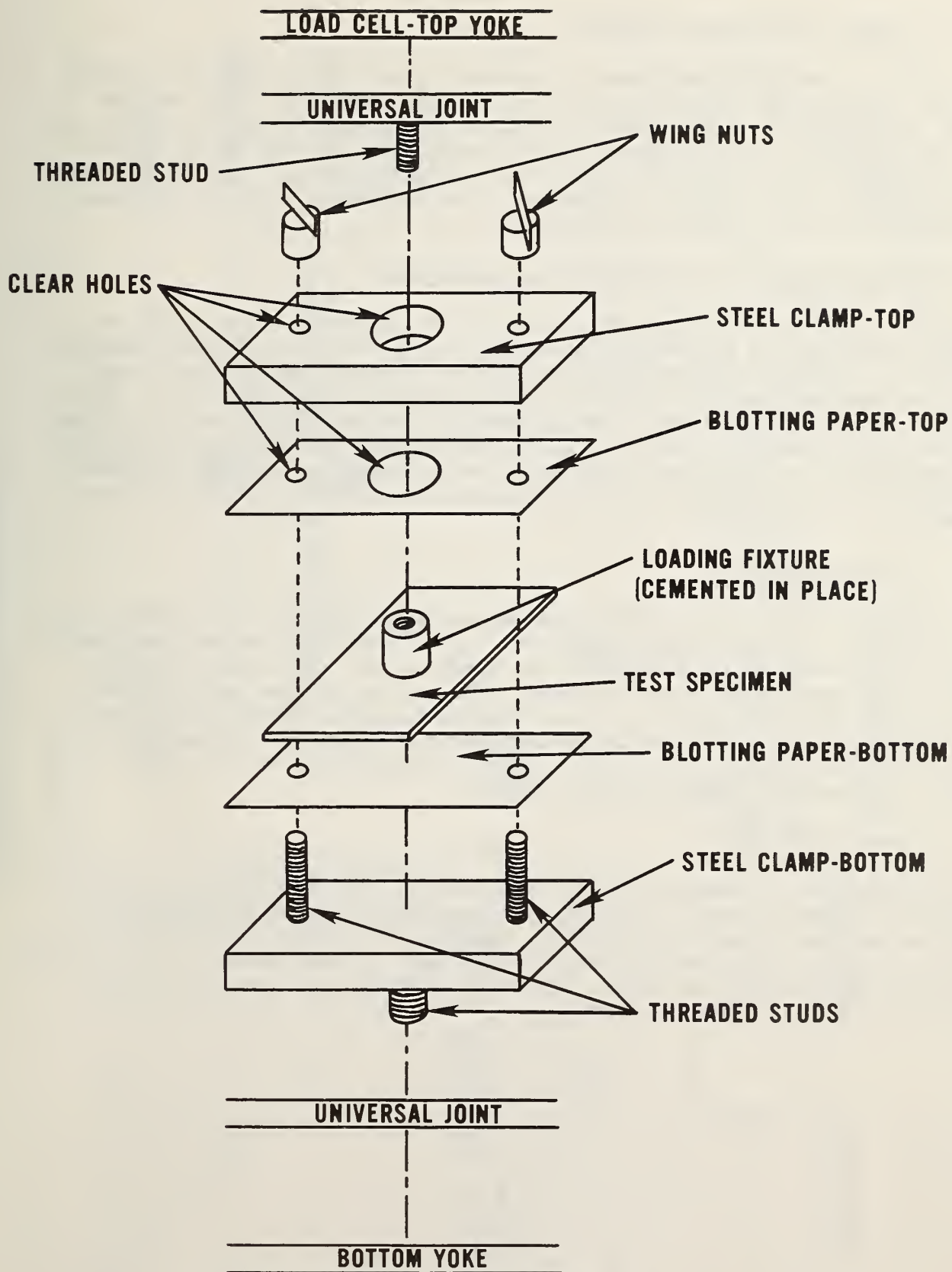


Figure 4. Schematic drawing illustrating procedure used for measuring adhesive with a laboratory tensile testing machine.

### 3.2.3 Effect of Substrate Thickness

The effect of substrate thickness on apparent adhesion strength was assessed because a wide range of thicknesses could be encountered in field testing. Three thicknesses of steel were used: 6.4 mm (0.25 in), 3.2 mm (0.125 in), and 0.8 mm (0.031 in). The thickness of the adhesive was 0.2 mm (0.008 in) and the loading fixture faces were 19 mm (0.75 in) in diameter. Six measurements were taken for each substrate thickness. The cross-head speed of the tensile testing machine was 1 mm/min (0.04 in/min).

### 3.2.4 Calibration of the Pneumatic Test Apparatus

A special apparatus was constructed so that the pneumatic tester could be calibrated using the load measuring system of the laboratory tensile testing machine. A schematic of the apparatus and the means to measure the force being applied are shown in figure 5. The apparatus was constructed to simulate an actual adhesion test. The calibration procedure involved forcing air between the membrane and metallic plate (piston) and measuring the force that is exerted on the load cell. This force was measured for several different pressures on the recording dial and recorded on the tensile testing machine readout.

### 3.2.5 Coating Tests

The adhesion of two alkyd coating systems was measured with the tensile testing machine. One coating system, applied on a steel substrate was an alkyd inhibitive primer with an alkyd topcoat; the other was the alkyd topcoat applied directly to sandblasted steel surfaces. The coating systems were applied to six 76 x 152 x 6.4 mm (3 x 6 x 0.25 in) steel plates and allowed to cure for 7 days at 21°C (70°F) and 50 percent relative humidity. Three pull-off tests were performed on each plate. The cross-head speed of the tensile testing machine was 1 mm/min (0.04 in/min).

### 3.3 PNEUMATIC TESTER

The measurements on coated substrates described in Section 3.2.5 were also made using the pneumatic tester. Surface preparation and curing conditions were the same as those described in 3.2.5. Eighteen 152 x 152 x 6.4 mm (6 x 6 x 0.25 in) steel plates were prepared for each set of measurements. One pull-off test was performed on each plate. The rate of stress was either 0.32 MPa/s (46 psi/s) or 0.39 MPa/s (56 psi/s).

# SCHEMATIC FOR CALIBRATING P.T.

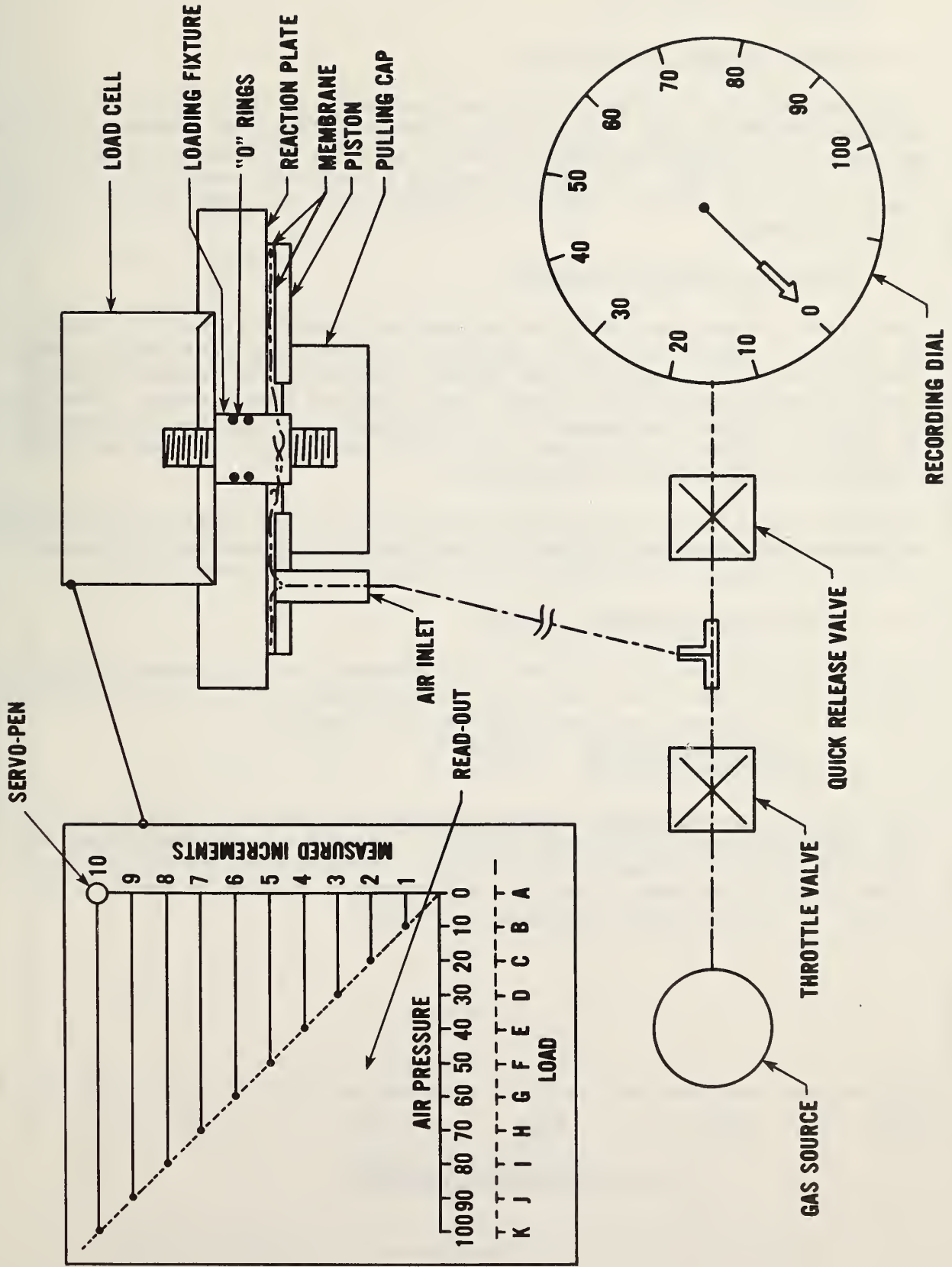


Figure 5. Apparatus used to calibrate pneumatic tester



## 4. RESULTS AND DISCUSSION

### 4.1 EFFECT OF ADHESIVE THICKNESS

The effect of the thickness of the epoxy adhesive on the stress at rupture is shown in figure 6. All failures were cohesive (within the adhesive). As can be seen, the strength did not vary appreciably for thicknesses of adhesive of 0.1 to 0.5 mm (0.004 - 0.020 in). This is a desirable property in pull-off tests, since the adhesive thickness, within a certain range, will not affect tensile strength.

### 4.2 EFFECT OF SUBSTRATE THICKNESS

The effect of substrate thickness on apparent adhesive strength is shown in figure 7. Since the tensile strength at rupture decreased with a reduction in substrate thickness, this seems to indicate that substrates which bend introduce peeling forces which lower apparent bond strength. The type of failure changed from cohesive to a combination of cohesive and adhesive as substrate thickness decreased. This is an additional indication of substrate bending which can cause peeling.

The fact that tensile strength obtained from the tests varies with substrate thickness could present problems in obtaining field measurements because a range of substrate thicknesses could be expected to be encountered.

### 4.3 CALIBRATION OF PNEUMATIC TESTER

A calibration curve for a 127 mm (5 in) diameter piston is shown in figure 8. A straight line was least square fit to the points. The curve was repeatable to within < 1 percent from one calibration to another.

### 4.4 COATING ADHESION TESTS

The results of the coating tests using both the laboratory tensile machine and the pneumatic tester are shown in table 1. Eighteen measurements were made to estimate the mean strength. The tensile testing machine and the pneumatic tester yielded comparable results. Importantly, as shown in table 1, both methods also yield comparable modes of failure.

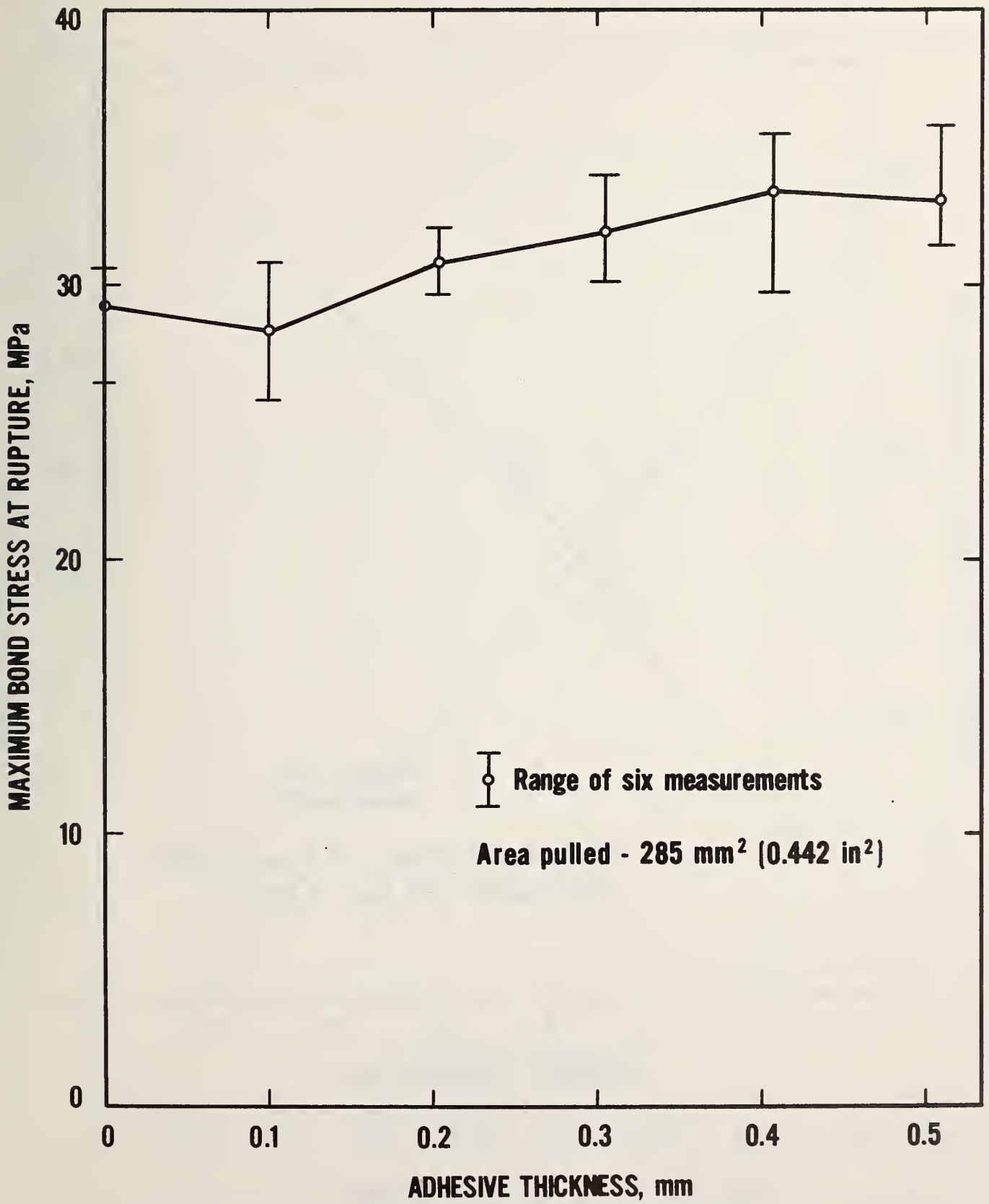


Figure 6. Thickness of epoxy adhesive vs stress at rupture

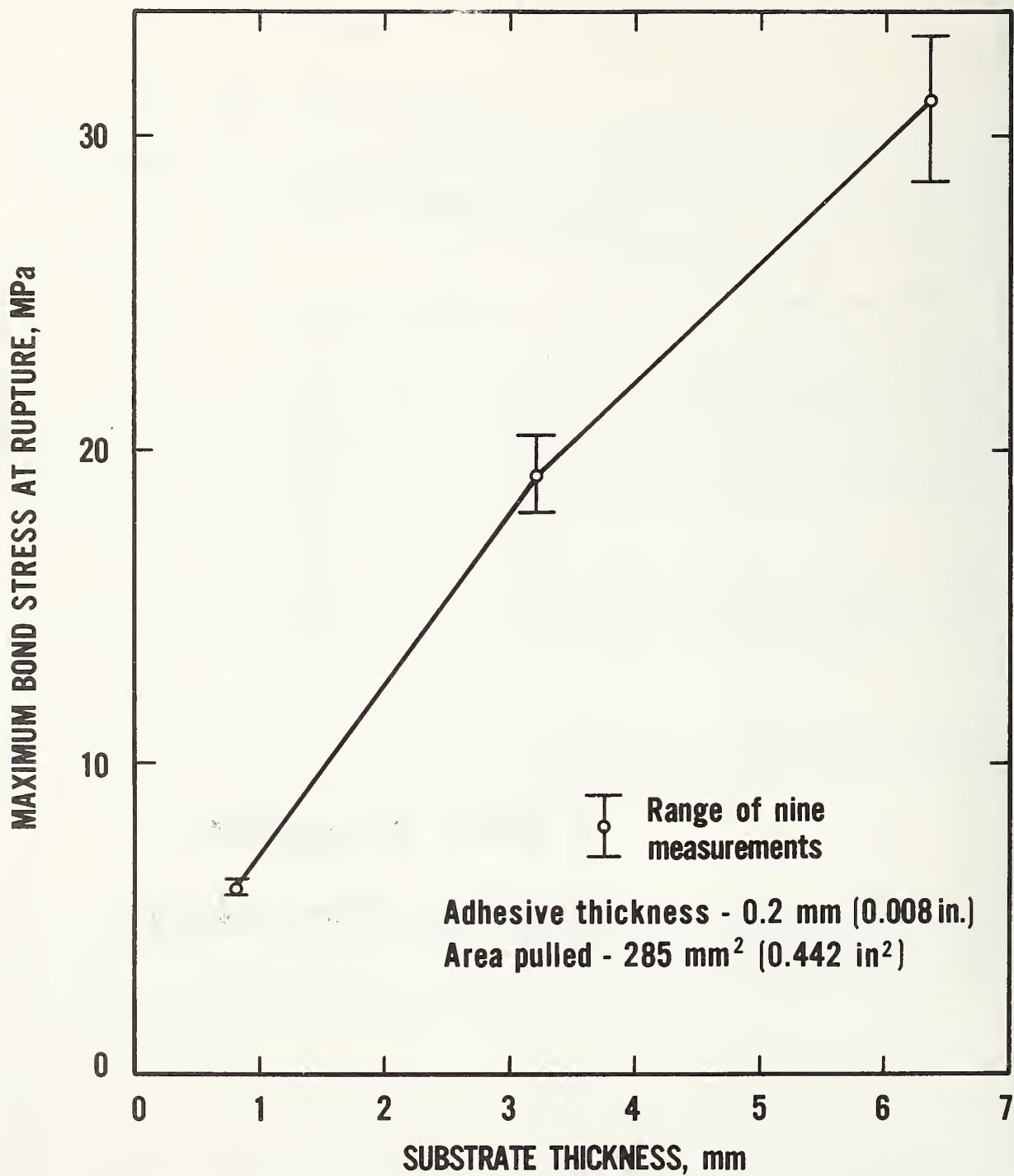


Figure 7. Substrate thickness vs stress at rupture for epoxy adhesive



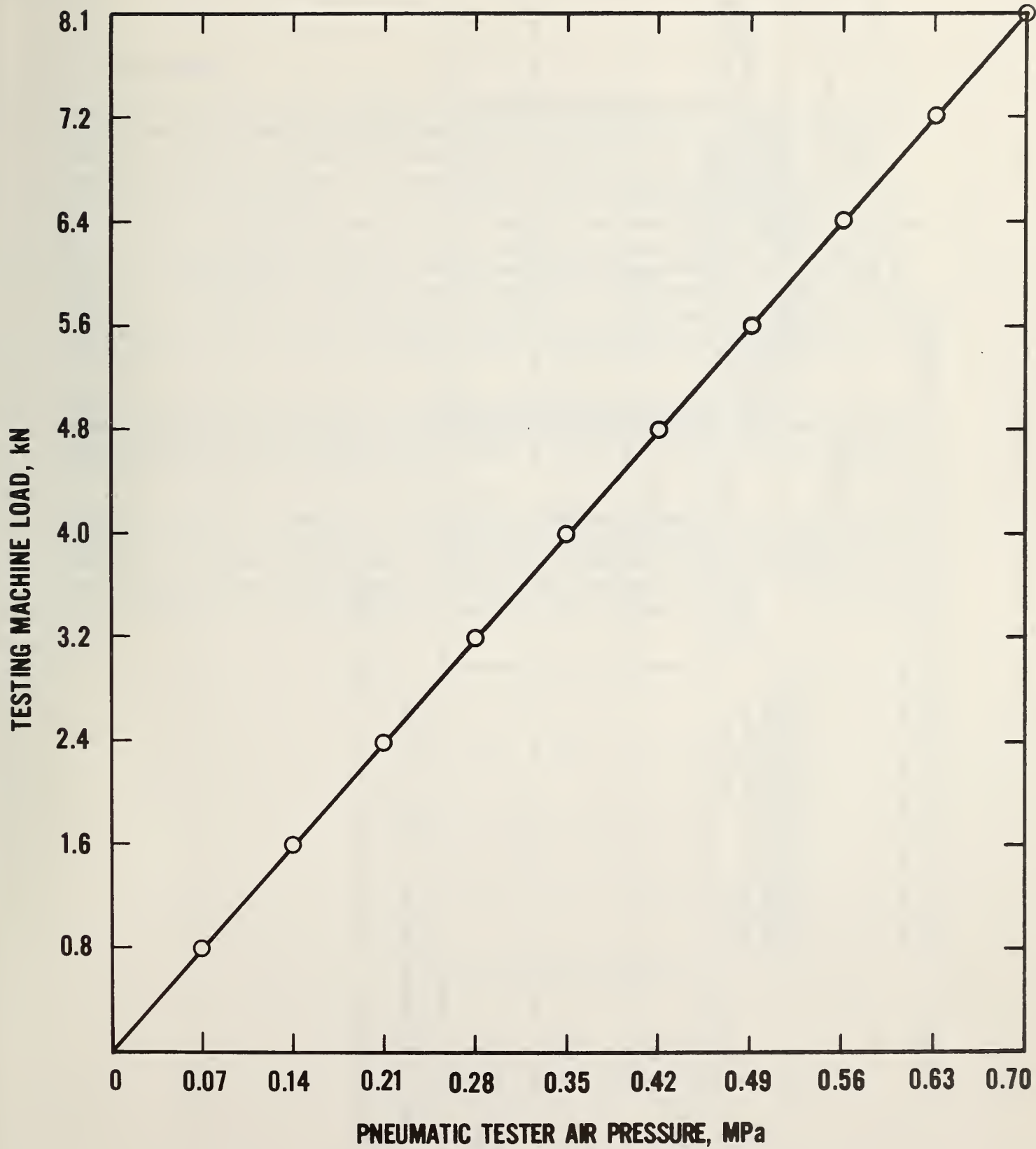


Figure 8. Calibration curve for pneumatic tester

Table 1. Results of Coating Adhesion Tests Using Laboratory Tensile Testing Machine and Pneumatic Test Apparatus

Coating Description	Test Instruments							
	Laboratory Tensile Machine				Pneumatic Tester			
	$\bar{x}$ , MPa <sup>1</sup>	Standard Deviation, MPa	Coefficient of Variation, Percent	Coating Failure <sup>2</sup>	$\bar{x}$ , MPa <sup>1</sup>	Standard Deviation, MPa	Coefficient of Variation, Percent	Coating Failure <sup>2</sup>
Alkyd Topcoat, Experiment 1	12.3	1.0	8.0	C	12.9	1.3	10.0	C
Alkyd Topcoat, Experiment 2	10.7	.7	7.0	C	12.1	1.3	11.0	C
Alkyd Topcoat & Alkyd Primer	6.9	.6	9.0	A	6.6	.8	12.0	A

<sup>1</sup>  $\bar{x}$  is the mean of 18 measurements, 1 MPa = 145 psi.

<sup>2</sup> Kind of failure noted, C - mostly cohesive, A - mostly adhesive.

## 5. CONCLUSIONS

The preliminary test data indicate that the pneumatic test apparatus is an effective and reliable tool for measuring the tensile forces required to remove protective coatings from metallic substrates. In particular, the pneumatic test apparatus overcomes many of the problems associated with existing pull-off devices: 1) it utilizes a constant rate of loading, 2) it has a linear calibration curve, 3) it removes the possible errors from a time dependent calibration curve, and 4) it minimizes peel forces. The repeatability of measurements obtained using the field tester was about the same as that with a laboratory tensile testing machine. The associated test method, described in detail in the Appendix, provides a procedure for obtaining results in the field. The size and weight of the test apparatus make it conducive for field use.

The thickness of the adhesive used for bonding the loading fixture has been shown to have little effect on the tensile strength of the adhesive obtained. The importance of the rigidity of a steel substrate on the values of the load at rupture has been demonstrated.

Further research is needed to 1) automate the loading mechanism so that operator error is minimized, 2) modify the piston assembly for use on curved surfaces, 3) assess the effect of substrate thickness on test results, 4) assess other adhesives for use in bonding the loading fixture, and 5) assess the performance of the apparatus in actual field tests.

6. REFERENCES

1. "Paints and Varnishes - Pull-off Test for Adhesion," International Standard, ISO 4624-1978(E), International Organization for Standardization.
2. "Messung de Haftestigkeit von Anstrichen und ähnlichen Beschichtungen nach der Abreissmethode," DIN 53 232-1973.
3. Sickfeld, J., "Experimental Aspects of Adhesion Testing, A IUPAC Study," J. Oil Col. Chem. Assoc., 61, 292 (1978).
4. Schwab, L. K. and Drisko, R. W., "Relation of Steel Surface Profile and Cleanliness to Bonding of Coatings," Materials Performance, 32, May 1981.
5. Bleile, H. and Rodgers, S., "Tensile Adhesion Testing for Measuring Coating Durability," Proceedings of the 2nd Int. Conf. on Durability of Building Materials and Components, National Bureau of Standards, September 1981.
6. Patent Pending No. 272231, Pneumatic Adhesion Tester, U.S. Patent Office, Washington, D.C.

APPENDIX

INSTRUCTION MANUAL FOR THE USE OF  
THE PNEUMATIC FIELD ADHESION TESTER



TABLE OF CONTENTS

	<u>Page</u>
INTRODUCTION .....	A-2
LIST OF APPENDIX FIGURES .....	A-3
INSTRUCTIONS FOR USING TESTER .....	A-4
I. PREPARATION FOR TESTING .....	A-4
II. TESTING MACHINE CONTROLS .....	A-9
III. PRE-TESTING .....	A-11
IV. TESTING .....	A-13



LIST OF APPENDIX FIGURES

	<u>Page</u>
Figure A-1. Materials for attaching loading fixture .....	A-4
Figure A-2. Loading fixtures, shims, and cleaning sleeves .....	A-5
Figure A-3. Clamping the loading fixture .....	A-8
Figure A-4. Controls - recording dial and pacemaker assembly .....	A-9
Figure A-5. Internal compressed air supply .....	A-10
Figure A-6. Pneumatic piston assembly .....	A-12

## INTRODUCTION

The purpose of this instruction manual is to provide stepwise directions on the use of the Pneumatic Field Adhesion Tester (PFAT); the manual is specifically directed toward measuring the adhesion of protective coatings in the field.

The manual is divided into four parts: I. Preparation for Testing; II. Testing Machine Controls; III. Pre-Testing; and IV. Testing. Each part is subdivided into stepwise procedures.

PFAT consists of four major parts:

- 1) the loading fixture which is bonded to the coated surface
- 2) the piston assembly which is attached to the loading fixture
- 3) the compressed air supply which provides the force required to perform the test, and
- 4) the rate of loading mechanism which allows the force to be increased at a constant rate as well as the means to measure the force at rupture of the loading fixture.

Each of these major parts will be described in detail in the instructions.

## INSTRUCTIONS FOR USING THE TESTER

### I. PREPARATION FOR TESTING

The materials for this process are shown in figure A-1, and are found in the left front section of the PFAT case.

#### Step I-1. Preparing the Surface of the Coating

- (1) Using a gauze pad dampened with distilled water, wipe debris from the area of the coated surface to be tested.
- (2) Lightly sand the test area with 200 grit sandpaper.
- (3) Using a gauze pad dampened with ethyl alcohol, wipe the sanded test area. Next, use fresh dry gauze pads and wipe the area until dry. The test area must be completely clean and dry before bonding the loading fixture to the coating.

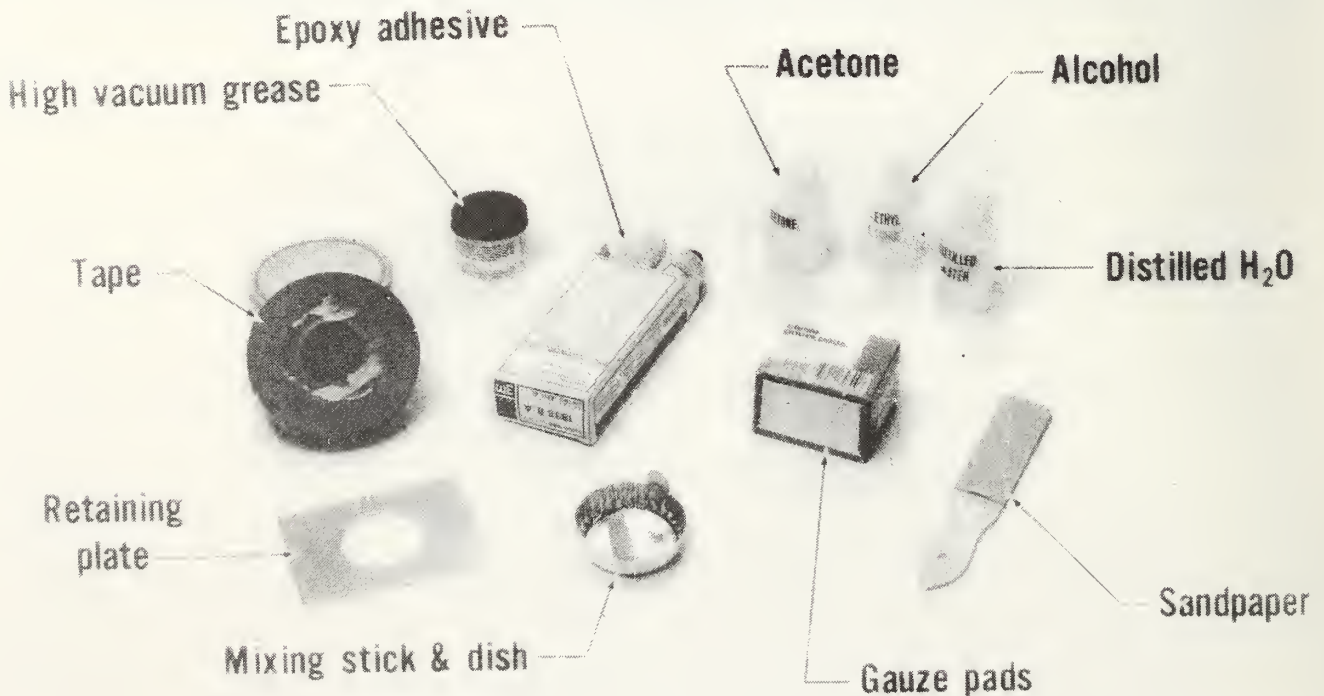


Figure A-1. Materials for attaching loading fixture

## Step I-2. Preparing the Loading Fixture

The loading fixture is shown in figure A-2 and can be found in the left front section of the PFAT case.

NOTE: Caution must be exercised when handling loading fixtures. Do not touch the fixture face. Oil from the skin, acting as a release agent, will cause premature bond failure between the adhesive and the loading fixture.

- (1) Remove the loading fixture and cleaning sleeve from their paper towel wrapping. (If loading fixtures have not been cleaned from previous use, go to step I-5 before proceeding further.)
- (2) Insert the loading fixture into the cleaning sleeve so that the fixture face protrudes about 3 mm (0.125 in) beyond the knife-shaped end of the sleeve.
- (3) Using a clean, dry gauze pad, wipe the face of the loading fixture with acetone.
- (4) Using another clean, dry gauze pad, spread liquid detergent around the knife-shaped end of the cleaning sleeve.

NOTE: It is very important that no liquid detergent gets on the loading fixture face.

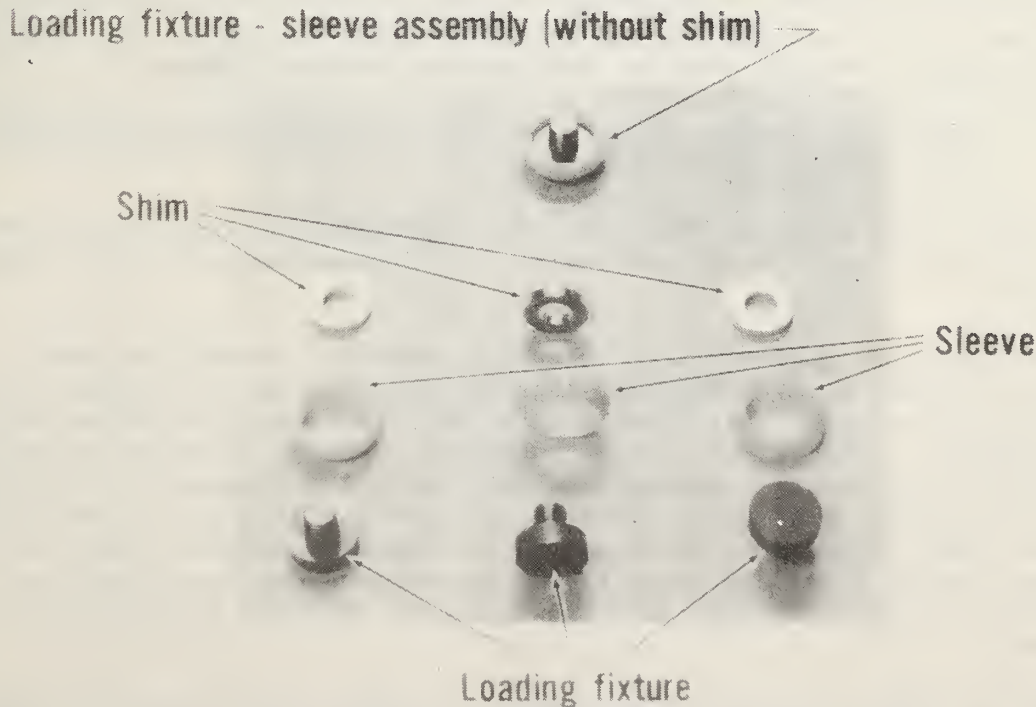


Figure A-2. Loading fixtures, shims, and cleaning sleeves



### Step I-3. Preparing the Epoxy Adhesive

The materials for this procedure are shown in figure A-1 and are found in the left front section of the PFAT case.

- (1) Set out an aluminum mixing dish, a mixing and application stick, and the two tubes of adhesive components.
- (2) From the tube containing the green colored material, squeeze out a straight bead across the center and touching both sides of the dish.
- (3) From the tube containing the cream colored material, squeeze out a straight bead parallel to the green bead but with ends barely touching the sides of the dish (slightly shorter than the green bead). The lengths of the two beads should approximate the 5:4 (green/cream) ratio required to obtain the necessary strength after hardening.
- (4) Using the wooden stick, mix the two parts of the material until the original green color becomes lighter and there are no signs of streaking. Allow to set for 5 minutes before application.
- (5) Using the wooden stick, apply the adhesive to the face of the loading fixture, carefully smoothing in a manner similar to frosting a cake. When the four feet on the face of the loading fixture are not visible, stop applying adhesive.

### Step I-4. Bonding the Loading Fixture

Depending upon the orientation of the coated surface, use one of the procedures described in Steps I-4a and I-4b for bonding the loading fixture.

#### Step I-4a. Bonding the Loading Fixture to Coated Surfaces Having Horizontal Orientations (Except those Overhead)

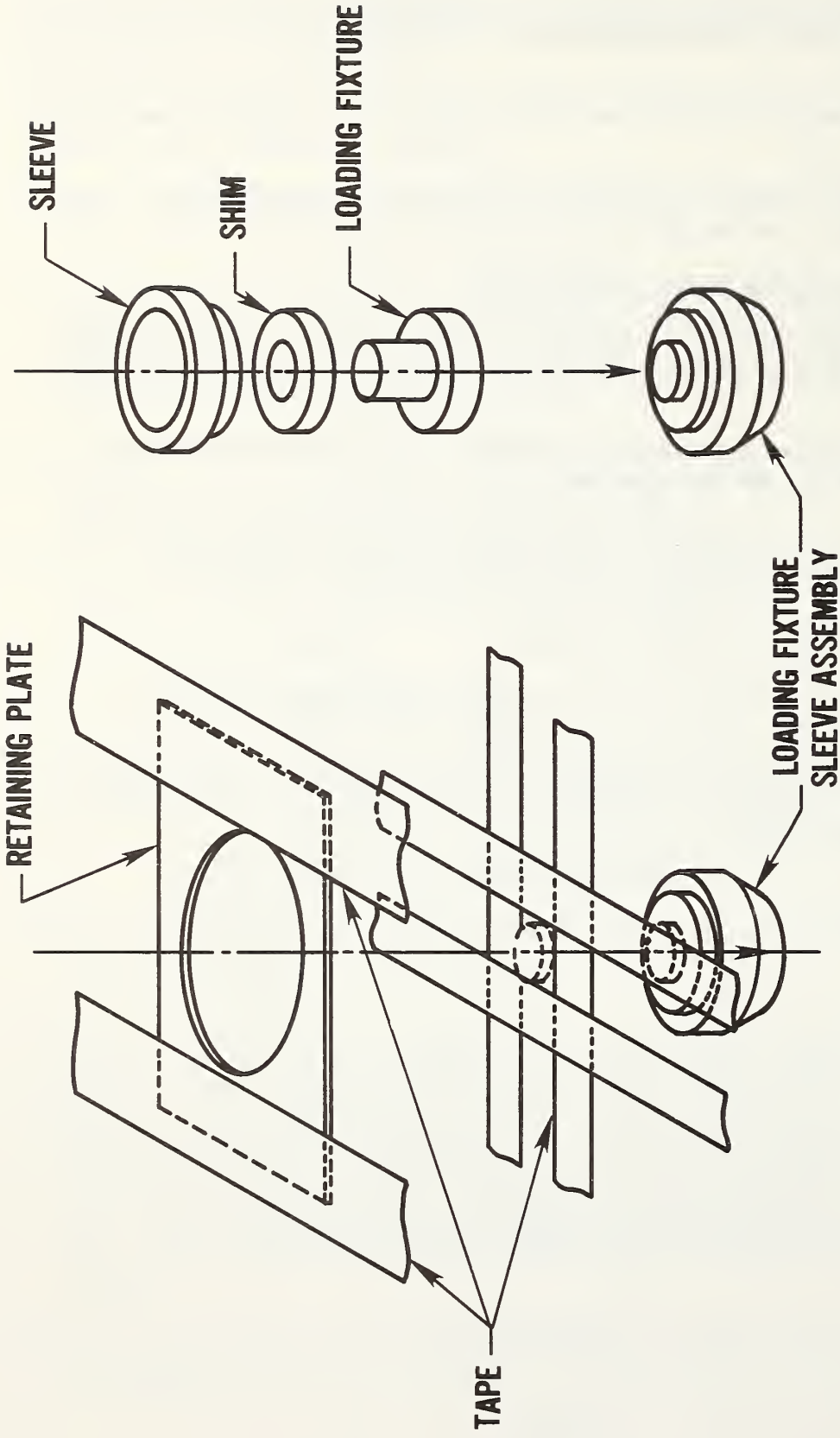
- (1) Grasp the sleeve with the thumb and middle finger while placing the end of the index finger against the end of the threaded section of the loading fixture.
- (2) Gently place the loading fixture with its adhesive covered face down against the test area. Gradually increase pressure on the loading fixture with the index finger until the feet on the fixture face touch the test area surface. (The fixture will stop making a slight slipping and sliding motion but will start "gripping" the test surface.) Placement of the fixture should take about 5 seconds. With the fixture in place on the test surface and the index finger still in place on the threaded section, grasp the sleeve with the thumb and index finger of the free hand and slide the sleeve down tightly against the test surface. Hold for an additional 2 seconds.
- (3) Remove both hands and allow the adhesive to harden for 24 hours at room temperature.

Step I-4b. Bonding the Loading Fixture to Coated Having Vertical Orientations or Surfaces Located Overhead (i.e. Ceilings)

- (1) Follow the same procedure given in steps I-4a(1) and I-4a(2) except that a shim (see figures A-2 and A-3) must be placed over the threaded section of the loading fixture.
- (2) While holding the loading fixture sleeve assembly completely still, tape it tightly against the test surface as shown in figure A-3.

Step I-5. Cleaning Loading Fixtures After Use

- (1) Place the fixture in distilled water and bring to a boil. Remove fixture from water and, with a small spatula, quickly separate adhesive from the fixture face.
- (2) Lightly sandblast the loading fixture face. This may be accomplished using a spark plug cleaning machine.
- (3) Visually examine the loading fixture face to insure that all old material has been removed and the face of the fixture has been completely sandblasted.
- (4) Clean loading fixture face with acetone.
- (5) Clean any hardened adhesive that may remain on the sleeve by lightly scraping with a knife blade and then wiping with a clean, soft paper towel.
- (6) Wrap loading fixture sleeve assembly in a clean, dry, soft paper towel and place in a container. If cleaning is in preparation for conducting tests, go to step I-2(2).



**Note: Shim is needed only when clamping is required**

Figure A-3. Clamping the loading fixture



## II. TESTING MACHINE CONTROLS

NOTE: Two people may be needed to conduct adhesion tests where the controls are more than an arms length from the test area.

### Step II-1. Placing the Tester Carrying Case

- (1) Orient the case so that the name tag is horizontal and can be plainly seen.
- (2) Orient the case in the open position so that the recording dial is in a horizontal plane. See figure A-4 for a picture of the recording dial, controls and identification of other components.

### Step II-2. Checking the Timing of the Pacer Wheel\*

- (1) As a reference, place a coin on the pacer wheel near the colored dot found closest to 0.62 MPa (90 psi) mark.
- (2) Push the toggle switch forward to start the pacer wheel.
- (3) Using a stop watch, start timing the dot as it crosses the zero mark and stop the timing as the dot crosses the 0.69 MPa (100 psi) mark. The watch should read  $74 \pm 1$  second.

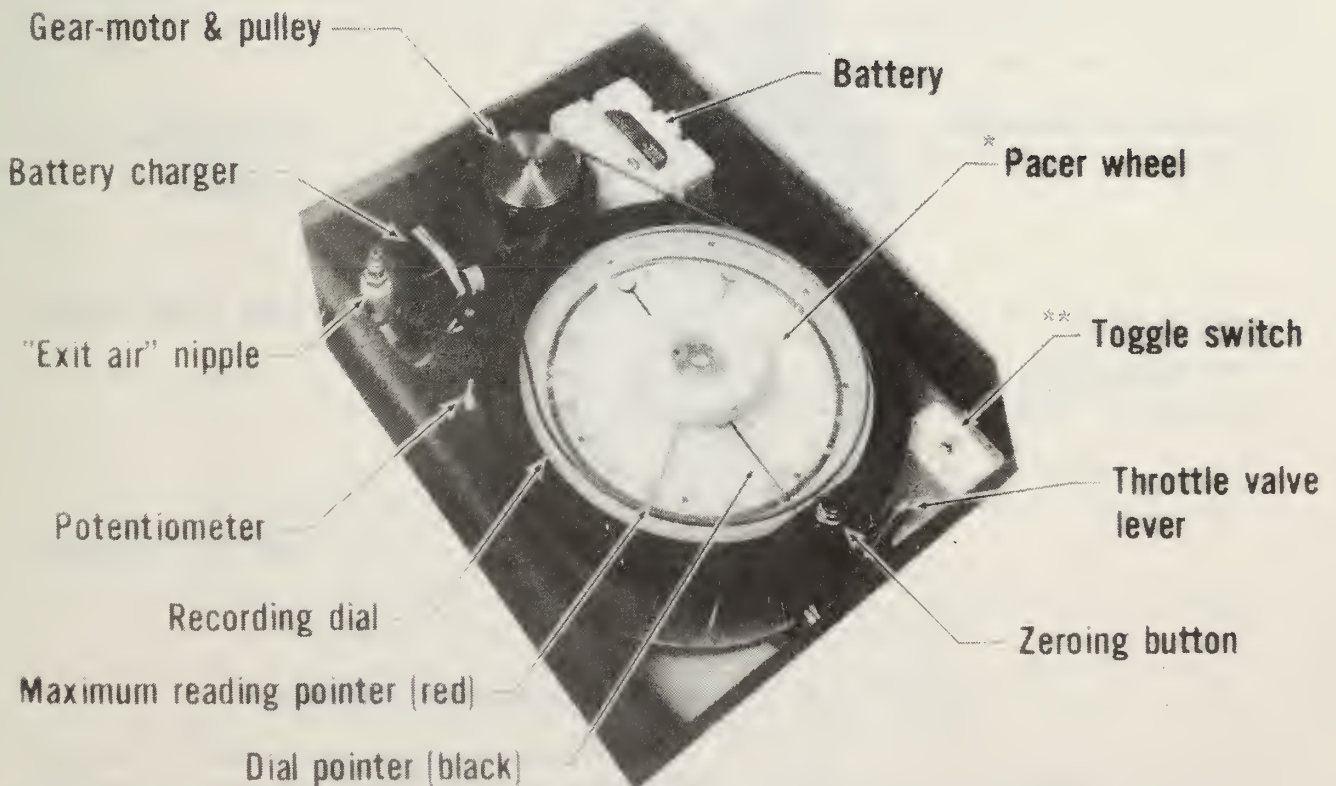


Figure A-4. Controls-recording dial and pacer assembly

\* The pacer wheel is the mechanism which allows the operator to control the air pressure and increase it at a constant rate.

\*\* Pacer on- push toggle switch to forward position. Charger on- push toggle switch to rearward position (plug in). Pacer and charger off- push toggle switch to center position. When charging, battery charger must be plugged into 110 v.a.c. single phase 60 cycle outlet. Do not charge below 40°C.

- (4) Adjustments can be made by rotating the potentiometer clockwise to increase and counterclockwise to reduce the time. If timing cannot be adjusted, charge the battery.

### Step II-3. Preparing The Recording Dial for Use

- (1) Using the larger knurled zeroing button located at the bottom of the recording dial, rotate the dial face until the zero mark lines up with the black dial pointer.
- (2) Using the knob located at the center of the recording dial face, rotate the red pointer counterclockwise until it rests against the black pointer.

### Step II-4. Activating the Air Pressure

Figure A-5 shows the compressed air source and it can be found in the center section of the carrying case. At the conclusion of this step (activating the

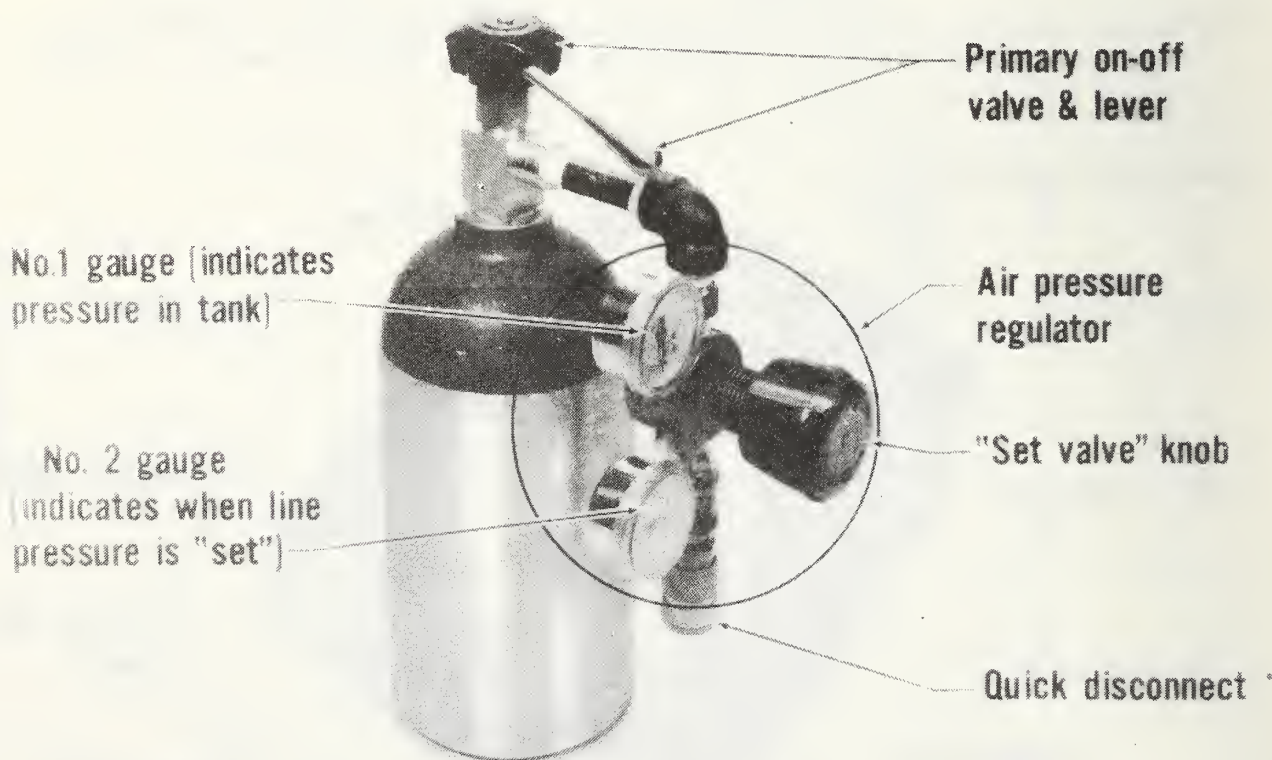


Figure A-5. Internal compressed air supply

\* External compressed air supply: Any source of compressed air may be used providing it is dry, not pulsating, and does not exceed 0.69 MPa (100 psi). To use external compressed air, attach one end of a hose to the "quick disconnect" with the green colored band. Detach the air line from the small tank by grasping tightly the knurled section of the "quick disconnect" (blue band) with one hand and the nipple line side with the other hand. Pull as in the manner of pulling on a rope. The nipple with the blue band may now be connected to the "quick disconnect" with the green band. Make sure the primary on-off valve is closed on the small tank.



air pressure) PFAT should be operating so that compressed air is getting to the throttle valve, no air is leaking, and the maximum reading pointer (red) operates properly.

- (1) Connect one end of the yellow, coiled, umbilical hose (figure A-6) to the yellow "exit air" nipple (figure A-4). Leave the free end of the hose unconnected at this point.
- (2) Push primary on-off lever to the right. Set line pressure (No. 2 gauge) by turning set valve knob clockwise.
- (3) Place the thumb on the top part of the zeroing button (tighting knob) to steady the hand and with the index finger move the throttle valve lever to the left. This activates and controls the air pressure change. As the throttle valve opens, the black dial pointer will rotate carrying the red pointer with it. The throttle valve closes automatically when the lever is released. The amount of trapped compressed air, given in psi, will be indicated by the final position of the black dial pointer. If air is leaking from the system, the speed at which the pointer drops toward zero will indicate the size of the leak. If a substantial leak exists, it should be corrected before continuing. If it is observed that compressed air is not getting to the throttle valve, the air line must be checked.
- (3) If the system proves to be air tight, evacuate the trapped air by pressing the eraser end of a pencil into the quick-disconnect at the free end of the umbilical hose.

### III. PRE-TESTING

#### Step III-1. Clearing the Loading Fixture

- (1) Remove all clamping devices (tapes, etc.) if the loading fixture is attached to an overhead or a vertical surface.
- (2) Very gently grasp the sleeve with a pair of pliers, gently loosen, and pull the sleeve away from the fixture (it may be possible to remove the sleeve with the bare fingers).
- (3) Insure that a clear and distinct separation exists between the periphery of the loading fixture and the hardened excess adhesive.

#### Step III-2. Installing the Piston Assembly

Figure A-6 shows the piston assembly.

- (1) Apply a thin layer of high vacuum grease on the piston membrane in a band about 25 mm (one inch) wide adjacent to and around the center hole.
- (2) Place the piston over the loading fixture with the membrane side down against the test surface. While holding the piston with one hand, carefully screw the pulling cap onto the loading fixture. Caution must

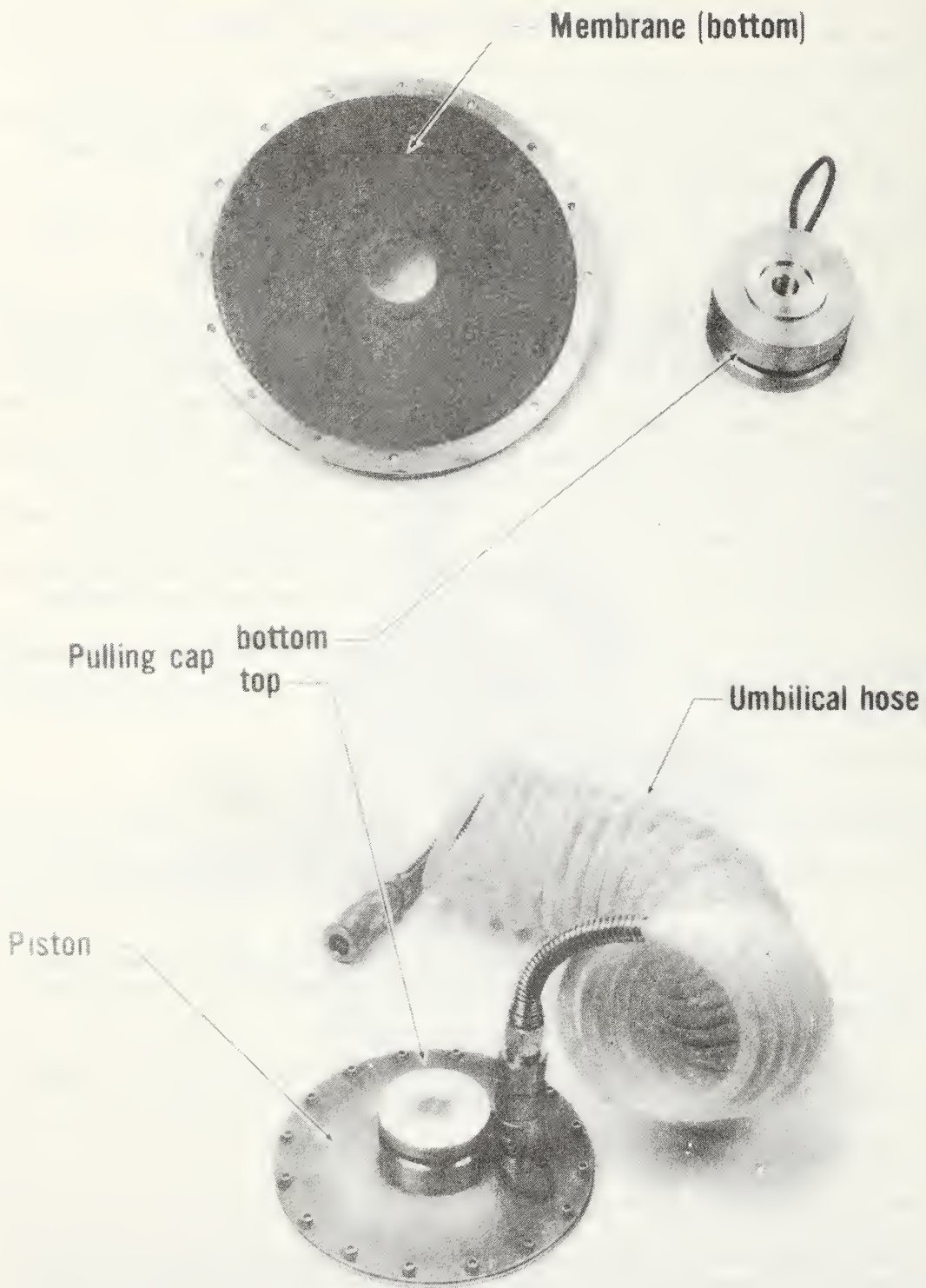


Figure A-6. Pneumatic piston assembly

be exercised to insure that the centering ring on the pulling cap properly engages the hole through the piston. Do not tighten the pulling cap because tightening will pre-load the loading fixture.

#### IV. TESTING

##### Step IV-1. Testing Check Points

- (1) Check line pressure. It should not exceed 0.69 MPa (100 psi).
- (2) Reset the red pointer against the black one.
- (3) Recheck zero alignment.
- (4) Make certain that the rubber loop on the pulling cap is over the inlet air nipple, and then attach the free end of the umbilical hose to the piston assembly.

##### Step IV-2. Pressurizing the Piston Assembly

- (1) With one hand, grasp the umbilical hose as close to the piston assembly as possible.
- (2) Turn pacer on.
- (3) With the free hand, operate the throttle valve.
- (4) As the throttle valve opens, the black dial pointer will rotate carrying the red pointer with it. Move the throttle so that the pointers travel at the same rate as one of the moving dots on the pacer wheel.
- (5) When failure occurs, continue holding the umbilical hose (especially on overhead and vertical surfaces) while releasing the throttle valve lever. Since at failure, the entire piston assembly breaks away along with the loading fixture, a net or other safety device may be substituted for holding the umbilical hose. The maximum air pressure reading, given in psi, is indicated by the red pointer.

##### Step IV-3. Recording the Data

- (1) Record the air pressure at rupture. To convert from air pressure to coating adhesion strength, see the formula given in the calibration data sheet which is provided with the instrument.
- (2) In addition to the air pressure at rupture, the records should contain all appropriate description information, i.e. date and location of test, name of operator, type and condition of coating tested, any deviations from one procedure described above, and mode of coating failure.



U.S. DEPT. OF COMM. <b>BIBLIOGRAPHIC DATA SHEET</b> <i>(See instructions)</i>	<b>1. PUBLICATION OR REPORT NO.</b> NBSIR 82-2535	<b>2. Performing Organ. Report No.</b>	<b>3. Publication Date</b>
<b>4. TITLE AND SUBTITLE</b>  Development of a Test Apparatus and Method for Measuring Adhesion of Protective Coatings			
<b>5. AUTHOR(S)</b> James F. Seiler, Mary E. McKnight, Larry W. Masters			
<b>6. PERFORMING ORGANIZATION</b> <i>(If joint or other than NBS, see instructions)</i>  NATIONAL BUREAU OF STANDARDS DEPARTMENT OF COMMERCE WASHINGTON, D.C. 20234		<b>7. Contract/Grant No.</b>	<b>8. Type of Report &amp; Period Covered</b>
<b>9. SPONSORING ORGANIZATION NAME AND COMPLETE ADDRESS</b> <i>(Street, City, State, ZIP)</i>  Tri-Service Building Materials Committee			
<b>10. SUPPLEMENTARY NOTES</b>  <input type="checkbox"/> Document describes a computer program; SF-185, FIPS Software Summary, is attached.			
<b>11. ABSTRACT</b> <i>(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)</i> <p>A pneumatic test apparatus and associated test method for measuring the adhesion of coatings have been developed with particular emphasis on: 1) overcoming some of the shortcomings of existing tests; and 2) providing a method which can provide quantitative information for both laboratory and field applications.</p> <p>The test apparatus utilizes compressed air to lift a stainless steel loading fixture (button) which is bonded with an adhesive to the surface of the protective coating. The rate at which the loading fixture is loaded is controlled by a precision air pressure gauge and the tensile force required to lift the button from one coating is measured. Assuming the level of adhesion of the adhesive to the coating is greater than that of the coating to the substrate, the tensile force provides a measure of the coating adhesion. Laboratory studies with two coating materials have been performed to assess the method.</p> <p>This report describes the test apparatus and associated test method and presents test data obtained to date, proposed modifications to the initial test apparatus design, and additional research needs. An Instruction Manual for use of the test apparatus is included in the Appendix.</p>			
<b>12. KEY WORDS</b> <i>(Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)</i>  Adhesion; measurement; protective coatings; test apparatus; test method			
<b>13. AVAILABILITY</b> <input checked="" type="checkbox"/> Unlimited <input type="checkbox"/> For Official Distribution. Do Not Release to NTIS <input type="checkbox"/> Order From Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402. <input checked="" type="checkbox"/> Order From National Technical Information Service (NTIS), Springfield, VA. 22161		<b>14. NO. OF PRINTED PAGES</b>  36	<b>15. Price</b>  \$7.50



