Designation: D 2711 – 01a

An American National Standard

Standard Test Method for Demulsibility Characteristics of Lubricating Oils[1]

This standard is issued under the fixed designation D 2711; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the measurement of the ability of oil and water to separate from each other. It is intended for use in testing medium and high-viscosity lubricating oils.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)[3]

3. Summary of Test Method

3.1 *For Oils That Do Not Contain Extreme Pressure (EP) Additives*—A 405-mL sample of the oil and 45 mL of distilled water are stirred together for 5 min at 82°C (180°F) in a special graduated separatory funnel. After a 5-h settling period following the stirring, a percentage of the water in the oil and the volumes of water and emulsion separating from the oil are measured and recorded.

3.2 *For Oils That Contain Extreme Pressure (EP) Additives*—A 360-mL sample of the oil and 90 mL of distilled water are stirred together for 5 min at 82 °C (180 °F) in a special graduated separatory funnel. After a 5-h settling period following the stirring, percentage of water in the oil and the volumes of water and emulsion separating from the oil are measured and recorded.

4. Significance and Use

4.1 This test provides a guide for determining the demulsibility characteristic of lubricating oils that are prone to water contamination and may encounter the turbulence of pumping and circulation capable of producing water-in-oil emulsions.

5. Apparatus

5.1 *Stirrer*, [4] constructed from parts shown in Fig. 1 and Fig. 2.

5.2 *Special Graduated Separatory Funnel*,[4] as shown in Fig. 3.

5.3 *Heating Bath*, sufficiently large and deep to permit the immersion of at least two test separatory funnels in the bath liquid up to their 500-mL graduation mark. The bath shall be capable of maintaining a temperature of 82 ± 1°C (180 ± 2°F) and shall be so equipped to hold the separatory funnels securely in a position so that the vertical axis of the stirrer corresponds to the center line of the separatory funnel during the mixing of the oil and water.

5.4 *Centrifuge*, as described in Test Method D 1796.

5.5 *Centrifuge Tubes*, long-form, 195 to 203 mm (8 in.) as described in Fig. 1 of Test Method D 1796.

6. Materials

6.1 *Cleaning Solvent*—Any suitable solvent capable of cleaning and effectively removing any oil or fluid from the stirrer and graduated cylinder. 1,1,1-Trichloroethane has been found suitable for use in this test method. (**Warning—** *1,1,1–Trichloroethane*, Harmful if inhaled or swallowed. Eye irritant. High concentration can cause unconsciousness or death.)

NOTE 1—In cases in which the use of 1,1,1 trichloroethane is unacceptable, some laboratories are using heptane or mineral spirits as alternative solvents. The effect on the precision of this test method when using an alternate solvent has not been determined.

6.2 *Water*—Type II reagent grade water conforming to Specification D 1193.

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.L on Industrial Lubricants.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 05.01.

[4] Available from Research Appliance Co., Moose Lodge Rd., Dept. T, Cambridge, MD 21613.

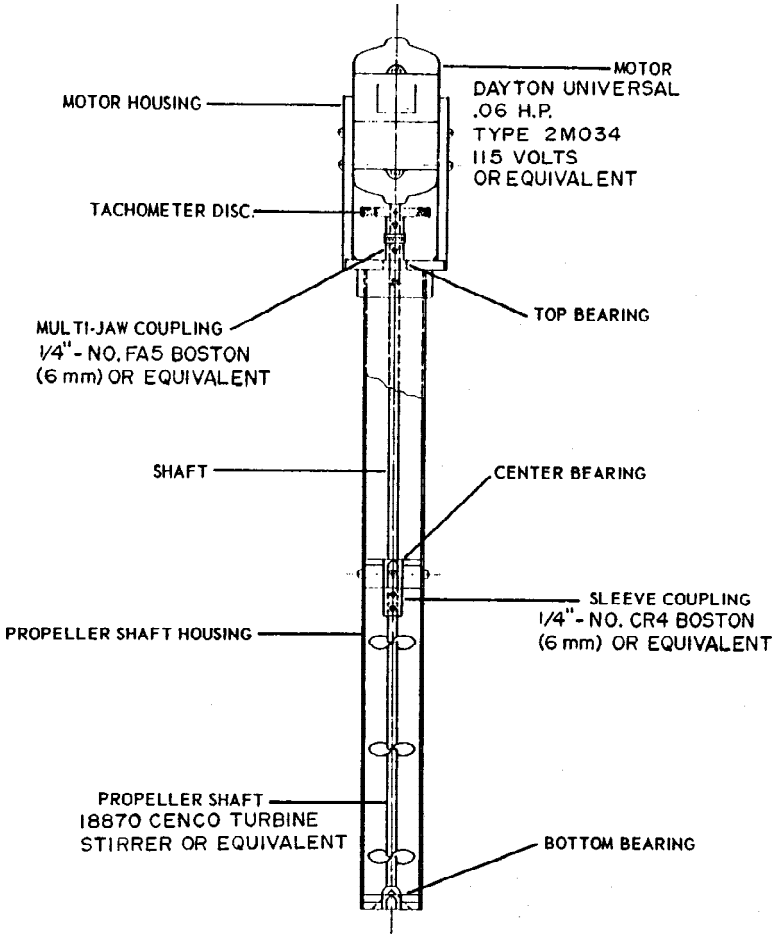


FIG. 1 Stirrer

7. Preparation of Apparatus

7.1 Clean the graduated separatory funnel by removing any film of oil with cleaning solvent followed by a wash first with acetone (**Warning—**Extremely flammable vapors may cause flash fires.) and then with tap water. Rinse thoroughly with tap water and then with reagent grade water.

7.2 Clean the stirrer by appropriate means, using the cleaning solvent (6.1). Allow the stirrer to air dry completely prior to use in the test.

8. Procedure A for Oils That Do Not Contain EP Additives

8.1 Heat the bath liquid to 82 ± 1°C (180 ± 2°F) and maintain this temperature throughout the test.

8.2 Measure the oil under test, at room temperature, directly into the separatory funnel to a volume of 405 ± 5 mL. Place the separatory funnel and oil in the constant-temperature bath and bring it to a temperature of 82°C (180°F). Add 45 ± 0.5 mL of distilled water, measured at room temperature, to the oil. Immerse the stirrer in the oil and position it carefully as follows: lower the stirrer until it touches the bottom of the funnel, then raise it approximately 25 mm (1 in.). Ensure that the vertical axis of the stirrer corresponds with the vertical center line of the funnel. Slowly bring the stirrer motor to a speed of 4500 ± 500 rpm within 25 to 30 s, and operate for a total of 5 min, including the start-up time. Then withdraw the stirrer from the oil-water mixture but not entirely out of the separatory funnel. Allow the stirrer to drain for 5 min, then remove from the separatory funnel and clean.

8.3 Five hours after stirring has stopped, withdraw a 50-mL sample from the center of the funnel and approximately 50 mm (2 in.) below the surface of the oil-water mixture, using a 50-mL pipet. Discharge the contents of the pipet into a centrifuge tube and determine the water present in the sample using Test Method D 1796. Record the results as "percent water in the oil."

8.4 With minimum delay, after sampling for the "percentage of water in the oil," remove the separatory funnel from the bath and draw off any free water that has separated from the oil-water mixture into a 50-mL graduated cylinder. Allow this water to reach room temperature, measure, and record the volume.

8.5 After removing the free water from the separatory funnel, reduce the volume of the fluid remaining to 100 mL by carefully siphoning the fluid off the top (end of siphon should not be more than 20 mm below the surface of the fluid at any time) down to the 100-mL graduation mark on the separatory funnel. Drain the remaining 100 mL of fluid (oil, water, and emulsion) directly into a centrifuge tube.

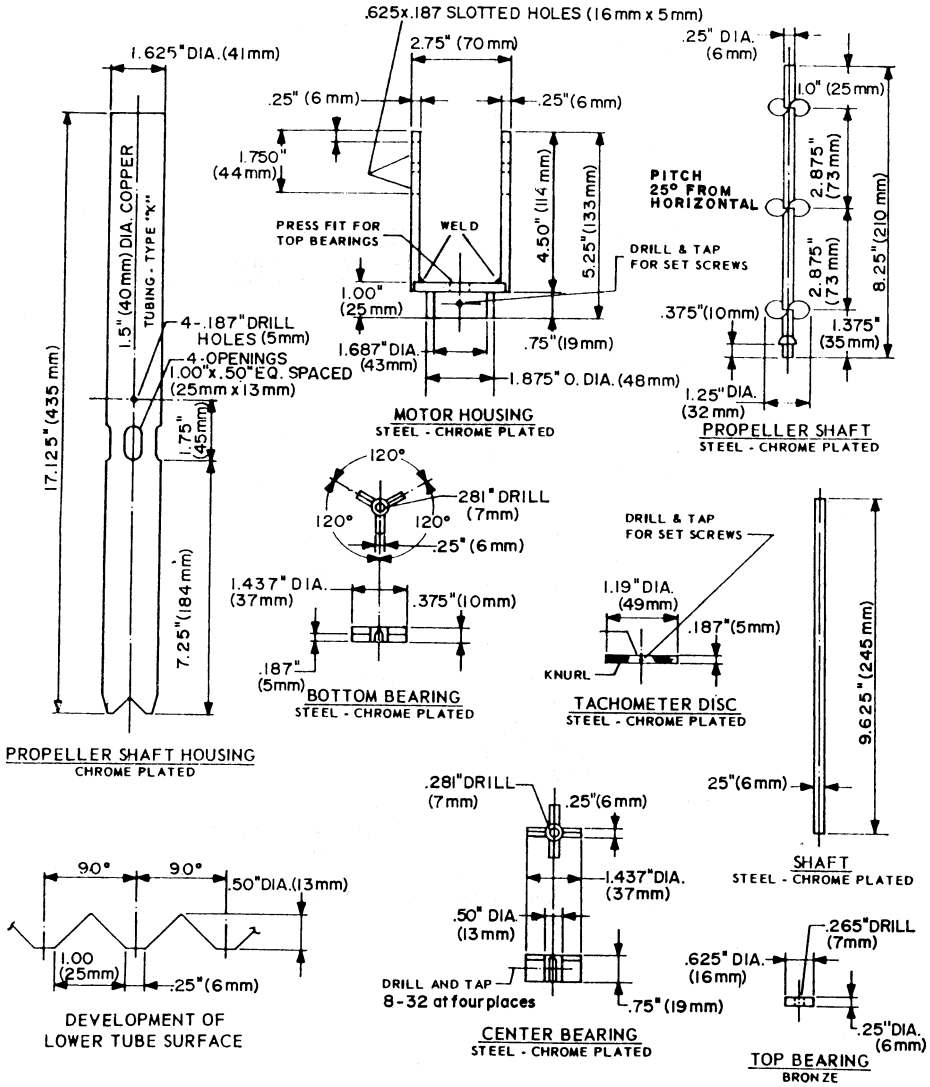


FIG. 2 Detailed Construction of Stirrer

8.6 Centrifuge the tube and its contents for a period of 10 to 15 min at a relative centrifugal force of 700. Record the volume of water and emulsion separated by centrifuging.

8.7 Perform a minimum of two determinations on each oil sample by repeating procedure 8.1-8.6. If the difference in the two sets of results obtained is outside the repeatability limits of Procedure A, discard them and obtain two additional sets of results.

9. Procedure B for Oils That Contain EP Additives

9.1 Heat the bath liquid to 82 ± 1 °C (180 ± 2 °F) and maintain this temperature throughout the test.

9.2 Measure the oil under test, at room temperature, directly into the separatory funnel to a volume of 360 ± 5 mL. Place the separatory funnel and oil in a constant-temperature bath and bring to a temperature of 82 °C (180 °F). Add 90 ± 0.5 mL of distilled water, measured at room temperature, to the oil. Immerse the stirrer in the oil and position it carefully as follows: lower the stirrer until it touches the bottom of the funnel, then raise it approximately 25 mm (1 in.). Ensure that the vertical axis of the stirrer corresponds with the vertical center line of the funnel. Slowly bring the stirrer motor to a speed of 2500 ± 250 rpm within 25 to 30 s, and operate for a total of 5 min, including the start-up time (Note 2). Then withdraw the stirrer from the oil-water mixture but not entirely out of the separatory funnel. Allow the stirrer to drain for 5 min, then remove it from the separatory funnel and clean.

9.3 Five hours after stirring has stopped, withdraw a 50-mL sample from the center of the funnel approximately 50 mm (2 in.) below the surface of the oil-water mixture, using a 50-mL pipete. Discharge the contents of the pipet into a centrifuge tube and determine the water present in the sample using Test Method D 1796. Record the results as “percent water in the oil”.

9.4 Immediately after sampling for the “percentage of water in the oil”, remove the separatory funnel from the bath and draw off any free water that has separated from the oil-water mixture into a 100-mL graduated cylinder. Allow this water to reach room temperature, measure, and record the volume.

9.5 After removing the free water from the separatory funnel, reduce the volume of the fluid remaining to 100 mL by carefully siphoning the fluid off the top (end of siphon should

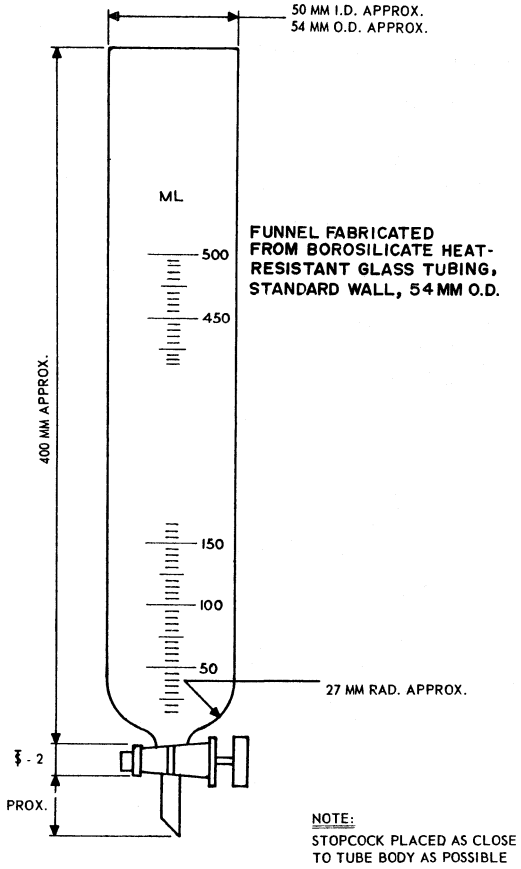


FIG. 3 Graduated Separatory Funnel

not be more than 20 mm below the surface of the fluid at any time) down to the 100-mL graduation mark on the separatory funnel. Drain the remaining 100 mL of fluid (oil, water and emulsion) directly into a centrifuge tube.

9.6 Centrifuge the tube and its contents for a period of 10 to 15 min at a relative centrifugal force of 700. Record the volume of water and emulsion separated by centrifuging.

9.7 Perform a minimum of two determinations on each oil sample by repeating procedure 9.1-9.3. If the difference in the two sets of results obtained is outside the repeatability limits of Procedure B, discard them and obtain two additional sets of results.

NOTE 2—The stirring motor may slow down when testing high-viscosity or emuslion-forming oils. Check speed frequently during the 5-min stirring period and adjust as necessary.

10. Report

10.1 Report the procedure used, that is, Procedure A or Procedure B.

10.2 Report the "percentage of water in the oil," (Note 3) "total millilitres of free water" (Note 4) and the "millilitres of emulsion" separated by centrifuging, for each determination and the average of each observation for all determinations. These are necessary factors in determining the demulsibility characteristics of a lubricating oil.

NOTE 3—Percent water in oil, record water present in amounts less than 0.1 % as <0.1 % or "trace."

NOTE 4—The "total millilitres of free water" is the sum of the millilitres of free water collected in the 50-mL graduated cylinder (8.4) and the millilitres of free water separated by centrifuging (8.6).

11. Precision for Procedure A

11.1 The precision for Procedure A does not use the matrix of laboratories and samples required by RR: D02 – 1007.[5] The following criteria should be used for judging the acceptability of results (95 % confidence). This precision statement is based on results obtained by seven laboratories on three oils and is applicable to oils with viscosity grades ranging from ISO 220 to ISO 460 (1000 SUS to 2000 SUS of 100°F).

11.1.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

Total free water, mL	4.0
Emulsion, mL	0.2

11.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, exceed the following values only in one case in twenty:

[5] Available from ASTM International Headquarters.

Total free water, mL	8.0
Emulsion, mL	0.3

12. Precision for Procedure B

12.1 The precision for Procedure B is based on results of cooperative tests from 13 laboratories on six laboratory-blended extreme-pressure lubricating oils. Test results, descriptions of the oils and analysis of data from this interlaboratory test program may be obtained from ASTM International Headquarters.[6] The test oils were classified in three groups with respect to level of demulsibility as measured by this test method - A, B and C - and precision data were developed for each classification. Classification groups and limits, and precision data are shown in Table 1.

12.1.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method exceed the values shown in Table 1 only in one case in twenty.

12.1.2 *Reproducibility*—The difference between single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, exceed the values shown in Table 1 only in one case in twenty.

13. Bias

13.1 The procedure in Test Method D 2711 has no bias because the values of total free water and emulsion can be defined only in terms of a test method.

14. Keywords

14.1 demulsibility; water contamination; water/oil separation

[6] Available from ASTM International Headquarters. Request RR: D02–1449.

TABLE 1 Precision for Procedure B on Extreme-Pressure Lubricating Oils

Demulsibility Classification	Classification Limits			Repeatability, *r*			Reproducibility, *R*		
	Water in Oil, %	Total Free Water, mL	Emulsion, mL	Water in Oil, %	Total Free Water, mL	Emulsion, mL	Water in Oil, %	Total Free Water, mL	Emulsion, mL
A	1.4 max	79 min	0.2 max	0.4	3.6	0.1[A]	0.8	5.1	0.2[A]
B	6.0 max	60 min	4.0 max	4.0	11	1.6	4.2	23	3.5
C	>6.0	<60	>4.0	5.6	18	~23	22	57	~96

[A] Estimated from inspection of data.

APPENDIX

(Nonmandatory Information)

X1. APPARATUS

X1.1 "*Gage and Centering Device*" (Fig. X1.1), is an aid in obtaining the 50-mL samples from the separatory funnel for the" percent water in the oil" determination (8.3).

X1.2 Fig. X1.2 illustrates a rapid method for reducing the volume of fluid remaining in the separatory funnel to 100 mL by siphoning the fluid off the top (end of siphon shall be not more than ¾ in. (20 mm) below the surface of the fluid at any time) down to the 100-mL graduation mark on the separatory funnel (8.5).

X1.2.1 The time required to drain the 100 mL of fluid from the separatory funnel into the centrifuge tube can be reduced, especially in the case of high-viscosity oils or the presence of a "mayonnaise"-type emulsion, by applying a slight amount of pressure (Fig. X1.3) to the open end of the separatory funnel to force the flow of the oil or emulsion, or both, into the centrifuge tube.

X1.3 Fig. X1.4(*a*) shows a suggested container for the solvent used to clean the stirrer after mixing the oil and water (8.2) and Fig. X1.4(*b*) shows a suggested method for air drying the stirrer after the above (solvent) washing.

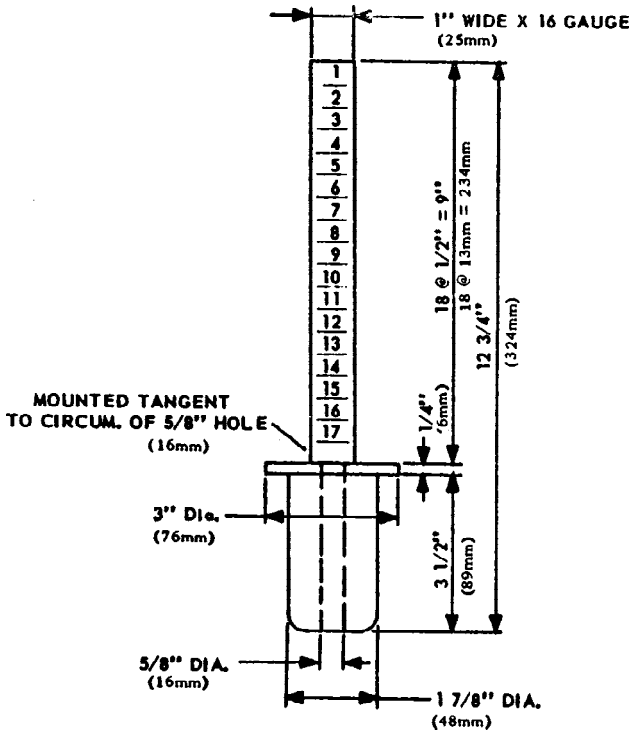


FIG. X1.1 Sampling Gage and Centering Device

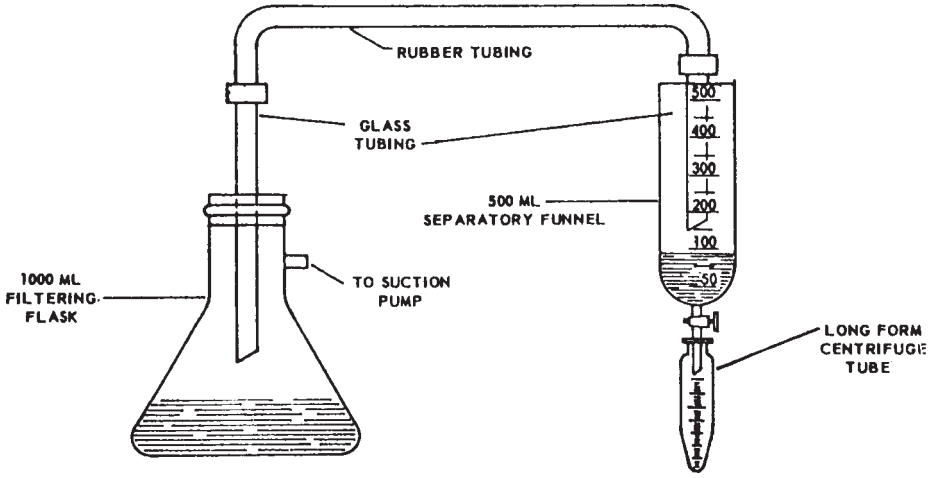


FIG. X1.2 Device to Reduce Volume in Separatory Funnel

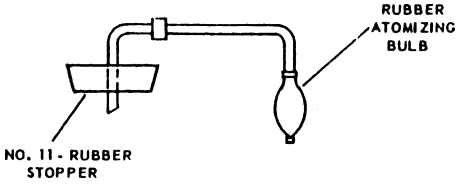
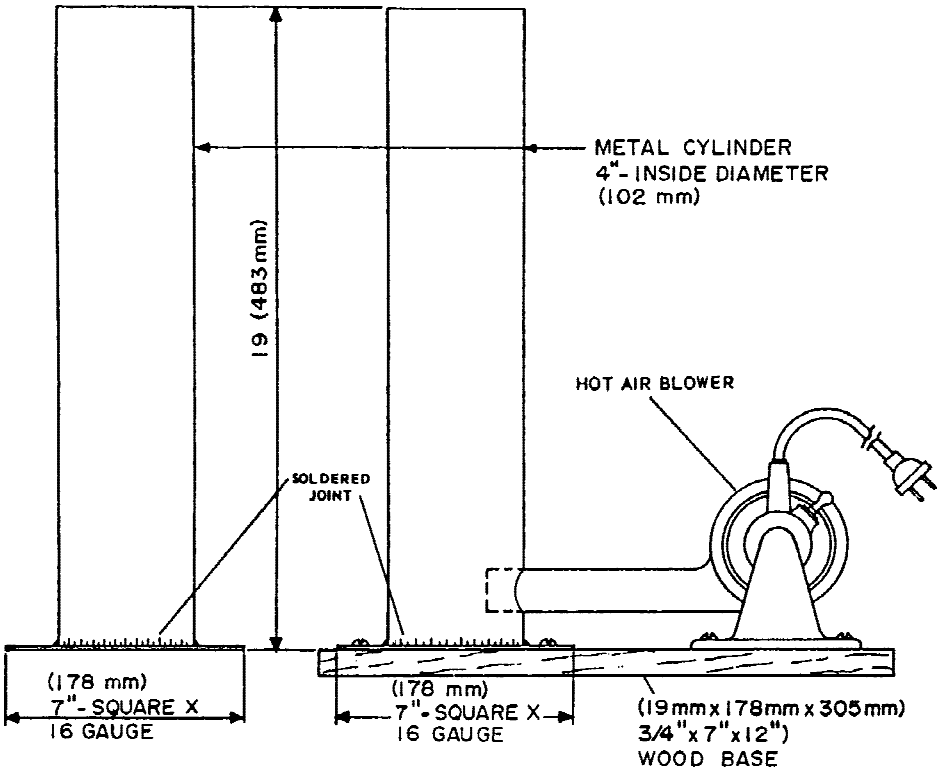


FIG. X1.3 Pressure Device



(*a*) Solvent Cleaning Tank (*b*) Forced Warm Air Dryer

FIG. X1.4 Optional Equipment