METHOD 9096

LIQUID RELEASE TEST (LRT) PROCEDURE

1.0 SCOPE AND APPLICATION

1.1 The Liquid Release Test (LRT) is a laboratory test designed to determine whether or not liquids will be released from sorbents when they are subjected to overburden pressures in a landfill.

1.2 Any liquid-loaded sorbent that fails the EPA Paint Filter Free Liquids Test (PFT) (SW-846 Method 9095), may be assumed to release liquids in this test. Analysts should ensure that the material in question will pass the PFT before performing the LRT.

2.0 SUMMARY OF METHOD

2.1 A representative sample of the liquid-loaded sorbent, standing 10 cm high in the device, is placed between twin stainless steel screens and two stainless-steel grids, in a device capable of simulating landfill overburden pressures. An absorptive filter paper is placed on the side of each stainless-steel grid opposite the sample (i.e., the stainless-steel screen separates the sample and the filter paper, while the stainless-steel grid provides a small air gap to prevent wicking of liquid from the sample onto the filter paper). A compressive force of 50 psi is applied to the top of the sample. Release of liquid is indicated when a visible wet spot is observed on either filter paper.

3.0 INTERFERENCES

3.1 When testing sorbents are loaded with volatile liquids (e.g., solvents), any released liquid migrating to the filter paper may rapidly evaporate. For this reason, filter papers should be examined immediately after the test has been conducted.

3.2 It is necessary to thoroughly clean and dry the stainless-steel screens prior to testing to prevent false positive or false negative results. Material caught in screen holes may impede liquid transmission through the screen causing false negative results. A stiff bristled brush, like those used to clean testing sieves, may be used to dislodge material from holes in the screens. The screens should be ultrasonically cleaned with a laboratory detergent, rinsed with deionized water, rinsed with acetone, and thoroughly dried.

When sorbents containing oily substances are tested, it may be necessary to use solvents (e.g., methanol or methylene chloride) to remove any oily residue from the screens and from the sample holder surfaces.

3.3 When placing the 76 mm screen on top of the loaded sample it is important to ensure that no sorbent is present on top of the screen to contact the filter paper and cause false positive results. In addition, some sorbent

residue may adhere to container sidewalls and contact the filter as the sample compresses under load, causing wet spots on the edges of the filter. This type of false positive may be avoided by carefully centering the 76 mm filter paper in the device prior to initiating the test.

3.4 Visual examination of the sample may indicate that a release is certain (e.g., free standing liquid or a sample that flows like a liquid), raising concern over unnecessary clean-up of the LRT device. An optional 5 minute Pre-Test, described in Appendix A of this procedure, may be used to determine whether or not an LRT must be performed.

4.0 APPARATUS AND MATERIALS

4.1 LRT Device (LRTD): A device capable of applying 50 psi of pressure continuously to the top of a confined, cylindrical sample (see Figure 1). The pressure is applied by a piston on the top of the sample. All device components contacting the sample (i.e., sample-holder, screens, and piston) should be resistant to attack by substances being tested. The LRTD consists of two basic components, described below.

4.1.1 Sample holder: A rigid-wall cylinder, with a bottom plate, capable of holding a 10 cm high by 76 mm diameter sample.

4.1.2 Pressure Application Device: In the LRTD (Figure 1), pressure is applied to the sample by a pressure rod pushing against a piston that lies directly over the sample. The rod may be pushed against the piston at a set pressure using pneumatic, mechanical, or hydraulic pressure. Pneumatic pressure application devices should be equipped with a pressure gauge accurate to within $\pm$ 1 psi, to indicate when the desired pressure has been attained and whether or not it is adequately maintained during the test. Other types of pressure application devices (e.g., mechanical or hydraulic) may be used if they can apply the specified pressure continuously over the ten minute testing time. The pressure application device must be calibrated by the manufacturer, using a load cell or similar device placed under the piston, to ensure that 50 $\pm$ 1 psi is applied to the top of the sample. The pressure application device should be sufficiently rugged to deliver consistent pressure to the sample with repeated use.

4.2 Stainless-Steel Screens: To separate the sample from the filter, thereby preventing false positive results from particles falling on the filter paper. The screens are made of stainless steel and have hole diameters of 0.012 inches with 2025 holes per square inch. Two diameters of screens are used: a larger (90 mm) screen beneath the sample and a smaller (76 mm) screen that is placed on top of the sample in the sample-holding cylinder.

4.3 Stainless-Steel Grids: To provide an air gap between the stainless-steel screen and filter paper, preventing false positive results from capillary action. The grids are made of 1/32" diameter, woven, stainless steel wire cut to two diameters, 90 mm and 76 mm.

4.4 Filter Papers: To detect released liquid. Two sizes, one 90 mm and one 76 mm, are placed on the side of the screen opposite the sample. The 76 mm diameter filter paper has the outer 6 mm cut away except 3 conical points used for centering the paper (see Figure 2). Blue, seed-germination filter paper manufactured by Schleicher and Schuell (Catalog Number 33900) is suitable. Other colored, absorptive papers may be used as long as they provide sufficient wet/dry contrast for the operator to clearly see a wet spot.

4.5 Spatula: To assist in loading and removing the sample.

4.6 Rubber or wooden mallet: To tap the sides of the device to settle and level the sample.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Reagent water. All references to water in this method refer to reagent water, as defined in Chapter One.

5.3 Acetone.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 All samples should be collected using a sampling plan that addresses the considerations discussed in "Test Methods for Evaluating Solid Wastes (SW-846)." The sampling plan should be designed to detect and sample any pockets of liquids that may be present in a container (i.e., in the bottom or top of the container).

6.2 Preservatives should not be added to samples.

6.3 Samples should be tested as soon as possible after collection, but in no case after more than three days after collection. If samples must be stored, they can be stored in sealed containers and maintained under dark, cool conditions (temperature ranging between 35° and 72° F). Samples should not be frozen.

7.0 PROCEDURE

The procedure below was developed for the original LRTD, manufactured by Associated Design and Manufacturing Company (ADM). Procedures for other LRTDs, along with evidence for equivalency to the ADM device, should be supplied by the manufacturer.

7.1 Disassemble the LRTD and make sure that all parts are clean and dry.

7.2 Invert the sample-holding cylinder and place the large stainless-steel screen, the large stainless-steel grid, then a 90 mm filter paper on the cylinder base (bottom-plate side).

7.3 Secure the bottom plate (plate with a hole in the center and four holes located on the outer circumference) to the flange on the bottom of the sample-holding cylinder using four knob screws.

7.4 Turn the sample holder assembly to the right-side-up position (bottom-plate-side down). Fill the sample holder with a representative sample until the sample height measures 10 cm (up to the etched line in the cylinder).

7.5 Tap the sides of the sample holder with a rubber or wooden mallet to remove air pockets and to settle and level the sample.

7.6 Repeat filling, and tapping until a sample height of 10 cm is maintained after tapping.

7.7 Smooth the top of the sample with a spatula to create a horizontal surface.

7.8 Place the small stainless-steel screen, then the small stainless-steel grid on top of the sample.

> NOTE: Prior to placing the stainless-steel grid on top of the screen, make sure that no sorbent material is on the grid side of the stainless-steel screen.

7.9 Place the 76 mm filter paper on top of the small stainless-steel grid, making sure the filter paper is centered in the device.

7.10 Using the piston handle (screwed into the top of the piston) lower the piston into the sample holder until it sits on top of the filter paper. Unscrew and remove the handle.

7.11 Place the loaded sample holder into position on the baseplate and lock into place with two toggle clamps.

7.12 Place the pressure application device on top of the sample-holder. Rotate the device to lock it into place and insert the safety key.

7.13 Connect air lines.

7.14 Initiate rod movement and pressure application by pulling the air-valve lever toward the operator and note time on data sheet. The pressure gauge at the top of the pressure application device should read as specified in the

factory calibration record for the particular device. If not, adjust regulator to attain the specified pressure.

NOTE: After pressure application, the air lines can be disconnected, the toggle clamps can be released, and the LRTD can be set aside for 10 minutes while other LRTDs are pressurized. LRTD pressures should be checked every 3 minutes to ensure that the specified pressure is being maintained. If the specified pressure is not being maintained to within ± 5 psi, the LRTD must be reconnected to the air lines and pressure applied throughout the 10 minute test.

7.15 After 10 minutes place the LRTD on the baseplate, reconnect air lines and toggle clamps, and turn off pressure (retract the rod) by pushing the air-valve lever away from the operator. Note time on data sheet.

7.16 When the air gauge reaches 0 psi, disconnect the air lines and remove the pressure-application device by removing the safety key, rotating the device, and lifting it away from the sample holder.

7.17 Screw the piston handle into the top of the piston.

7.18 Lift out the piston.

7.19 Remove the filter paper and immediately examine it for wet spots (wet area on the filter paper). The presence of a wet spot(s) indicates a positive test (i.e., liquid release). Note results on data sheet.

7.20 Release toggle clamps and remove sample holder from baseplate. Invert sample holder onto suitable surface and remove the knob screws holding the bottom plate.

7.21 Remove the bottom plate and immediately examine the filter paper for wet spots as described in Step 7.19. Note results on data sheet. Wet spot(s) on either filter indicates a positive test.

8.0 QUALITY CONTROL

8.1 Duplicate samples should be analyzed every twenty samples or every analytical batch, whichever is more frequent. Refer to Chapter One for additional QC protocols.

9.0 METHOD PERFORMANCE

9.1 Precision and accuracy data are not available at this time.

10.0 REFERENCES

1. Hoffman, P., G. Kingsbury, B. Lesnik, M. Meyers, "Background Document for the Liquid Release Test (LRT) Procedure"; document submitted to the Environmental Protection Agency by Research Triangle Institute: Research Triangle Park, NC

under Contract No. 68-01-7075, Work Assignment 76 and Contract No. 68-WO-0032, Work Assignment 12.

FIGURE 1.
LRT DEVICE

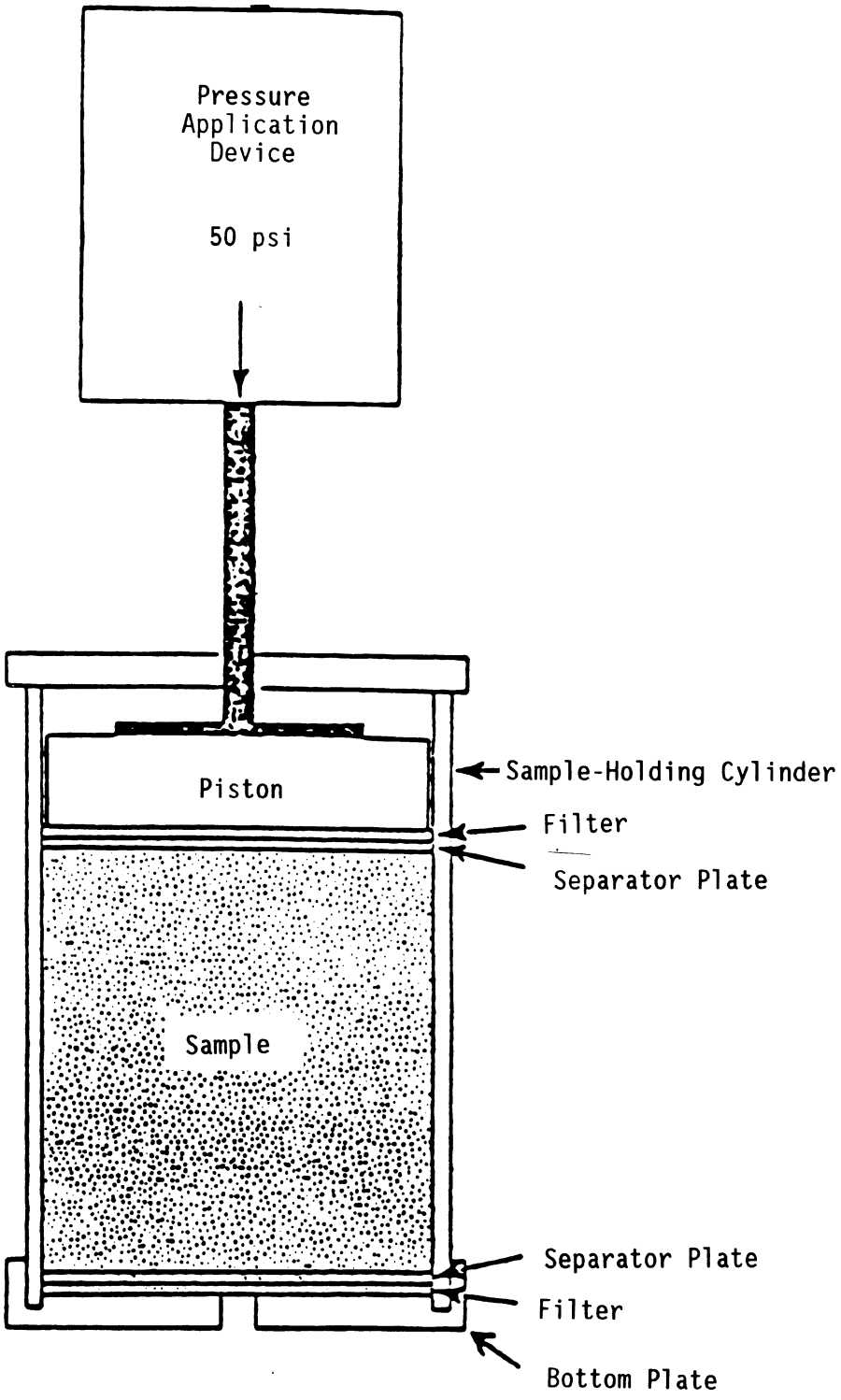


FIGURE 2.
76 MM DIAMETER FILTER PAPER

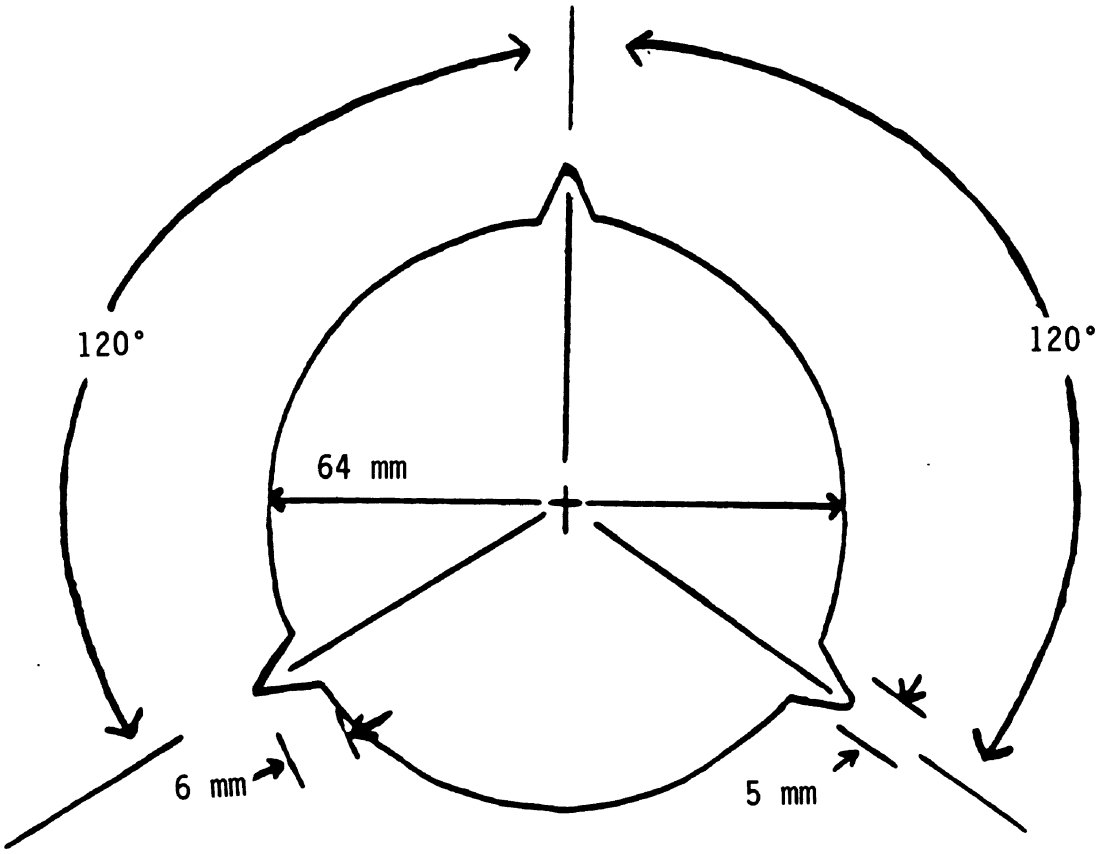


FIGURE 3.
GLASS GRID SPECIFICATIONS.

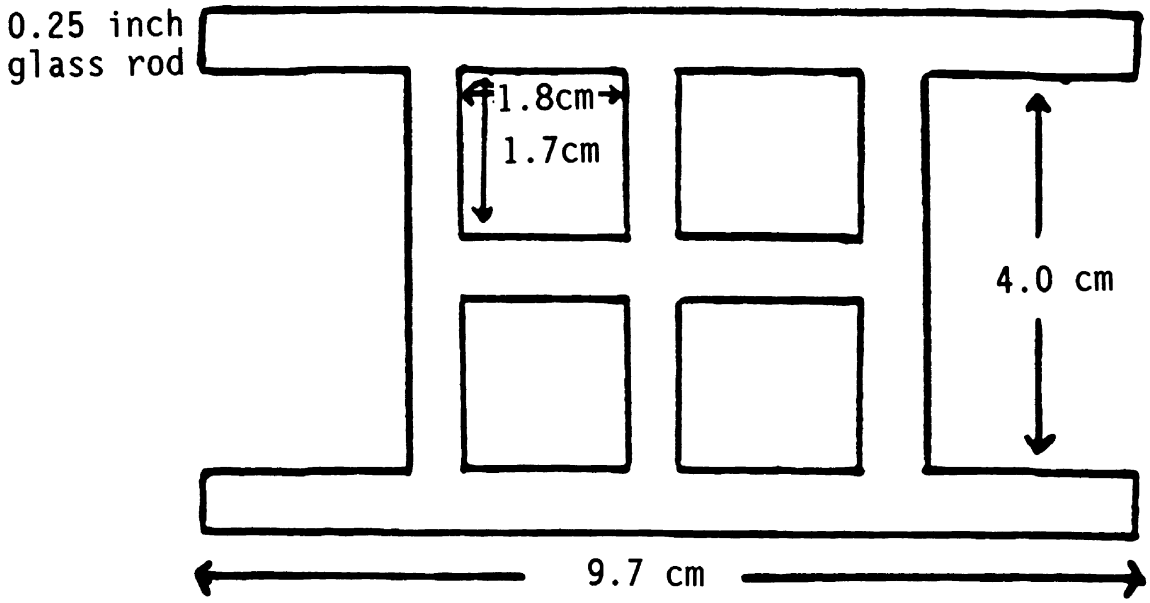
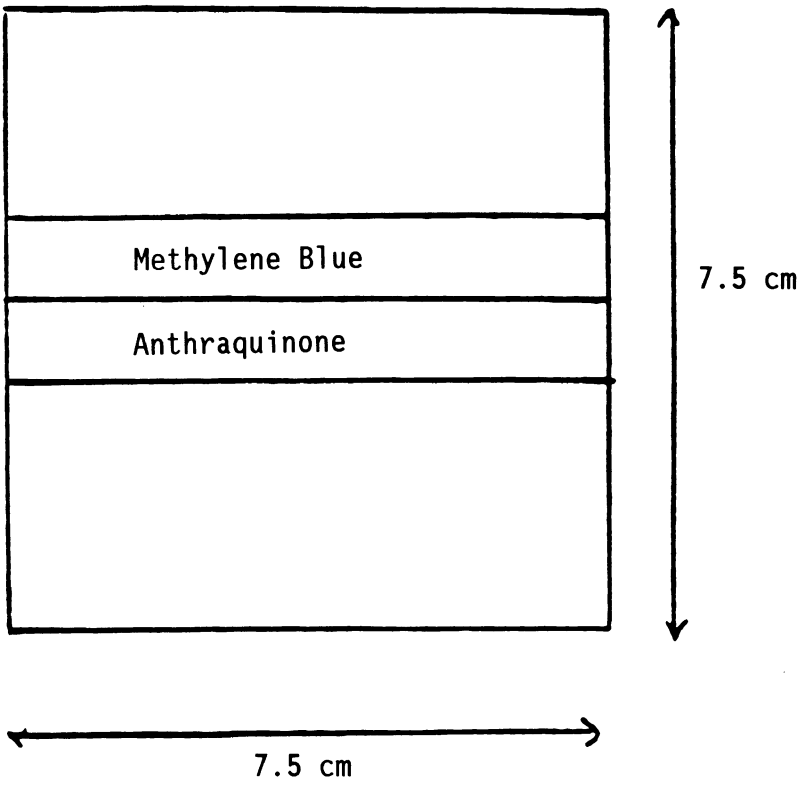


FIGURE 4.
POSITIONING OF DYE ON GLASS PLATE



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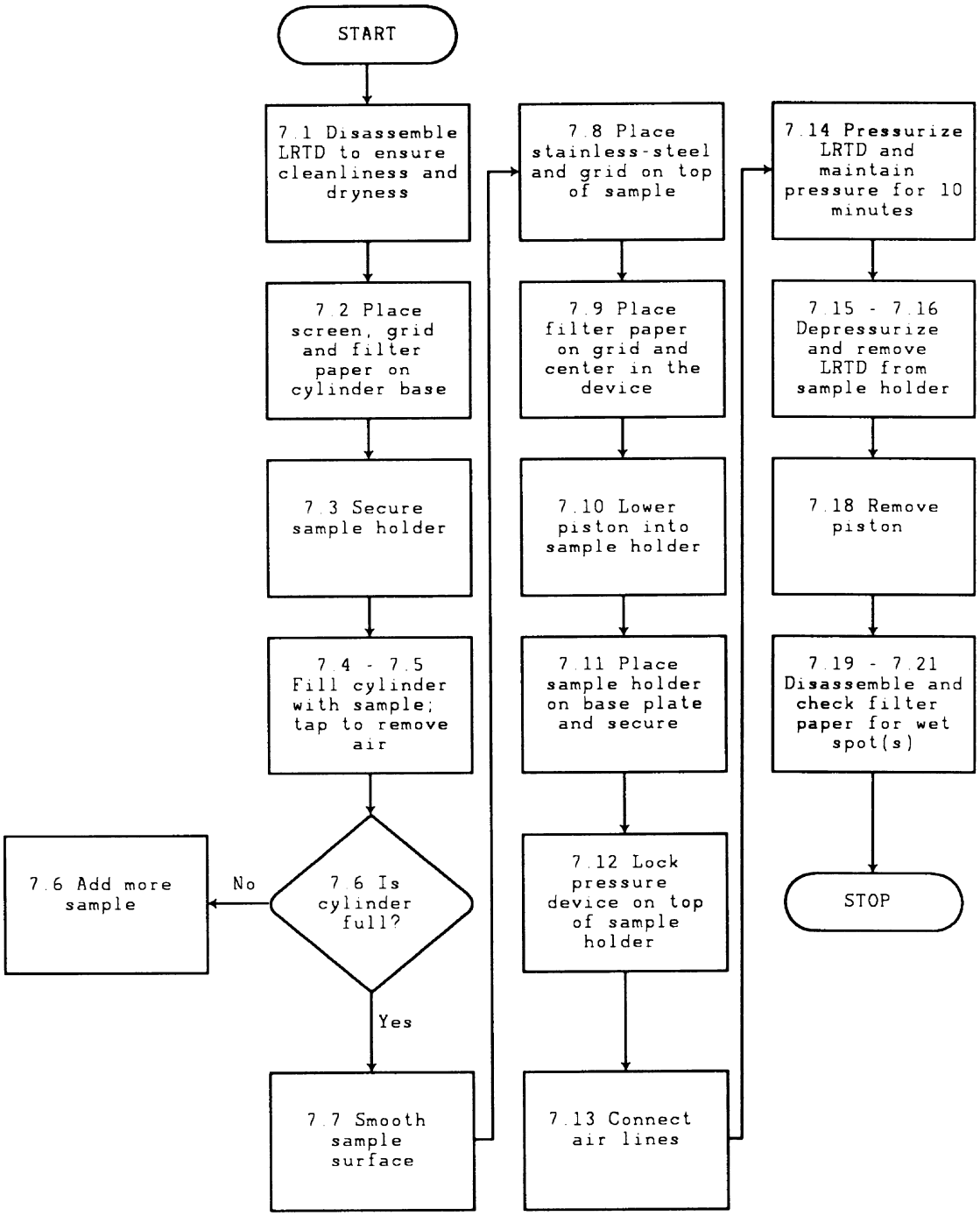


APPENDIX A

LIQUID RELEASE TEST PRE-TEST

1.0 SCOPE AND APPLICATION

1.1 The LRT Pre-Test is an optional, 5 minute laboratory test designed to determine whether or not liquids will be definitely released from sorbents before applying the LRT. This test is performed to prevent unnecessary cleanup and possible damage to the LRT device.

1.2 This test is purely optional and completely up to the discretion of the operator as to when it should be used.

2.0 SUMMARY OF METHOD

A representative sample will be loaded into a glass grid that is placed on a glass plate already stained with 2 dyes (one water soluble and one oil soluble). A second glass plate will be placed on top and a 2 lb. weight placed on top for 5 minutes. At the end of 5 minutes the base of the glass grid is examined for any dye running along the edges, this would indicate a liquid release.

3.0 INTERFERENCES

A liquid release can be detected at lower Liquid Loading Levels with extremely clean glassware. The glass plates and glass grid should be cleaned with a laboratory detergent, rinsed with Deionized water, rinsed with acetone, and thoroughly dried.

4.0 APPARATUS AND MATERIALS

4.1 Glass Plate: 2 glass plates measuring 7.5 cm x 7.5 cm.

4.2 Glass Grid: See Figure 3.

4.3 Paint Brush: Two small paint brushes for applying dyes.

4.4 Spatula: To assist in loading the sample.

4.5 Weight: 2.7 kg weight to apply pressure to the sample.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Methylene Blue dye in methanol.

5.3 Anthraquinone dye in toluene.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

See LRT Procedure.

7.0 PROCEDURE

7.1 Paint one strip, approximately 1 cm wide, of methylene blue dye across the center of a clean and dry glass plate (see Figure 4). The dye is allowed to dry.

7.2 Paint one strip, approximately 1 cm wide, of anthraquinone dye across the center of the same glass plate (see Figure 4). This strip should be adjacent to and parallel with the methylene blue strip. The dye is allowed to dry.

7.3 Place the glass grid in the center of the dye-painted glass plate.

7.4 Place a small amount of sample into the glass-grid holes, pressing down gently until the holes are filled to slightly above the grid top.

7.5 Place a second, clean and dry, glass plate on top of the sample and grid.

7.6 Place a 2.7 kg weight on top of the glass for 5 minutes.

7.7 After 5 minutes remove the weight and examine the base of the grid extending beyond the sample holes for any indication of dyed liquid. The entire assembly may be turned upside down for observation. Any indication of liquid constitutes a release and the LRT does not need to be performed.

8.0 QUALITY CONTROL

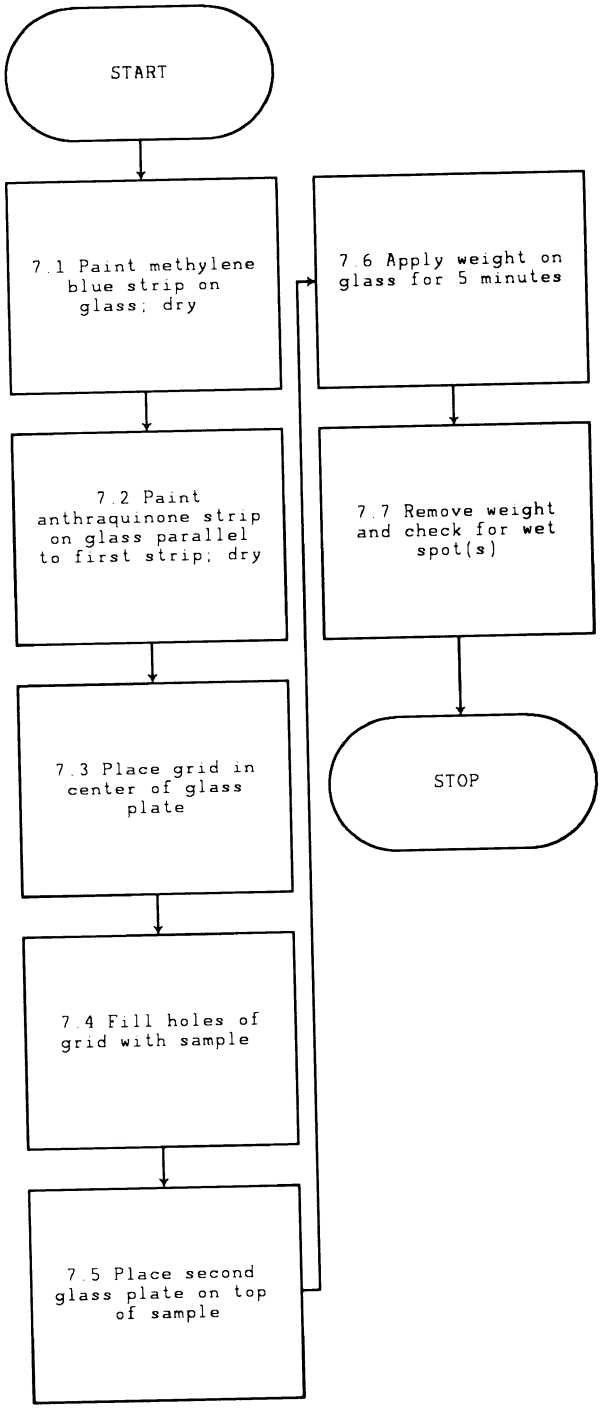
8.1 Refer to Chapter One for specific quality control procedures.

9.0 METHOD PERFORMANCE

9.1 Precision and accuracy data are not available at this time.

10.0 REFERENCES

1. Research Triangle Institute. "Background Document for the Liquid Release Test: Single Laboratory Evaluation and 1988 Collaborative Study". Submitted to the Environmental Protection Agency under Contract No. 68-01-7075, Work Assignment 76 and Contract No. 68-WO-0032, Work Assignment 12. September 18, 1991.


START
7.1 Paint methylene blue strip on glass; dry
7.2 Paint anthraquinone strip on glass parallel to first strip; dry
7.3 Place grid in center of glass plate
7.4 Fill holes of grid with sample
7.5 Place second glass plate on top of sample
7.6 Apply weight on glass for 5 minutes
7.7 Remove weight and check for wet spot(s)
STOP