Method 310 - Appendix A

PROPELLANT COLLECTION PROCEDURES

1 APPLICATION

The procedure applies to modify ASTM D 3074-**94** ~~**72**~~ and D 3063-**94** ~~**79**~~ to allow collection of the propellant for analysis and density measurement for metal aerosol containers and glass aerosol containers, respectively. These modified procedures also retain the aerosol standard terminology listed in ASTM D 3064-89. The aerosol product container is pierced and the propellant is bled into an evacuated manifold. After the manifold reaches atmospheric pressure, approximately 1 liter of the propellant is collected in a clean, evacuated Tedlar bag. For density measurement the propellant is collected into an evacuated 250 mL glass dilution bulb that has been weighed to the nearest 0.1 mg. After filling, the dilution bulb is re-weighed to determine the density of the propellant. Alternately, density may be determined using a Density/Specific Gravity Meter. The Tedlar bag with the propellant aliquot is taken to the laboratory for analysis.

2 LIMITATIONS

Nitrogen analysis: Nitrogen may be used as a component of the propellant system. Ambient air is 78 percent nitrogen and may be present as a contaminate in the system prior to sample collection. This is eliminated by completely evacuating the propellant collection system and sweeping out any connecting lines to the Tedlar bag with product before starting sample collection. This procedure will eliminate or reduce nitrogen contamination to less than 0.1% by weight of the sample and the analysis of the propellant gas will be unaffected.

3 APPARATUS AND MATERIALS

3.1 Propellant Collection System[1]: See Figure 1. The system was built from 1/4" stainless steel and Teflon tubing. The vacuum pump is of bellows diaphragm design.

3.2 Tedlar Bags, 1 liter, equipped with slip valve and septum

3.3 Density Measurement

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[3] [4] The metal piercing adapter is available from Mid-West Screw Products, Inc., 3523 North Kenton Ave., Chicago, IL 60641. Interim Part Number: 8013A-3/4 Longer SS. The gasket is available from Alltech Associate 2051 Waukegan road, Deerfield, IL 60015, part number 80-16. The glass aerosol adapter is available from Modern Machine Ship, Inc. P.O. Box 826, 123 N. Hazel Street, Danville, IL 61832.

3.3.1 250 mL gas dilution bulb**, or**

3.3.2 ***~~or, an~~*** Density/Specific gravity meter meeting the following minimum specifications:

3.3.2(a) Measurement Method: Natural Oscillation Type

3.3.2(b) Range: 0 - 3 g/cm^3

3.3.2(c) Measurement Temperature Range: 4 °C ~ 70 °C.

3.3.2(d) Temperature Accuracy: +/- 0.02 °C (10 °C ~ 30 °C) and +/-0.05 °C (4 °C ~70 °C).

3.3.2(e) Temperature Control Accuracy: +/- 0.01 °C.

3.3.2(f) Measurement Time: 1- 4 minutes.

3.4 Gas tight syringe, 100 μl

3.5 Balance, capable of accurately weighing to 0.1 mg

3.6 Can Piercing Platform. See Figure 2 (metal cans) and Figure 3 (glass containers).

3.7 Platform Shaker, equivalent to Thermolyne M49125

4 PROCEDURE

4.1 Propellant Collection for Metal Aerosol Containers

4.1.1 Turn on vacuum pump, close valves and evacuate the system (see Figure 1).

4.1.2 Remove the valve actuator on the aerosol can and weigh can to the nearest 0.01 g. Invert the can into cork holding ring on the piercing apparatus, center and snug against the gasket. (Figure 2)

4.1.3 Connect Tedlar bag to output 2, evacuate bag and seal. Connect 250 mL glass dilution bulb to output 1, evacuate bulb and seal.

4.1.4 Slowly raise the hydraulic jack until the can is pierced. Record the pressure of the can.

4.1.5 Vent the can until the pressure is at about 25 psi. Collect the propellant in the Tedlar bag.

4.1.6 After the propellant is collected, close and remove the Tedlar bag and vent the remainder of the propellant.

4.1.7 Weigh the evacuated 250 mL bulb to the nearest 0.1 mg. Use gloves while handling the bulb. Connect the bulb to the Tedlar bag and open to fill the bulb. Close the valves and re-weigh the dilution bulb, record the weight gain and calculate the propellant density in gm/l.

4.1.8 After the flow ceases from the can, it is removed from the assembly and allowed to vent overnight. The can may be placed on a platform shaker to vent the remainder of the propellant.

4.1.9 Reweigh can to the nearest 0.01 gm and record weight loss (total gms propellant). The can may now be opened for analysis of the liquid product.

4.2 Propellant Collection for Glass Aerosol Containers

4.2.1 Turn on vacuum pump, close valves and evacuate the system (see Figure 1).

4.2.2 Connect Tedlar bag to output 2, evacuate bag and seal. Connect 250 mL glass dilution bulb to output 1, evacuate bulb and seal.

4.2.3 The gauge assembly is prepressurized in order to minimize product expulsion and system contamination.

4.2.4 Remove actuator from valve of the aerosol glass container, and weigh container to the nearest 0.01 gm.

4.2.5 With container in an inverted position place the valve onto the tapered adaptor. Bring the top plate down to the flat of the container and tighten the nuts. A cork ring may be required to stabilize the container.

4.2.6 Record pressure of container and vent until the pressure is approximately one-half of recorded pressure. Collect propellant sample into the Tedlar bag.

4.2.7 After the propellant is collected, close and remove the Tedlar bag and vent the remainder of the propellant.

4.2.8 Weigh the evacuated 250 mL bulb to the nearest 0.1 mg. Use gloves while handling the bulb. Connect the bulb to the Tedlar bag and open to fill the bulb. Close the valves and re-weigh the dilution bulb, record the weight gain and calculate the propellant density in gm/l.

4.2.9 Continue to vent container on the platform assembly overnight.

4.2.10 Remove container from platform and loosen valve assembly, do not remove valve assembly at this time.

4.2.11 Place container on a platform shaker to vent the remainder of the propellant.

4.2.12 Reweigh container and valve assembly to the nearest 0.01 gm and record weight loss (total gms propellant). The container may now be opened for analysis of the liquid product.

FIGURE 1

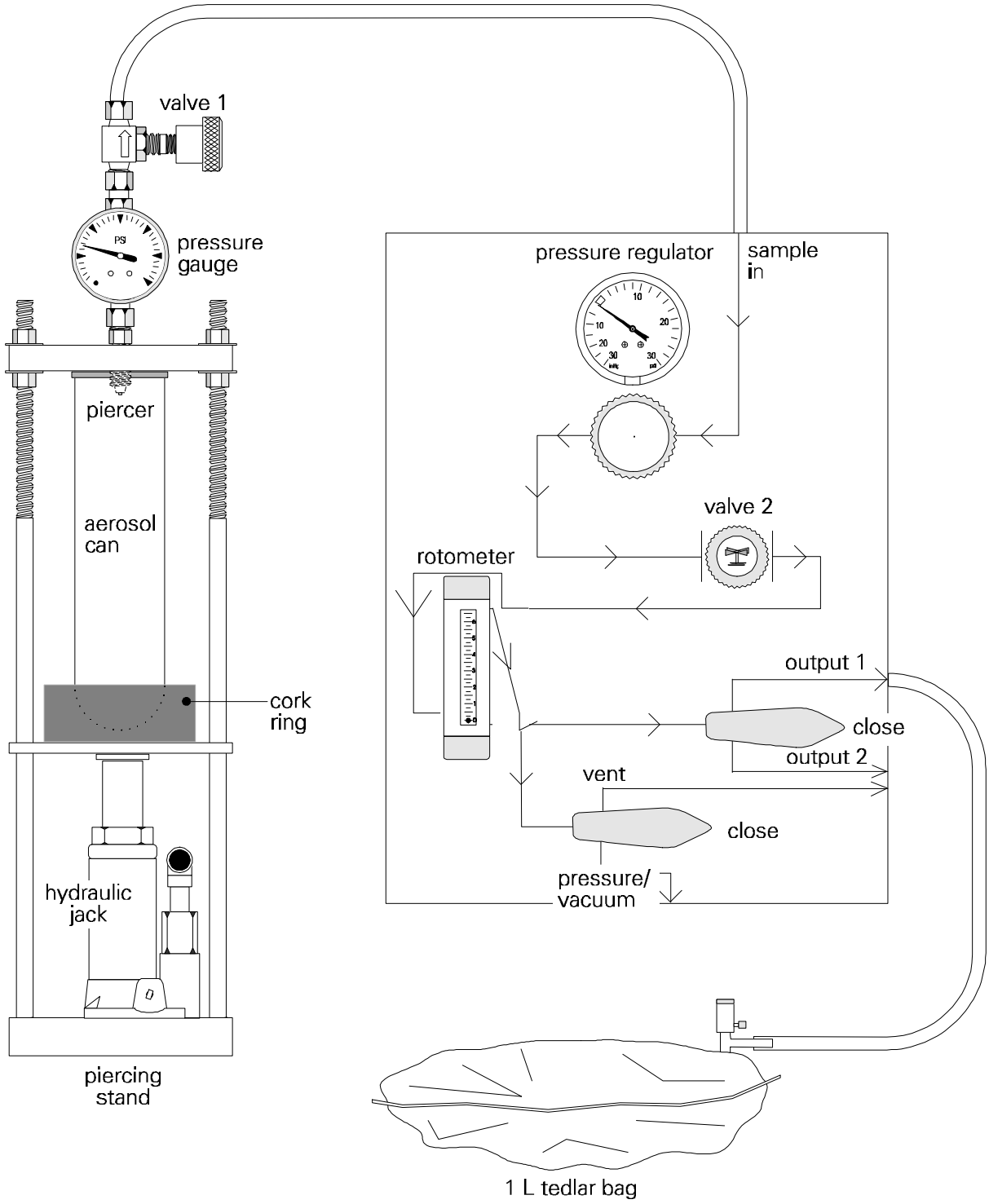
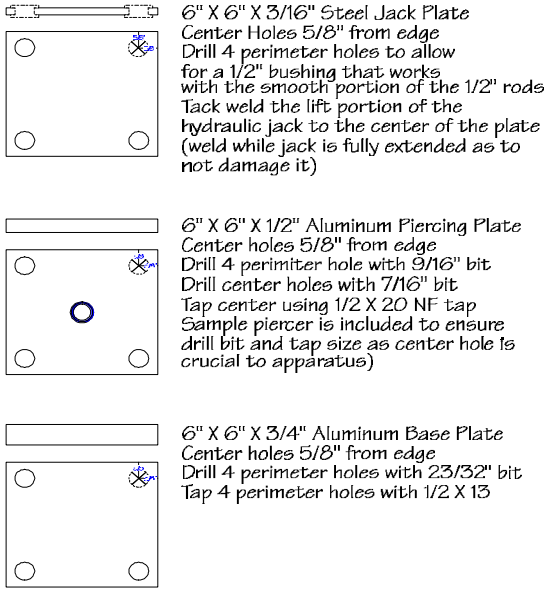
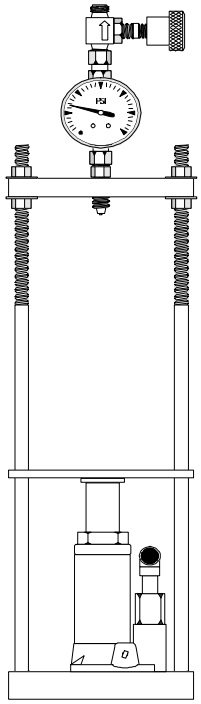

valve 1
pressure gauge
piercer
aerosol can
cork ring
hydraulic jack
piercing stand
pressure regulator
sample in
valve 2
rotometer
output 1
close
output 2
vent
close
pressure/ vacuum
1 L tedlar bag

FIGURE 2


6" X 6" X 3/16" Steel Jack Plate
Center Holes 5/8" from edge
Drill 4 perimeter holes to allow
for a 1/2" bushing that works
with the smooth portion of the 1/2" rods
Tack weld the lift portion of the
hydraulic jack to the center of the plate
(weld while jack is fully extended as to
not damage it)
6" X 6" X 1/2" Aluminum Piercing Plate
Center holes 5/8" from edge
Drill 4 perimiter hole with 9/16" bit
Drill center holes with 7/16" bit
Tap center using 1/2 X 20 NF tap
Sample piercer is included to ensure
drill bit and tap size as center hole is
crucial to apparatus)
6" X 6" X 3/4" Aluminum Base Plate
Center holes 5/8" from edge
Drill 4 perimeter holes with 23/32" bit
Tap 4 perimeter holes with 1/2 X 13



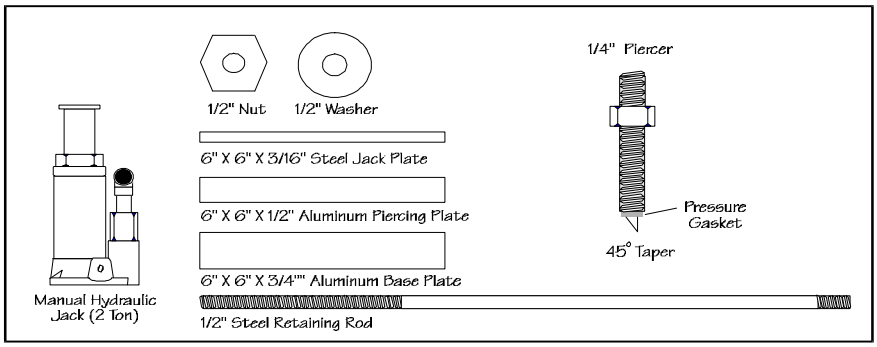

1/2" Nut
1/2" Washer
1/4" Piercer
6" X 6" X 3/16" Steel Jack Plate
6" X 6" X 1/2" Aluminum Piercing Plate
Pressure
Gasket
45° Taper
6" X 6" X 3/4"" Aluminum Base Plate
Manual Hydraulic
Jack (2 Ton)
1/2" Steel Retaining Rod

FIGURE 3