BSR/ASHRAE Standard 172-2017R

Public Review Draft

Method of Test for Insoluble Materials in Lubricant and Refrigerant Systems

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(Complete Draft for Full Review)

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CONTENTS

1. PURPOSE 3
2. SCOPE 3
3. DEFINITIONS 3
4. SAFETY 4
5. APPARATUS 5
6. TEST MATERIAL SPECIFICATION AND THEIR PREPARATION FOR USE 8
7. PROCEDURE 9
8. SIGNIFICANCE OF RESULTS 16
9. REFERENCES 16
Informative appendix A 18

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FOREWORD

Partially soluble substances in the refrigerant/lubricant mixture that are soluble under one set of temperatures and pressures in the refrigeration system but precipitate under other conditions or in select components or locations of the system can adversely impact the operation and reliability of refrigeration and HVAC equipment. Such substances can be transported within the system to select components where they can form permanent deposits. The process of selective precipitation and deposit formation not only may remove a critical component of the refrigerant/lubricant mixture from locations in the system where it is needed but also form undesirable insulating films or constrictions to flow. The impact can be most damaging to fixed-orifice, thermally controlled or electronically controlled expansion devices, where even small deposit formation can dramatically alter the diameter or completely block the orifice. Such changes can dramatically decrease the efficiency of the refrigeration equipment and/or lead to complete failure of the equipment.

1. PURPOSE

The purpose of this standard is to define a test method to determine the formation of insoluble materials in lubricant and refrigerant systems.

2. SCOPE

The test will determine the presence of materials that separate from refrigerant and lubricant mixtures over a range of temperatures and concentrations. The test is based on the precipitation of insoluble material in a lubricant/refrigerant combination. The results can be used to compare lubricants and refrigerants.

3. DEFINITIONS

charging apparatus: a device that allows the accurate vacuum transfer of small volumes of gaseous refrigerants to the sealed tube (or metal test cell) containing precharged lubricant. This apparatus consists of a manifold (metal or glass), vacuum pump, pressure gauge, high-vacuum gauge, refrigerant cylinder, valves, and filling ports. The function of this apparatus is to evacuate the tube, degas the lubricant, add refrigerant, and seal it. It is calibrated so that the required mass of refrigerant is added very accurately by following the change in pressure on the pressure gauge as refrigerant is added to the tube.

lubricant: a stable fluid that is compatible with system components, will form a friction reducing film between rubbing surfaces and seal critical clearances, and has low-temperature transport properties suitable for the application in which it is used [1].

***metal test cell*:** a steel cell containing a charge valve and windows that allow the operator to clearly observe the refrigerant/lubricant mixture for any visual changes. The cell must be rated for the maximum pressure anticipated for the test conditions for the particular refrigerant and possess a pressure relief device.

personal protective equipment (PPE): equipment worn to minimize exposure to a variety of hazards [2]. Examples of PPE include such items as gloves, foot and eye protection, protective hearing devices (earplugs, muffs), hard hats, respirators, face shields, safety shields, and full-body suits.

precipitate: a material that separates from the lubricant and refrigerant mixture at a certain temperature (ex., lubricant additives, contaminants, manufacturing chemicals, extractables).

precipitation temperature: temperatures at which the lubricant and refrigerant mixture forms a precipitate that is visible to the naked eye.

refrigerant: the fluid used for heat transfer in a refrigerating system; the refrigerant absorbs heat and transfers it at a higher temperature and a higher pressure, usually with a phase change. Substances added to provide other functions, such as lubrication, leak detection, absorption, or drying, are not refrigerants [3].

refrigeration equipment: systems containing refrigerant and lubricant for use in HVAC&R applications.

sealed glass tube: *a* borosilicate glass tube with one end formed into a round bottom. The tube is charged with the refrigerant and materials to be tested and then sealed in a rounded tip at the other end [4]. The glass tube must be rated for greater than the maximum pressure anticipated for the test conditions for the refrigerant.

test apparatus: a system of equipment with specific purpose. Such items include the charging manifold and controlled temperature bath.

4. SAFETY

4.1 Introduction. There are inherent hazards when handling sealed glass or metal vessels and the materials being tested. At times, the absolute pressure inside the tube is in excess of 6000 kPa (870 psia). It is not unusual for a sealed glass tube to rupture. Therefore, it is mandatory that the operator follow the safety procedures herein and be aware of the possible hazards at every step of the procedure.

4.2 Safety Shield. The operator shall stand behind a large safety shield made of safety glass or plastic whenever examining a vessel that may be under pressure. This safety shield shall protect the operator’s head, face, and body.

4.3 Personal Protective Equipment. Personal protective equipment (PPE) shall include a face shield, heavy cloth lab coat, and heavy gloves (see Figure 1). A neck protector is optionally added to the mandatory PPE. The face shield shall extend down to protect the neck and upper chest. The lab coat must be of suitable construction to protect the arms and body in the event that a tube under pressure ruptures. The heavy gloves must be insulated to protect the hands from hot temperatures and cold temperatures incurred when handling glass tubes that have been flame sealed or removed from a very cold cooling batch. The heavy gloves must also be of suitable design to protect the hands in the event that a tube ruptures.

Figure 1 Example of suitable PPE for working with glass tubes under pressure.

4.4 Handling Flammable Solvents. Solvents used in this method are highly flammable. Provide adequate ventilation; wear proper gloves (e.g., neoprene, nitrile); and avoid sparks, flame, or heat. Know the location of the nearest fire extinguisher and take appropriate fire precautions

4.5 Charging Manifold. Safeguards must be taken to avoid excessive pressure on the glass system that could cause a rupture while using the charging manifold. Even with these safeguards in the design and use of the equipment, the operator shall wear a face shield, heavy cloth lab coat, and leather gloves and follow other safety precautions for protection in case of an accident. The operator shall also make certain the glass in the charging apparatus is of a wall thickness great enough to withstand vacuum without risk of implosion.

4.6 Eye Protection from High-Temperature Flame. While sealing a glass tube with an oxygen-gas torch, protect the eyes from the yellow flare in the gas flame by using dark glasses (e.g., didymium).

4.7 Refrigerant Handling. Several safety considerations are required for handling refrigerants under pressure. The operator shall be thoroughly familiar with the information about the environmental impact of refrigerants [5] and RSES Service Application Manual, Section 24, regarding refrigerant cylinders [6]. When charging steel cylinders with liquid refrigerant, always reserve 20% of the volume as vapor space (use the refrigerant's room temperature liquid density for this calculation). This allows space for expansion of the liquid when the temperature increases. These cylinders shall also contain appropriately rated pressure relief valves or rupture discs and have known compatibility with the cylinder materials of construction. Due to the incompressible nature of the liquids, a considerable rupture hazard exists when a cylinder or other enclosed volume is completely filled with liquid. A slight rise in temperature in such a system, completely filled with liquid refrigerant, will result in a very large increase in the pressure within the system and potential rupture.

4.8 Handling Liquid Nitrogen or Other Nonflammable Low-Temperature Cooling Solutions. Nonflammable low-temperature cooling solutions in a Dewar flask are used for cooling the sealed glass tube to load it with refrigerant and for a subsequent analysis. When handling such cooling solutions, protective clothing must be worn to prevent frostbite.

4.9 Compatibility of Materials. All materials of construction used in the test apparatus that come in contact with the test materials must exhibit sufficient compatibility to prevent rupture or general failure of the equipment.

5. APPARATUS

This method of test shall be conducted by charging the test materials, including lubricant and refrigerant, in sealed glass tubes or metal test cells. The glass tubes and the manifold used to charge the refrigerant are similar to the tubes and manifold used in ASHRAE Standard 97 [4].

5.1 Sealed Glass Tubes or Metal Test Cells

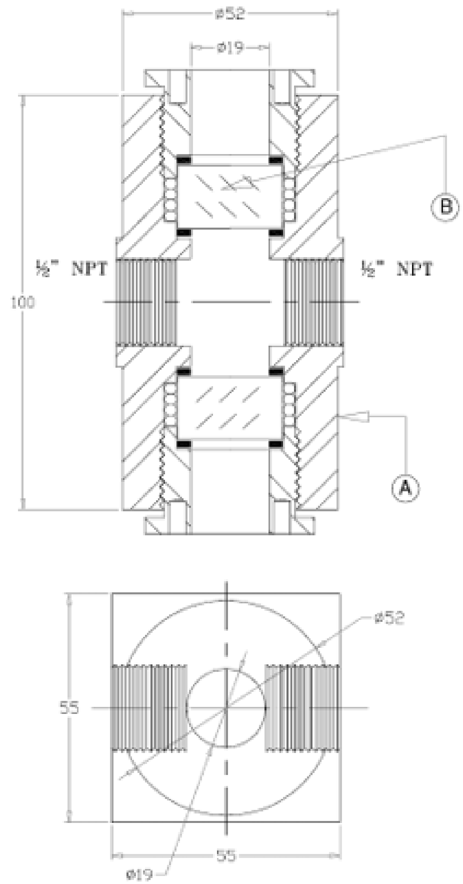
5.1.1 Sealed Glass Tubes. The preparation of the tubes shall be performed by someone skilled in the art of glass blowing. A skilled glass blower shall take into consideration such factors as

a. proper storage of the glass tubing;
b. proper cleanliness of the tubing;
c. cutting to obtain square ends;
d. the use of a small, sharply pointed oxygen-gas flame and proper glass blower's torch;
e. obtaining a uniform wall thickness throughout; and
f. proper safety precautions (see Section 4).

The tubes are made from borosilicate glass tubing. The wall thickness shall be sufficient to protect workers and withstand the temperatures and pressures of the testing. The glass tube is made by sealing one end to form a rounded bottom with the open end fire polished and shall be annealed to remove stress from the glass by an appropriate glass annealing process. The size of the glass tube shall be determined by the total volume of lubricant and refrigerant to be tested. The total volume of lubricant and refrigerant shall not be more than 70% of the total volume of the sealed glass tube.

5.1.2 Metal Test Cell. The metal test cell design shall allow charging of refrigerant and lubricant as well as visualization of the liquid and vapor phases. An example design is shown in Figure 2. The cell is constructed of stainless steel and high-pressure borosilicate sight glass. The metal test cell design shall incorporate a charging port and optional temperature measurement port and pressure relief device. The size of the test cell shall be determined by the total volume of lubricant and refrigerant to be studied. The total volume of lubricant and refrigerant shall not be more than 70% of the total volume of the metal test cell.

5.2 Charging Manifold. The charging manifold is illustrated in Figure 3. This apparatus consists of a manifold (metal or glass), vacuum pump, pressure gage, high-vacuum gage, refrigerant cylinder, valves, and filling ports. The function of this apparatus is to evacuate the tube, add refrigerant along with the test materials, and seal it. It is calibrated so that the refrigerant is added very accurately by following the change in pressure on the vacuum gage as refrigerant is added to the tube or metal test cell.



TECHNICAL DATA SHEET
A) Pressure Vessel Body
B) Borosilicate Toughened Glass
C) MOC 88 304
D) Temperature 150 °C (302 °F)
E) Pressure, 6895 KPa (1000 PSI)
F) O-Ring
G) PTFE Ring (Packing)
H) ½ inch Connection for ¼ inch NPT Ball Valve (Ball Valve Manufacturer: Swagelok)
I) ½ inch NPT Connection for ¼ inch NPT for Thermocouple Temperature Probe

Figure 2 Design drawing of a metal test cell.

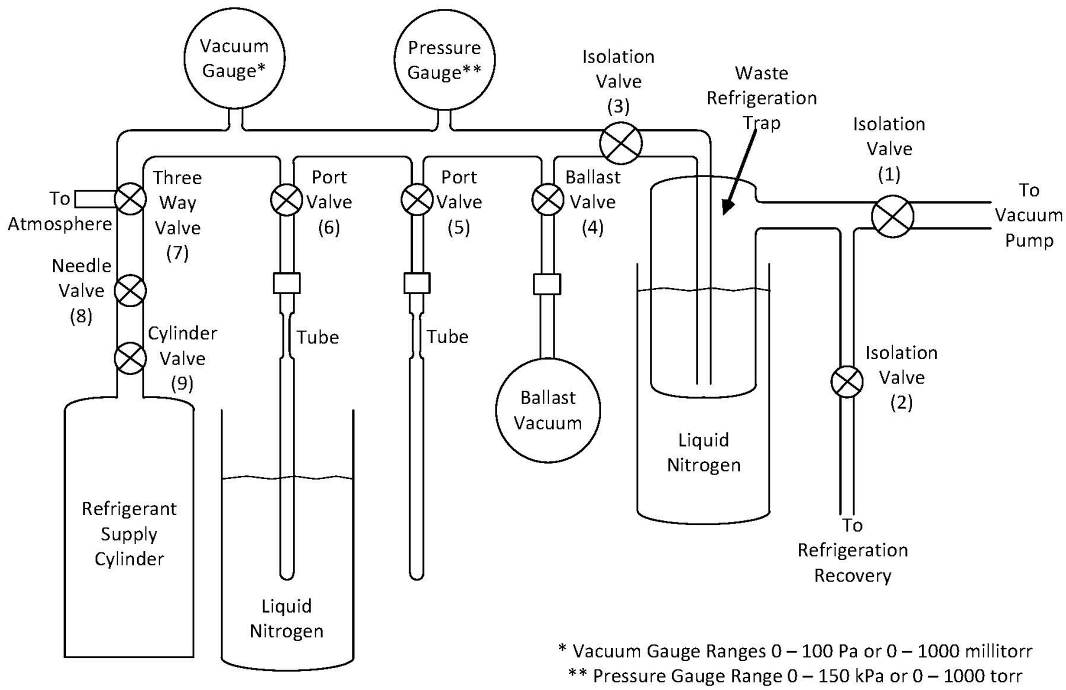


Figure 3 Charging manifold.

5.3 Temperature Controlled Bath. A cooling bath is required to achieve the necessary temperature control and must be large enough for testing three sample tubes at one time. The bath is constructed of a reservoir filled with alcohol, glycol, or acetone and utilizes dry ice, liquid nitrogen, or a refrigeration system for cooling. The use of liquids such as ethanol is necessary because such liquids have the advantage of reduced frosting when the sample tube is raised from the bath for observation. A mechanical stirrer is required to eliminate temperature differences. A small piece of aluminum foil in the liquid will indicate the degree of stirring obtained. When an electric motor is used, it is required to be of a type safe for use above the flammable cooling medium. A wire mesh basket with small openings shall be provided in the bath for the introduction of the dry ice to prevent small pieces of dry ice from coming into contact with the sample tube causing local cold spots. Temperature control using dry ice is achieved by raising or lowering the wire mesh basket.

5.4 Temperature Measuring Devices. The temperature is measured with a calibrated temperature device with accuracy ±1°C (±1°F). Suitable devices are type K, type T, type J, platinum resistance thermometers, or alcohol thermometers. Digital thermocouple must be calibrated according to ASTM E220 [7]. Thermometers meeting the criteria of ASTM standard thermometers 6F or 6C shall be used for this purpose. For accuracy, thermometers shall be calibrated as covered in ASTM Specification E1-14 [8].

5.5 Graph Paper. Graph paper is used with a glass plate as a background to judge the cloudiness of the test sample. The graph shall have a minimum of 20 lines per 1.0 in. (25.4 mm).

5.6 Mechanical Vacuum Pump. A mechanical vacuum pump that provides an absolute pressure of 13 Pa (100 mTorr) shall be used.

6. TEST MATERIAL SPECIFICATION AND THEIR PREPARATION FOR USE

6.1 Glass Tube Cleanliness and Inspection. The tubes must be scrupulously clean. The tubes shall be stored in a sealed container so that they do not collect contaminants. Prior to use, the tubes shall be cleaned to remove all contaminants. After forming the rounded bottom, the tubes shall be flushed with distilled or deionized water, followed by a rinse with acetone or other suitable solvent. The tubes shall be dried at 125°C (257°F) and cooled in a desiccator. Improved cleaning is accomplished by annealing the glass tube for one hour at 580°C (1076°F). After drying, the tubes shall be stored in a desiccator. Before use, inspect all tubes for cleanliness and for any cracks, severe scratches, or other faults in the glass. Discard any defective tubes.

6.2 Alternate—Metal Test Cell Cleanliness and Inspection. The metal test cell must be scrupulously clean and dry. The test cell should be free of any residues and the windows clean. The cell shall be dried by placing on the charging manifold and pulling a vacuum to less than 27 Pa (200 mTorr).

6.3 Refrigerant Purity and Supply Cylinder Requirements. The refrigerant used shall be of known purity, meeting specifications of AHRI Standard 700 [9]. Refrigerant transfer cylinders shall be fitted with a pressure relief device and an adjustable needle valve.

6.4 Lubricant. The refrigeration lubricant shall be isolated from atmospheric moisture and light. The moisture content shall be measured by ASTM D 1533[10] or ASTM D 6304 [11], and the value shall be noted in the final report.

7. PROCEDURE

7.1 Charging Lubricant to Glass Tubes. Load a syringe possessing a needle narrow enough to fit into the top of the glass tube with lubricant to be used in the test. Insert the needle into the top of the glass tube and inject the desired amount of lubricant into the glass tube. Withdraw the syringe from the glass tube.

Informative Note: Be particularly careful to avoid leaving any lubricant on the upper portion of the glass tube as this area will be flame sealed after charging the refrigerant. One way to avoid getting lubricant on the upper part of the glass tube is to use a funnel made from a narrower diameter glass tube, yet still have an inside diameter large enough to accommodate the syringe needle. For example, for glass tubes with an ID = 7 mm, (0.275 in.) it is possible to insert a smaller diameter 2 mm (0.08 in.) outside diameter glass tube into the glass tube to be loaded with lubricant. The smaller diameter tube shall have a handle or clamp at one end to prevent its falling into the larger glass tube. A #18 needle will fit through the 2 mm (0.08 in.) glass tube. The glass tube prevents the last drop of lubricant on the syringe from contacting the upper surface of the glass tube. Purge the tube with a gentle flow of dry nitrogen, stopper or cap the open end, and store in a desiccator.

7.2 Forming Capillary in Glass Tube. Remove the sample tubes from the desiccator, remove the stopper or cap, and place a 30 mm (1.2 in.) length of rubber or PVC tubing over the open end of the tube so that 15 mm (0.6 in.) of the sample tube is covered. Sixty millimeters (2.4 in.) from the unsealed end, reduce the tube to a capillary size just large enough to pass a #18 needle using an oxyacetylene flame.

***Informative Note**:* This operation must be performed by an individual skilled in glass blowing. Commercially available nut and ferrule connections or compression O-ring seals are acceptable alternatives.

7.3 Charging Lubricant to a Metal Test Cell. The procedure is very similar to that used for charging lubricant to a glass sample tube.

7.3.1 Secure the metal test cell in an appropriate clamped support.

7.3.2 Ensure that the ball valve is open.

7.3.3 Load a 5 mL (0.169 oz) syringe with lubricant to be used in the test, and, using a syringe with a locking needle, attach a needle that has sufficient length and gage to allow it to be inserted through the open ball valve and into the test cell chamber. Record the amount of lubricant in the syringe.

7.3.4 Insert the needle through the ball valve opening into the test cell. Inject sufficient lubricant into the cell so that the lubricant level is approximately halfway up the site-glass windows of the cell. Note the volume of lubricant remaining in the syringe. The net lubricant in the cell is the difference between the starting and ending volume of lubricant in the syringe.

7.3.5 Purge the test cell with a gentle flow of dry nitrogen, close the ball valve, and store in a secure location in an upright position.

7.4 Preparing the Manifold and the Refrigerant Tube Loading Apparatus for Use. The interior of this apparatus shall be clean, dry, and thoroughly leak tested. A proper leak-free system shall be capable of being evacuated to a pressure of 2.0 Pa (15 mTorr) and having a rise of no more than 27 Pa (200 mTorr) in an hour.

7.4.1 At the start of the operations, and before the vacuum pump is turned on, the manifold valves must be set as designated in Table 1.

Valve ID	Initial Setting
Isolation Valve 1	Open
Isolation Valve 2	Closed
Isolation Valve 3	Open
Ballast Valve 4	Open
Port Valve 5	Closed
Port Valve 6	Closed
Three Way Valve 7	Open to Atmosphere
Needle Valve 8	Open
Cylinder Valve 9	Closed

Table 1. Initial Manifold Valve Settings.

7.4.2 Fill the Dewar for the waste refrigerant trap with liquid nitrogen.

7.4.3 Turn on the vacuum pump. Change the setting of three-way valve 7 such that it is closed to the atmosphere and open to needle valve 8.

7.4.4 Pump the system down to 2.0 Pa (15 mTorr). Close isolation valve 1 and monitor the vacuum gage to see if there is any rise in pressure. If the pressure remains stable, proceed to step 7.4.5. If not, check and repair any leaks. Restart at step 7.4.1.

7.4.5 Reopen isolation valve 3.

7.4.6 Remove one of the glass tubes with capillary and containing oil (prepared in Section 7.2) from its holder. Hold the glass tube carefully by the flexible tubing covered section and slide it over the stem of port valve 5 on the manifold.

7.4.7 Open port valve 5 slowly to gradually evacuate the tube. In some cases, the lubricant foams vigorously due to dissolved gas. Avoid letting this foam rise to the capillary area. Warm air from a hot-air gun is used to collapse the foam. Tap the tube gently with a pencil covered with a piece of flexible tubing to accelerate the out-gassing. After foaming subsides, continue evacuating the manifold and tube until the pressure is below 6.7 Pa (50 mTorr)

7.4.8 Close port valve 5.

Informative Note: If the manifold accommodates more than one tube, repeat the procedures in steps 7.4.6 and 7.4.7 until all tubes are attached to a port on the manifold and are evacuated. The tubes are now ready for addition of refrigerant.

7.4.9 If the metal test cell is used, follow steps 7.4.1 through 7.4.5.

7.4.10 Attach the metal test cell to port valve 5 through the appropriate connector/adapter, ensuring that the test cell is properly supported.

7.4.11 Open port valve 5 slowly to gradually evacuate the metal test cell. In some cases, the lubricant foams vigorously due to dissolved gas. Avoid letting this foam rise past the ball valve area of the cell. Warm air from a hot-air gun is used to collapse the foam. Tap the cell gently with a pencil covered with a piece of flexible tubing to accelerate the out-gassing. After foaming subsides, continue evacuating the manifold and cell until the pressure is below 6.7 Pa (50 mTorr).

7.4.12 Close port valve 5.

7.5 Charging Refrigerant to the Glass Tubes

7.5.1 CAUTION: Overcharged sealed tubes can be very dangerous and can fail violently when warmed back to room temperature. To guard against this happening, refer to Informative Appendix A, section A1 for guidelines and procedures for ensuring that the proper volume of refrigerant is charged for volume of glass tube used and the volume of oil charged in Section 7.1. Refer to Informative Appendix A, section A2 for details of calibrating the volume of the manifold as well as section A3 for details of charging a specific amount of refrigerant by change in manifold pressure. Before proceeding with step 7.5.2, check to ensure that the manifold valves are set as designated in Table 2.

Valve ID	Initial Setting
Isolation Valve 1	Open
Isolation Valve 2	Closed
Isolation Valve 3	Open
Ballast Valve 4	Open
Port Valve 5	Closed
Port Valve 6	Closed
Three Way Valve 7	Open to Needle Valve 8
Needle Valve 8	Closed
Cylinder Valve 9	Closed

Table 2. Setting of Manifold Valves after Completing Steps in Section 7.4.

7.5.2 Place a Dewar flask under each tube and support with a lab jack. Ensure that the glass tubes do not touch the bottom of the Dewar flasks. Fill each Dewar with liquid nitrogen so that the level is 20 mm (0.8 in.) below the capillary.

7.5.3 Close isolation valve 3 and open cylinder valve 9.

7.5.4 Gradually open needle valve 8 to allow refrigerant to enter the isolated manifold. Monitor the vacuum gage and pressure gage as the refrigerant enters the manifold. Single-component refrigerants are drawn from the liquid phase or vapor phase in the refrigerant supply cylinder. All refrigerant blends (azeotropic or nonazeotropic) must be drawn from the liquid phase in the refrigerant supply cylinder to ensure proper blend component ratios. Close needle valve 8 when the desired amount of refrigerant has been charged to the manifold.

Informative Note: It is preferable that the pressure of the manifold always remain below atmospheric pressure during normal refrigerant transfer operations, but there are circumstances when a positive pressure of refrigerant will make it possible to charge all tubes on the manifold in a single charge of refrigerant from the cylinder. Regardless, never let the pressure rise above 100 kPa (750 torr) in the manifold. Single-component refrigerants are drawn from the liquid phase or vapor phase in the refrigerant supply cylinder. All refrigerant blends (azeotropic or nonazeotropic) must be drawn from the liquid phase in the refrigerant supply cylinder to ensure proper blend component ratios.

7.5.5 Close the refrigerant cylinder valve 9 and ensure that needle valve 8 is also completely closed.

7.5.6 Record the values of both the pressure and vacuum gages. These values will be required to determine the volume of refrigerant charged to the glass tube.

7.5.7 Slowly open port valve 5. The pressure inside the manifold will begin to drop as refrigerant is condensed into the glass tube. Knowing the initial pressure of gas in the manifold, along with the volume of the manifold, the pressure change associated with the transfer of a known mass of refrigerant to the tube is calculated using the Ideal Gas Law. Close port valve 5 when the required change in pressure is obtained that corresponds to the desired mass of refrigerant transferred to the tube.

7.5.8 Repeat steps 7.5.6 and 7.5.7 for the remaining tubes on the manifold.

7.6 Charging Refrigerant to the Metal Test Cell

7.6.1 Before proceeding with step 7.6.2, ensure that the manifold valves are set as designated in Table 3.

Valve ID	**Initial Setting**
Isolation Valve 1	Open
Isolation Valve 2	Closed
Isolation Valve 3	Open
Ballast Valve 4	Open
Port Valve 5	Closed
Port Valve 6	Closed
Three Way Valve 7	Open to Needle Valve 8
Needle Valve 8	Closed
Cylinder Valve 9	Closed

Table 3. Setting of Manifold Valves after Completing Steps in Section 7.5.8.

7.6.2 Place a Dewar flask under the metal test cell and support with a lab jack. Ensure that the test cell does not touch the bottom of the Dewar flask. Fill the Dewar with liquid nitrogen so that the main body of the metal cell is immersed.

7.6.3 Close isolation valve 3 and open cylinder valve 9.

7.6.4 Gradually open needle valve 8 to allow refrigerant to enter the isolated manifold. Monitor the vacuum gage and pressure gage as the refrigerant enters the manifold. Close needle valve 8 when the desired amount of refrigerant has been charged to the manifold.

Informative Note: It is preferable that the pressure of the manifold always remain below atmospheric pressure during normal refrigerant transfer operations, but there are circumstances when a positive pressure of refrigerant will make it possible to charge all tubes on the manifold in a single charge of refrigerant from the cylinder. Regardless, never let the pressure rise above 100 kPa (750 torr) in the manifold. Single-component refrigerants are drawn from the liquid phase or vapor phase in the refrigerant supply cylinder. All refrigerant blends (azeotropic or nonazeotropic) must be drawn from the liquid phase in the refrigerant supply cylinder to ensure proper blend component ratios.

7.6.5 Close the refrigerant cylinder valve 9 and ensure that needle valve 8 is also completely closed.

7.6.6 Record the values of both the pressure and vacuum gages. These values will be required to determine the volume of refrigerant charged to the metal test cell.

7.6.7 Slowly open port valve 5. The pressure inside the manifold will begin to drop as refrigerant is condensed into the metal test cell. Knowing the initial pressure of gas in the manifold, along with the volume of the manifold, the pressure change associated with the transfer of a known mass of refrigerant to the tube is calculated using the Ideal Gas Law. Close port valve 5 when the required change in pressure is obtained (corresponding to the desired mass of refrigerant transferred to the test cell).

7.7 Flame Sealing the Glass Tubes

CAUTION: The sealed glass tube and corresponding waste glass (still attached to the manifold) will still remain hot for some time after the sealing process. Use heat-resistant gloves during all steps during flame sealing.

7.7.1 Lower the lab jack under the tube on port valve 5 sufficiently to allow space to seal the tube at the capillary above the Dewar flask. Hold the tube with gloved hand about 30 mm (1.2 in.) below the capillary (use either cotton or leather gloves). Seal and detach the tube with a glass blowing torch. After sealing, the tip of the tube shall be annealed. This is done by gradually reducing the oxygen supply to the torch until the oxygen is shut off and carbon is deposited on the tube.

7.7.2 Place the tube in an individual pipe chamber or in a heating block, whichever is being used, keeping in mind that the tip of the glass tube is still hot.

7.7.3 Repeat steps 7.7.1 and 7.7.2 for all other glass tubes on the remaining ports of the manifold.

7.7.4 Leave the tubes in the individual pipe chambers or heating block at ambient temperature.

7.8 Alternative Glass Tube Flame Sealing Method

7.8.1 Attach two test tube clamps—one on either side of the tube—just below the capillary. Lower the lab jack so that the clamps are about 30 mm (1.2 in.) above the top of the Dewar. Heat the capillary area with a glass blowing torch to seal the tube. As the glass becomes molten, the tube gently falls until the clamps rest on top of the Dewar flask. Complete the seal and anneal as described in Section 7.7.1.

7.8.2 Place the sealed tube in an appropriate form of secondary containment to allow them to warm to room temperature. Acceptable forms of containment would be a double-end threaded black pipe with a pipe screw cap at each end.

7.8.3 Repeat steps 7.8.1 and 7.8.2 for all other glass tubes on the remaining ports of the manifold.

7.8.4 Place the tubes in a safe location until they have reached ambient temperature before removing from the secondary containment.

7.9 Unused Refrigerant Recovery and Securing the Charging Manifold

7.9.1 After all tubes have been charged with refrigerant and flame sealed, the valves must be set as shown in Table 4.

Valve ID	Setting after Flame Sealing of Tubes
Isolation Valve 1	Open
Isolation Valve 2	Closed
Isolation Valve 3	Closed
Ballast Valve 4	Open
Port Valve 5	Closed
Port Valve 6	Closed
Three Way Valve 7	Open to Needle Valve 8
Needle Valve 8	Closed
Cylinder Valve 9	Closed

Table 4 Manifold Valve Settings after Step 7.7.3 or 7.8.3.

7.9.2 Connect a refrigerant recovery unit to isolation valve 2. Turn on the refrigerant recovery unit to evacuate the line between the unit and isolation valve 2.

Informative Note: A vast array of commercial refrigerant recovery systems are available from any local refrigeration supply store that are used in the routine maintenance of refrigeration equipment. Always ensure that the operator has read the operation manual in its entirety and is completely qualified to operate the device.

7.9.3 Close isolation valve 1 and open isolation valve 2.

7.9.4 Remove the liquid nitrogen bath from the waste refrigerant trap.

7.9.5 Open isolation valve 3.

7.9.6 Continue pumping with the refrigerant recovery unit until the waste refrigerant trap has warmed to ambient temperature.

7.9.7 Close isolation valve 2 and shut off the refrigerant recovery unit. Ensure that all valves on the unit are closed.

7.9.8 Slowly open three-way valve 7 so that it vents to the atmosphere.

7.9.9 Reopen isolation valve 2 to vent the refrigerant recovery line.

7.9.10 Disconnect the refrigerant recovery unit from the manifold. The entire manifold shall now be at atmospheric pressure and vented.

7.10 Inspection of Tubes. After the sealed tubes have been stored in the protective pipes or heating block overnight at ambient temperature, carefully remove each tube for inspection (see Section 4, “Safety”). Wipe each tube with a tissue and inspect the tube for the following:

a. Liquid appearance (single phase, two phases)
b. Proper volume of liquid
c. Appearance of glass, especially in the vicinity of the tube seal
d. Absence of extraneous materials, such as metal fines

If any tube does not pass the visual inspection, dispose of it as described in Section 7.12.

CAUTION: Whenever inspecting tubes, use protective gloves, a safety shield to protect the entire body, a face shield, and a lab coat. Ensure that people in the vicinity are not directly exposed to the tubes.

7.11 Measurement of the Precipitation Temperature

7.11.1 Inspect the tubes for the presence of any insoluble materials. For any tubes showing insoluble materials, report the temperature of formation as the current ambient room temperature as the result.

7.11.2 Immerse the sealed glass tube (or metal test cell) in the temperature-controlled bath containing a temperature measurement device as described in Sections 5.1.3 and 5.1.4. The initial temperature of the bath shall be ambient temperature.

7.11.3 Begin cooling the bath below ambient temperature at a rate of about 0.25°C (0.25°F) per minute.

7.11.4 Hold the sample at a fixed temperature for 10 minutes at every incremental decrease in temperature of 5°C (41°F) to allow time for crystals or solids to precipitate.

7.11.5 Observe the solution of lubricant and refrigerant in the tube for any signs of phase separation or formation of solids as it cools. Graph paper with a minimum of 20 lines per 1.0 in. (25.4 mm) is used as a background grid to aid in detecting the first signs of precipitation or solids formation.

7.11.6 The temperature at which the first visible signs of precipitate formation are observed is recorded at the result of the test.

Informative Note: Phase separation in this test is not the determinant of end point. The end point for this test is the first temperature at which a precipitate or crystals are observed.

7.12 Disposal of Sealed Glass Tubes. For disposal of sealed glass tubes, place the tube in a Dewar containing liquid nitrogen until frozen. Tubes are removed one at a time and scored with a file, and the tops are then snapped off. The tubes without a top are then placed in a suitable open container in a location within a fume hood to allow them to warm up to room temperature. The tubes will off-gas refrigerant as they warm to ambient temperature. After all of the refrigerant has evaporated, the tubes may be washed with a suitable mineral spirit or acetone solvent to remove the lubricant from the tubes. The tubes are disposed of as contaminated glass per local regulations. The solvent rinse shall be disposed of as waste in accordance with local, state, and federal regulations.

8. SIGNIFICANCE OF RESULTS

This test method is intended for the simple and efficient screening of substances for solubility characteristics in a refrigerant/lubricant mixture or for assessing the propensity of a formulated lubricant to form precipitates in the presence of refrigerants. Information to be included in reporting of results should include:

a) Lubricant and refrigerant tested.
b) Concentration of the lubricant in the refrigerant.
c) Temperature at which the change in appearance was observed.
d) A description of the observed change.

Such testing provides valuable information on the suitability for use of lubricant/refrigerant combinations in refrigeration equipment. However, this standard cannot be used as final determination of the suitability for use. Final approval for commercial use of the refrigerant/lubricant mixture is only demonstrated through OEM-sponsored long-term testing in a full system.

9. REFERENCES

1. ASHRAE. 2017. ANSI/ASHRAE Guideline 40-2017, *Refrigeration Oil Description*. Atlanta: ASHRAE.

2. OSHA. 2003. OSHA 3151-12R, *Personal Protective Equipment*. Washington DC: U.S. Department of Labor.

3. ASHRAE (1991). ASHRAE Terminology of Heating, Ventilation, Air Conditioning and Refrigeration. Atlanta, ASHRAE.

4. ASHRAE. 2007. ANSI/ASHRAE Standard 97-2007 (RA2017), *Sealed Glass Tube Method to Test the Chemical Stability of Materials for Use within Refrigerant Systems*. Atlanta: ASHRAE.

5. RSES. 2007. What Technicians Need to Know About Phasing Out HCFC Refrigerants. Service Application Manual Form #620-117, Refrigeration Service Engineers Society, Rolling Meadows, IL.

6. RSES. 2010. Refrigerant Cylinders. Service Application Manual Form #620-52B, Refrigeration Service Engineers Society, Rolling Meadows, IL.

7. ASTM. 2013. ASTM Standard E220, *Standard Test Method for Calibration of Thermocouples by Comparison Techniques*. West Conshohocken, PA: ASTM International.

8. ASTM. 2014. Specification E1, *Standard Specification for ASTM Liquid-in-Glass Thermometers*. West Conshohocken, PA: ASTM International.

9. AHRI. 2012. AHRI Standard 700, *Specification for Fluorocarbon Refrigerants*. Arlington, VA: Air-Conditioning, Heating, and Refrigeration Institute.

10. ASTM. 2012. ASTM Standard D1533, *Standard Test Method for Water in Insulating Liquids by Coulometric Karl Fischer Titration.* West Conshohocken, PA: ASTM International.

11. ASTM. 2007. ASTM Standard D6304, *Standard Test Method for Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fischer Titration.* West Conshohocken, PA: ASTM International.

12. ASHRAE (2017) ASHRAE Handbook, Fundamentals, Chapter 29, Refrigerants. American Society of Heating, refrigerating, and Air-Conditioning Engineers, Atlanta.

(This appendix is not part of this standard. It is merely informative and does not contain requirements necessary for conformance to the standard. It has not been processed according to the ANSI requirements for a standard and may contain material that has not been subject to public review or a consensus process. Unresolved objectors on informative material are not offered the right to appeal at ASHRAE or ANSI.)

Informative Appendix A

A1. EXAMPLE: CALCULATION OF REFRIGERANT PRESSURE IN SEALED TUBES CONTAINING OIL AND 1 GRAM REFRIGERANT

This example uses the information shown below to predict the pressure inside sealed tubes containing a mixture of refrigerant and lubricant at a given temperature. Note that SI units are used for this example. The basic procedure involves calculation of the Specific Volume and Density. The Pressure inside the tube is read from a particular refrigerant Pressure-Enthalpy Diagram using the Density and Temperature of interest. Please keep in mind that the dimensions of the tube, volume of oil, and mass of refrigerant may be different for your experiments.

Tube internal diameter (d): 7 mm
Tube height (excluding solid sealed tip)(h): 180 mm
Volume of oil (OV): 1 mL
Mass of Refrigerant: 1 gram

The values above are used to determine the available free volume inside the tube as shown below.

$$Available\ Free\ Volume\ =\ Total\ Free\ Volume - Oil\ Volume$$
$$Total\ Free\ Volume =\ \pi h \left(\frac{d}{2}\right)^2$$

Example: $\pi(7\ \text{mm}/2)^2 * 180\ \text{mm} = 6{,}927\ \text{mm}^3 = 6.9\ \text{cm}^3 = 6.9\ \text{mL}$

$$Available\ Free\ Volume = 6.9\ \text{mL} - 1\ \text{mL} = 5.9\ \text{mL}$$

The Specific Volume is the Available Free Volume divided by the mass of refrigerant. For this example, we are using 1 gram of refrigerant.

$$Specific\ Volume = Available\ Volume/Grams\ of\ Refrigerant = 5.9\ \text{mL/g}$$

Density is the inverse of the Specific Volume. Convert the Density to units of measure Kg/m^3.

$$Density = \frac{1}{Specific\ Volume} = \frac{1\ g}{5.9\ mL} = 0.169\ g/mL = 169\ Kg/m^3$$

Density in Kg/m^3 units can be used directly to determine the pressure inside the tube at any given temperature in degrees Celsius using a Pressure-Enthalpy Diagram (SI Units) for a given refrigerant. The pressure (in MPA) along the x-axis is taken from the point of intersection of the Specific Density and the Temperature lines. Pressure-Enthalpy Diagrams for most common refrigerants can be found in the ASHRAE Handbook, Fundamentals, Chapter 29[12].

Examples of pressures for several refrigerants at select temperatures for the example above are shown in Table 5.

	R22	R134a	R600a
125 °C	4.6	3.7	3.1
175 °C	5.7	4.8	5.4
100 °C	6.3	5.3	6.5

Table 5. Pressures for Various Refrigerants Determined from Pressure-Enthalpy Charts for a Specific Density of 169 Kg/m³. All pressure values in MPa.

A2. CALIBRATION OF MANIFOLD VOLUME

Refer to Figure 3.

A2.1 Measure and record the weight of a pre-necked glass tube as M_1 and connect to the manifold at port valve 6.

A2.2 Evacuate the manifold, including the ballast and tube, to 13 Pa (100 millitorr) or less. Close Isolation Valve 3 between the refrigerant trap and the manifold.

A2.3 Open three-way valve 7 to the refrigerant supply cylinder and allow a refrigerant to expand into the manifold (note, do not exceed atmospheric pressure). Close three-way valve 7, disconnecting the refrigerant supply. Measure and record as P_1 the pressure inside the manifold.

A2.4 Cool the bottom of the pre-necked glass tube in liquid nitrogen. Open port valve 6 and allow a portion of refrigerant to condense and freeze inside of a tube to a designated pressure, P_2, (typically around, 30 kPa or 228 torr). Close port valve 6 and ballast valve 4 and evacuate the manifold to below 13 Pa (100 millitorr). While evacuating, allow the refrigerant to fully solidify inside the tube, and then reopen port valve 6 to remove trace vapor and evacuate to below 13 Pa (100 millitorr).

A2.5 Seal the tube and remove the stub end from the manifold.

A2.6 Measure and record the weight of the sealed glass tube including the stub end as M_2.

A2.7 The mass of refrigerant, $M_{refrigerant}$, is the difference between M_2 and M_1

A2.8 Determine the density of the vapor refrigerant at P_1 and P_2, record as ρ_1 and ρ_2 (ideal gas law, equation of state, etc., where T and P are known). Estimate the internal volume of the manifold, $V_{manifold}$ and calculate the initial and final mass, m_1 and m_2 from $V_{manifold}*\rho_1$ and $V_{manifold}*\rho_2$. Compare the mass difference of m1 and m2 with the mass of refrigerant, $M_{refrigerant}$ from A2.7 and modify the guess on $V_{manifold}$ until convergence is found.

A2.9 Additional tube sealing iterations may be required to improve the confidence level.

A3. CALCULATION FOR REFRIGERANT CHARGING BY CHANGE IN MANIFOLD PRESSURE

This section does not account for real gas behavior. Users will need to refine their method for precise charging. The example uses SI units. The pressure change required to add the desired quantity of refrigerant (0.84 grams of R-22) is calculated as follows from the ideal gas law:

$PV = nRT$

where

	SI Units
P = pressure change	kPa
V = volume of manifold	L
n = moles of refrigerant	mol
R = gas constant	8.314 kPa · L /mol · K
T = absolute temperature	K

The number of moles of refrigerant is determined by the following relation:

$n = m/MW$,

where m is the refrigerant mass and MW is the refrigerant molecular weight, (example 86.5 g/gmol for R-22). Substituting this ratio for n in the equation $PV = nRT$ leads to the more readily used equation

$P = mRT/MW*V$.

The manifold volume (from A2 above) is used. For the below example, the manifold volume is 0.42 Liters. The temperature for the example is 298 K (25°C).

A3.1 SI Units

$P_{R22} = 0.84 \times 8.314 \times 298/86.5 \times 0.42 = 57.3$ kPa (difference between starting pressure to ending pressure)