STANDARD

ANSI/ASHRAE Standard 172-2017

# Method of Test for Insoluble Materials in Synthetic Lubricants and HFC Refrigerant Systems

Approved by ASHRAE on March 31, 2017, and by the American National Standards Institute on April 1, 2017.

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 ISSN 1041-2336

Approved American National Standard

www.ansi.org

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**NOTE**

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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 synthetic lubricants and hydrofluorocarbon (HFC) 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 gage, high-vacuum gage, refrigerant cylinder, valves, and filling ports. The function of this apparatus is to evacuate the tube, degas the lubricant, add refrigerant along with the test materials, 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 gage 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.

***precipitation temperature:*** the highest temperature at which the working fluid forms a precipitate that is visible to the naked eye.

***refrigerant:*** the working 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:*** borosilicate glass tube 9 mm (0.35 in.) OD × 7 mm (0.27 in.) ID × approximately 180 mm (7.1 in.) long with one end formed into a round bottom. The above are the finished dimensions. The tube is charged with the refrigerant and materials to be tested and then sealed in a rounded tip at the other end [4].

***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

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

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.

**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 any 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 glass tube is made from 9 mm (0.35 in.) OD standard wall borosilicate glass tubing. This tubing has an ID of 7 mm (0.27 in.), and the tube shall be cut into 240 mm (9.45 in.) lengths. One end is sealed to form a rounded bottom with the open end fire polished. The preparation of these 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).

**5.1.2 Metal Test Cell.** The metal test cell shall be of the design shown in Figure 2. The cell is constructed of stainless steel and has two threaded holes on opposite sides of the cell capable of accepting commercially available high-pressure borosilicate site glasses. On the remaining two sides perpendicular to the site glasses are two opposing female NPT threaded holes. One of the threaded holes is designed to accept a male NPT thermocouple fitting, and the second hole is designed to accept a threaded ball valve with a pressure relief device between the cell and the ball valve. The ball valve should have an opening large enough to accept a #18 needle required to charge with lubricant. The ball valve will also have an adapter on top to allow for safe attachment to the charging manifold.

**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