BSR/ASHRAE STANDARD 218P

Public Review Draft

Method of Test for Lubricant and Refrigerant Miscibility Determination

First Public Review (September 2018)
(Draft Shows Complete Proposed New Standard)

This draft has been recommended for public review by the responsible project committee. To submit a comment on this proposed standard, go to the ASHRAE website at www.ashrae.org/standards-research--technology/public-review-drafts and access the online comment database. The draft is subject to modification until it is approved for publication by the Board of Directors and ANSI. Until this time, the current edition of the standard (as modified by any published addenda on the ASHRAE website) remains in effect. The current edition of any standard may be purchased from the ASHRAE Online Store at www.ashrae.org/bookstore or by calling 404-636-8400 or 1-800-727-4723 (for orders in the U.S. or Canada).

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(This foreword 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.)

FOREWORD

Lubricant and refrigerant miscibility is liquid / liquid property under a prescribed temperature and composition. It is relevant to an HVAC system when the refrigerant is in the liquid phase. When a liquid refrigerant and lubricant are mixed together and remain a single-phase solution, they are then referred to as "miscible." Adequate miscibility between the lubricant and refrigerant is desired in most HVAC system designs to maintain performance. With lubricant being miscible within the refrigerant, the viscosity is greatly reduced. This promotes circulation of the lubricant throughout the system and returning it to the compressor. It also minimizes the lubricant film thickness coating the heat exchanger coils, which helps promote maximum heat transfer.

1. PURPOSE

The purpose of this standard is to establish a test procedure to determine the critical solution locus of miscible properties of a lubricant and refrigerant mixture.

2. SCOPE

This standard applies to pure component refrigerant and lubricants and multi-component refrigerant and lubricant mixtures.

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 pre-charged 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 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 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].

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.

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: A borosilicate glass tube with one end formed into a round bottom. The tube is charged with the refrigerant and lubricant to be tested and then sealed in a rounded tip at the other end [4]. The glass tube must be rated for 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.

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.

4. SAFETY

There are inherent hazards when handling pressurized glass tubes or metal vessels. Therefore, the operator shall follow the safety procedures herein and be aware of the possible hazards at every step of the procedure. See Informative Appendix A for special areas of concern.

5. APPARATUS

This standard shall be conducted by charging lubricant and refrigerant in sealed glass tubes or metal test cells.

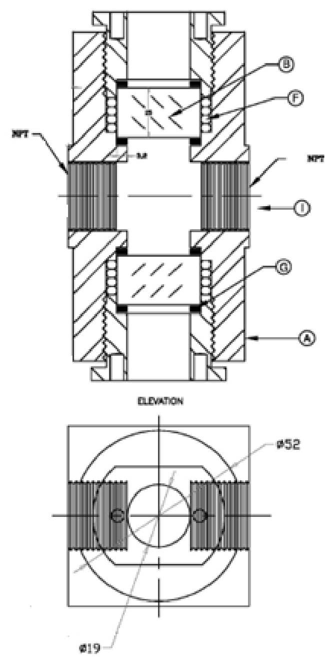
5.1 Sealed Glass Tubes

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 (Appendix A).

5.2 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 1. 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.



TECHNICAL DATA SHEET
A) PRESSURE VESSEL BODY
B) BOROSILICATE TOUGH EN GLASS
C) MOC - SS 304
D) TEMPERATURE - 150 C
E) PRESSURE - 1000 PSI
F) O-RING
G) P.T.F.E RING (PACKING)
H) 1/2" NPT CONNECTION FOR 1/4" NPT BALL VALVE BALL VALVE MAKE - SWAGELOK
I) 1/2" NPT CONNECTION FOR 1/4" NPT THERMOCOUPLE TEMPERATURE PROBE

Figure 1. Design Drawing of a Metal Test Cell

5.3 Charging Manifold

The charging manifold is illustrated in Figure 2. 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 or metal test cell, add refrigerant, and seal it.

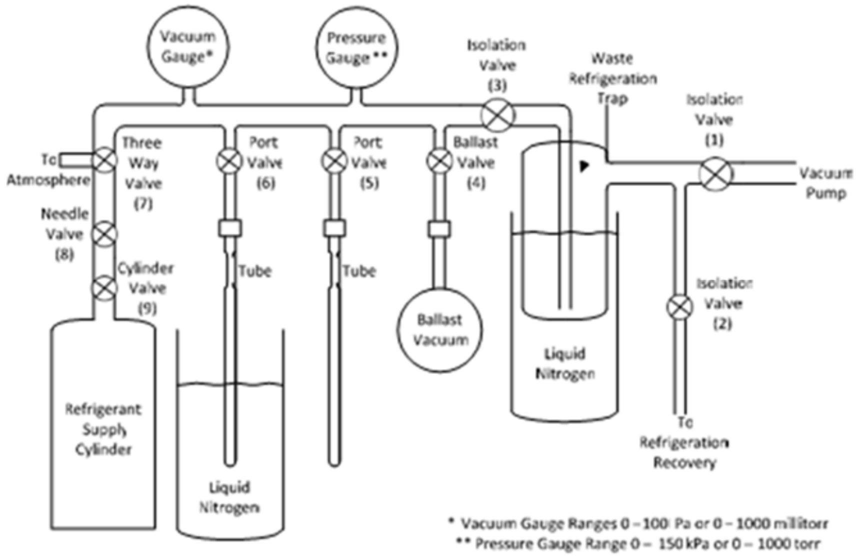


Figure 2. Charging Manifold

5.4 Temperature Controlled Bath

A temperature controlled bath (air or liquid) is required to achieve the necessary temperature control and be large enough for testing multiple samples at one time. The bath is constructed to provide a stable temperature environment for extended periods of time.

5.5 Temperature Measuring Devices

The temperature shall be measured with a calibrated temperature device with accuracy ± 1 ºC (± 1.8 ºF) or better. Suitable devices are type K, type T, type J, thermistor, platinum resistance thermometers, or alcohol thermometers. Digital thermocouple must be calibrated according to ASTM E 220 [5] or ASHRAE 41.1 [6].

5.6 Mechanical Vacuum Pump

A mechanical vacuum pump that provides an absolute pressure of 13 Pa (100 millitorr) shall be used.

6. TEST MATERIAL SPECIFICATION AND THEIR PREPARATION FOR USE

6.1 Glass Tube Cleanliness and Inspection

The tubes shall be scrupulously clean and dry. 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 shall be scrupulously clean and dry. The test cell shall 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 millitorr).

6.3 Refrigerant Purity

The refrigerant used shall meet specifications of AHRI Standard 700 [7], or be of known purity.

6.4 Lubricant

The refrigeration lubricant shall be clean and dry per manufacturer's recommendations. Lubricants can be of two categories: formulated and basestocks. Lubricant shall be identified in the final report.

7. PROCEDURE

7.1 Preparation of Glass Tubes for Testing

Tubes shall be prepared as described in ASHRAE Standard 97 [4].

7.2 Preparation of Metal Test Cell for Testing

Metal test cells shall be prepared as described in ASHRAE Guideline 38 [8]. **[Note:** This guideline is in the process of being published.**]**

Calibrate the cell volume versus height of liquid. Fill the cell to the very top with water/acetone/IPA, then use a syringe to withdraw known volumes of liquid. Measure the height for each withdrawal which gives the approximate vapor volume as a function of height in the sight glass. Report to the nearest 0.1cc.

7.3 Inspection of Glass Tubes or Metal Test Cells

Wipe each tube or test cell window with a tissue to inspect for the following:

a. Proper volume and height of liquid.
b. Appearance of glass, especially in the vicinity of the tube seal or cell window, and the absence of extraneous materials, such as visual particulates. If any tube does not pass the visual inspection, do not use it for the test.

7.4 Measurement of the Miscibility Point

1) Note liquid appearance in sealed tube or metal test cell (single phase, two-phase).
2) Immerse the sealed glass tube (or metal test cell) in the temperature controlled bath containing a temperature measurement device as described in Sections 5.4 and 5.5. The initial temperature of the bath shall be ambient temperature and shall be recorded.
3) Begin heating or cooling the bath starting from ambient temperature. Increase or decrease the bath temperature stepwise. Hold until the test fluid or reference material achieves temperature equilibrium defined as < 2°C (3.6°F) / hour. Ensure vapor / liquid equilibrium by agitating the test cell and return to temperature bath until test fluid or reference material achieves temperature equilibrium defined as < 2°C (3.6°F) / hour.
    a. Observe the solution of lubricant and refrigerant in the test cell for any signs of phase change.
    b. The number of liquid phases at each temperature measurement is recorded.
    c. Temperature shall be further increased or decreased; repeat until desired temperature range is achieved.
4) The lubricant and refrigerant composition shall be corrected for the mass of refrigerant in the vapor space.
    a. For glass tubes, the vapor volume is the inner diameter of the glass tube times the height plus the volume of the cone; measure the distance from the liquid to a point 1/3 of the way up the cone and multiply by the inner diameter (mathematically the same).
    b. For metal cells, see Section 7.2
    c. Then the vapor space corrections are:

$$m_{r,liq} = m_{r,tot} - \rho_{r,vap,sat}(T) * Vol_{vap}$$

and conc becomes

$$conc_{r,liq} = \frac{m_{r,liq}}{m_{r,liq} + m_{lub}}$$

where:

$m_{r,liq}$ = mass of refrigerant in the liquid phase (calculated)

$m_{r,tot}$ = total mass of refrigerant charged into the tube or cell (measured)

$\rho_{r,vap,sat}(T)$ = saturated refrigerant vapor density at the immiscible temperature (equation of state)

Vol_{vap} = volume of the vapor space at the immiscible temperature (measured)

m_{lub} = total mass of lubricant charged into the tube or cell (measured). Lubricant is assumed to be a liquid and be sufficiently small in vapor space as not to affect analysis.

$conc_{r,liq}$ = liquid concentration of the refrigerant in the liquid phase. (calculated)

8. SIGNIFICANCE OF RESULTS

This test method is intended for the simple and efficient screening of lubricant and refrigerant miscibility characteristics. Such testing provides valuable information on screening 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 long term system testing.

8.1 Results Reporting

The following information shall be reported:

1) Description of Test Fluids (Refrigerant and Lubricant).
2) Concentration of refrigerant in lubricant (using calculation to account for vapor space).
3) Number of phases at each test temperature.
4) Any other relevant observations. (Cloudiness, phase inversions, droplets, etc.).

Sample Miscibility Report

Test Cell Temperature C	F	Refrigerant Concentration (%m) 100% 1	95% 2	90% 3	80% 4	50% 5
55	131					
50	122					
45	113					
40	104					
35	95		OP			
30	86		TP			
25	77					
20	68					
15	59					
10	50					
5	45					
0	32					
-5	23					
-10	14					
-15	5					

Solution Description:
- AI - Additive Insoluble
- SH - Slight Haze
- PC - Pale Coloration
- HH - Heavy Haze
- C - Clear
- TP - Two Phase
- OP - One Phase

Figure 3. Sample Miscibility Report

This method does not specify quantitative limits or ranges for pass/fail. The method only describes the general recommendations and common practices for preparing and pre/post-test analysis of the various components being tested (refrigerant, oil). It is left to the method users to establish the appropriate criteria of pass/fail depending on their application requirements.

9. REFERENCES

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

2. OSHA. 2004, OSHA 3151-12R-2004. *Personal Protective Equipment*. US Department of Labor. Washington DC.

3. ASHRAE 2016. ANSI/ASHRAE Standard 34-2016. *Designation and Safety Classification of Refrigerants*. Atlanta. ASHRAE

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

5. ASTM 2013. ASTM Standard E220-13. *Standard Test Method for Calibration of Thermocouples by Comparison Techniques*. ASTM International. West Conshohocken, PA.

6. ASHRAE 2013. ANSI/ASHRAE Standard 41.1-2013. *Standard Method for Temperature Measurement.* Atlanta. ASHRAE

7. AHRI 2012. AHRI Standard 700-2012. *Specification for Fluorocarbon Refrigerants*. Arlington, VA.

8. ASHRAE 2018. ASHRAE Guideline 38-2018. *Guideline for Using Metal Pressure Vessels to Test Materials Used in Refrigeration Systems.* Atlanta. ASHRAE

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INFORMATIVE APPENDIX A— SAFETY

Known potential hazards when using this method of test (MOT) are not limited to the following: high and low temperature, high and low pressure, rupture of test cells, toxic and/or flammable materials, sharps, and intense light.