BSR/ASHRAE Standard 219P

Public Review Draft

Method of Testing the Ability of Liquid Line Filter Driers or Adsorbents to Remove Organic Acid

First Public Review (September 2020)
(Draft Shows Complete Proposed New Standard)

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FOREWORD

This standard is a method of test to calculate the amount of organic acid adsorbed onto an adsorbent material. For the purposes of this standard, the terms "adsorbent desiccant material," "adsorbent material," and "adsorbent" are used interchangeably all to refer to the adsorbent material. This information is useful to HVAC/R system designers to properly size filter driers used in their systems.

Though this standard contains cautionary notations, it does not address all safety concerns. Evaluating materials using this test method can expose the operator to hazards such as both high and low pressures, high and low temperatures, toxic fumes, electric currents, mechanical energy, and other physical hazards. This test method should only be performed by individuals with experience in mitigating these dangers as well as handling refrigerants and general chemical knowledge.

1. PURPOSE

This standard establishes a suitable laboratory apparatus and test method for determining the ability of various adsorbents and refrigerant liquid line filter driers to remove specific organic acids from refrigerant-lubricant mixtures.

2. SCOPE

This standard applies to the measurement of the mass of a specific organic acid removed from a refrigerant-lubricant mixture by a liquid line filter drier containing an adsorbent desiccant material or through a specific adsorbent material which can remove acid by adsorption and/or chemical reaction. The standard applies to methods associated with the extraction of both short and long chain carboxylic acids.

3. DEFINITIONS AND SYMBOLS

3.1 Definitions

Acid Capacity: The grams of organic acid removed per 100 grams of adsorbent material when the equilibrium with a specified concentration of acid dissolved in refrigerant containing 3% lubricant by volume. If a commercial filter-drier is used in the test, then the acid capacity is the grams of acid removed by the specified drier type, under the above conditions.

Adsorbent Material: Materials capable of removing contaminants including water, acids, and other compounds formed from the breakdown of refrigerant and lubricant.

Contaminant Loading: The mass of test contaminant that is added to the test apparatus, grams.

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].

Mixed Acid POE Lubricant: a polyolester lubricant composed of linear and branched carboxylic acids

Organic Acid: organic compound with acidic properties. Common organic acids are the carboxylic acids, whose acidity is associated with the carboxyl group –COOH. Hexanoic acid is a carboxylic acid.

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].

TAN: Total Acid Number with units mg KOH/g of sample as determined by ASTM D974.

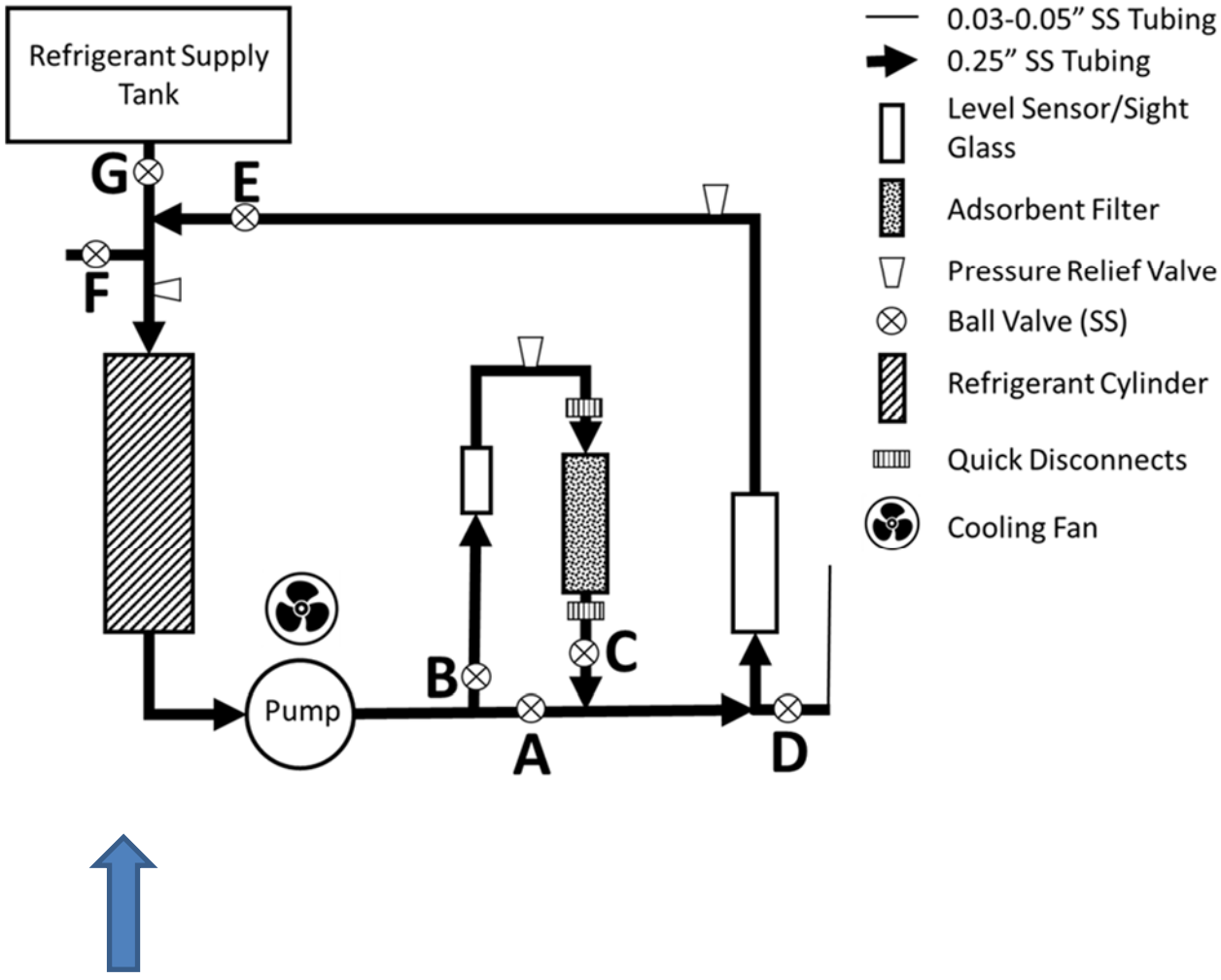
Test Fluid: Oil/Acid/Refrigerant mixture.

4. APPARATUS AND REAGENTS

4.1 Test Apparatus

Test Requirements:

- 2L (2000cc) to 3L (3000cc) SS Reservoir
- Refrigerant/Adsorbent material ratio between 10:1 and 30:1
- 80% Full Reservoir
- 3-10% Lubricant Mix
- 40g (0.088lbs) to 240g (0.529lbs) adsorbent material
- No Starting TAN specified, it is determined



Orientation

4.1.1 Refrigerant Reservoir: Test apparatus shall be operated in an upright orientation during the test and in such a manner that will allow circulation of the test fluids. The refrigerant reservoir shall be a 2L (2000cc) to 3L (3000cc) 304 or 316 stainless steel cylinder. The reservoir shall be in the vertical orientation and positioned with the outlet feeding directly into the refrigerant circulation pump. A tee fitting shall be placed directly above the refrigerant reservoir with ball valves (E, F, & G) placed on all three sides of the tee to complete the flow path, allow the isolation of the refrigerant reservoir, and allow for the oil/acid mixture to be transferred directly into the line leading into the refrigerant reservoir. Valve "F" can also serve as a vent to bleed off air if needed.

4.1.2 Circulation Pump: The circulation pump shall be a magnetic drive pump with seals compatible with the intended refrigerant and pressure rated for the refrigerant type. A cooling device such as a small fan must be used to cool the circulation pump and prevent the refrigerant from transitioning into a vapor state as it leaves the pump. The pump shall be sized to deliver a flow between 0.5 SLPM (0.018cfm) and 1 SLPM (0.035cfm).

4.1.3 Sample Loop: The sample loop shall be vertically oriented following the circulation pump. A ball valve "A" shall be placed at the bottom of the sample loop in between the inlet and outlet legs of the loop for the purpose of diverting the liquid flow through the sample loop when required. Additional ball valves

"B" & "C" shall be placed at the base of the vertical inlet and outlet legs to close off the sample loop inlet allowing the flow to bypass the sample loop.

4.1.4 Filter Drier or Adsorbent Material Holder: Quick connects shall be placed directly before and after the filter drier or adsorbent material holder to allow it to be removed with minimal refrigerant loss.

4.1.5 Sight Glass: A sight glass shall be placed in the vertical inlet leg above ball valve "B" to visually confirm that the refrigerant is in a liquid state as it enters the inlet on top of the sample filter. A flow indicator must be placed in the vertical return line to monitor the flow of test fluid and the filter drier or adsorbent material holder is liquid full.

4.1.6 Sampling Capillary Tube: When using a refrigerant with a boiling point below 23°C (73.4°F), an 8" (203mm) to 10" (254mm) long stainless steel capillary tube with an ID of 0.03" to 0.05" (0.75mm to 1.25mm) shall be installed in the return line to the refrigerant reservoir with ball valve "D" in place to isolate it from the path of flow until a sample is needed. When using a refrigerant with a boiling point at or above 23°C (73.4°F), the size of the sampling tube shall be sized to achieve a controlled flow.

4.1.7 Safety: Three pressure relief valves must be installed for safety, one in the sample loop, the other in the return line, and the third protecting the refrigerant reservoir. Relief valves must be sized according to the lowest pressure rated component of the system including the sample.

4.2 Reagents

4.2.1 Oil Description: The oil used shall not contain additives that may interact with the acid or desiccant due to concerns with additive interactions with adsorbent materials and acid in circulation.

4.2.2 Adsorbent Description: The adsorbent materials used for evaluation shall be loose fill adsorbent material or manufactured commercial filter driers

5. METHOD OF TEST

5.1 Sample Preparation

5.1.1 Loose Adsorbent Material: Weigh out approximately 100g (0.22lbs) of adsorbent material and reactivate it according to the manufacturer's instructions. The test fluid to adsorbent material ratio shall be between 10:1 and 30:1 by weight. (Note that the adsorbent material may lose about 1-1.5% of its weight during reactivation).

5.1.1.1 Preparation of Adsorbent Material Holder: Measure approximately 2.5-3g (0.0055lbs – 0.0066lbs) of silane treated, angel hair-type glass wool and divide it in half. Remove the adsorbent material holder from the test rig, pack half of the glass wool into the bottom of the adsorbent material cylinder, then add 100g (0.22lbs) of the reactivated adsorbent material (amount to obtain a ratio between 10:1 and 30:1 ratio of test fluid to adsorbent material). Tap the side of the cylinder to settle/compact the adsorbent material, and then pack the other half of the glass wool into the top of the adsorbent material holder. Wrap the pipe threads of adsorbent material holder with PTFE thread seal tape and reattach to the test rig. Tighten all connections on the test rig and pump head.

5.1.2 Commercial Driers: Commercial Driers must be tested "AS Received." First, cut open one unused drier to verify the mass of adsorbent material contained within the filter housing. Next, calculate the charge necessary to achieve a ratio of test fluid to adsorbent material between 10:1 and 30:1 while maintaining a test apparatus fill per 5.1.2.1. Report the ratio used.

5.1.2.1 Based on the calculated test fluid charge taking care not to exceed 80% of the apparatus capacity at ambient temperature, calculate the quantity of lubricant required to achieve a final concentration of between 3% and 5% mass.

5.1.3 Preparation of the Oil/Acid Mixture: Prepare an oil /acid mixture with a starting mass of acid to give you a desired TAN at the end of the test.

5.1.3.1 Calculate the TAN at the start of the test.

Informative note: *Desired TAN is typically 0.05 to 0.2.*

5.2 Apparatus Preparation

5.2.1 Addition of Test Fluids: Pressurize the test apparatus with Zero Grade Nitrogen to the lower of 200 psig (1379 kPa) or 80% of the maximum working pressure of the drier. Ensure that no leaks are present then release the pressure to ambient conditions. Next, evacuate the apparatus approximately 1000 microns (0.0142psi) or lower. Weigh the oil/acid mixture container. Draw in the lubricant/acid mixture through valve "F" at top of the refrigerant reservoir. Evacuate the apparatus to less than 1000 microns (0.0142psi) and close valves "B" and "C" to bypass the filter drier. Add the calculated quantity of test fluid to the reservoir through valve "G."

5.2.2 Preconditioning the Apparatus: Energize the circulation pump with the test rig in bypass condition to allow the test fluid to circulate without passing through the filter drier for a minimum of 30 minutes to allow the system to reach equilibrium. Visually verify that there is flow through the system through the site glass. Following this initial 30 minute period, draw a 25ml (25cc) or approximately 30g to 50g (0.066lbs – 0.11lbs) sample through the sample port attached to valve "D" into a pre-weighed container such as a Goetz bulb or Erlenmeyer flask, evaporate the refrigerant, and record the oil removed. Then analyze the lubricant per ASTM D974 Standard Test Method for Acid and Base Number by Color-Indicator Titration to determine a baseline TAN mgKOH/g.

5.3 Equilibrium Testing Procedure

5.3.1 Starting the Test: Redirect the flow through the top of the sample drier by opening valves "B" and "C" and closing valve "A." The refrigerant should flow in the designed direction of flow where indicated and continue to circulate the mixture through the sample filter for a period of 7 days.

Informative note: *If you are doing a kinetic study,* On day 2, 5, and 7 draw approximately 25mL of test fluid through the sample port attached to valve "D*" into a pre weighed container such as a Goetz bulb or Erlenmeyer flask, evaporate the refrigerant, record the mass of the oil removed, then analyze the oil per ASTM D974 Standard Test Method for Acid and Base Number by Color-Indicator Titration*. During this process, you must account for the cumulative acid removed for each TAN sampling in your calculations.

5.4 Recovery of Remaining Materials from the Test Apparatus

5.4.1 Extracting Test Fluids: Extract all remaining fluids through the sample port attached to valve "D" into a pre-weighed 2L (2000cc) Erlenmeyer flask containing several boileezers. *Note: you may need to energize the circulation pump for a brief moment as well as toggle all ball valves to completely empty the test rig.*

5.4.2 Separating Remaining Oil/Acid Mixture from Test Fluid: Place the flask in a water bath at a temperature between 40°C and 60°C (104°F and 140°F) above the boiling point of the test fluid to allow all test fluid to evaporate within or under a ventilation device. Then place the 2L (2000cc) flask along with

the sample drier or adsorbent material apparatus with all valves open into a 100°C (212°F) oven for one hour to bake off the entrained test fluid.

5.4.3 Quantify the Mass of Oil/Acid Mixture Post Test: Remove the 2L (2000cc) flask and filter drier from the oven and allow them to cool to ambient temperature. The filter drier must be capped and allowed to cool in a desiccator to prevent adsorption of water. Immediately reweigh both items. Next, follow Section 6.1 to calculate the M_{oapt}.

5.5 Testing Recovered fluid

Test the TAN of the recovered fluid per *ASTM D974 Standard Test Method for Acid and Base Number by Color-Indicator Titration* .

6. CALCULATIONS AND METHOD OF ANALYSIS

6.1 % Mass of Oil and Acid Mixture Post Test (M_{OAPT}):

EQ.1

$$M_{OAPT} = (M_{fi}-M_{ff}) + M_{er} + M_{tan}$$

M_{fi} = Initial mass of the filter or adsorbent material holder before the test
M_{ff} = Final mass of the filter or adsorbent material holder following the test
M_{er} = Mass of the oil/acid mixture extracted from the remaining test fluid
M_{tan} = Mass of the oil extracted for the TAN sampling

Informative note *regarding Kinetic Study:*

$$M_{OAPT} = (M_{fi}-M_{ff}) + M_{er} + \Sigma M_{tan1} + M_{tan2} + M_{tan3}.....M_{tani}$$

M_{fi} = Initial mass of the filter or adsorbent material holder before the test
M_{ff} = Final mass of the filter or adsorbent material holder following the test
M_{er} = Mass of the oil/acid mixture extracted from the remaining test fluid
M_{tan} = Mass of the oil extracted with TAN sampling

EQ.2

$$\% M_{oapt} = \left(1 - \left(\frac{M_{oai} - M_{oapt}}{M_{oai}}\right)\right) * 100$$

M_{oai} = Initial mass of the oil and acid mixture added to the apparatus
M_{oapt} = Final mass of the oil and acid mixture recovered post test

If the % M_{oapt} is less than 95%, then the test must be repeated

6.2 Acid Remaining Following the Test

EQ. 3 $$M_{ra} = \frac{TAN * MW_{oa}}{56.1*1000}$$

M_{ra} = Mass of Remaining Acid Following the Test
MW_{oa} = Molecular Mass of the Organic Acid

Note: When multiple titrations are made, they are averaged to yield the average weight of acid remaining per gram of oil.

Note: Molecular mass of KOH = 56.1 g/mol (0.124 lbs/mol)

EQ.4 $PAC = M_{ai} - M_{tra}$

PAC = Preliminary Acid Capacity of the Adsorbent Material or Filter

M_{ai} = Initial mass of acid added to the apparatus at the start of the test
M_{tra} = Total mass of the acid remaining in the system after reaching equilibrium at the conclusion of the test.

EQ.5

$$EAC_{100} = \frac{PAC \times 100}{M_{ad}}$$

EAC_{100} = Estimated Acid Capacity per 100g of adsorbent material
PAC = Preliminary Acid Capacity of the Adsorbent Material or filter
M_{ad} = Mass of Adsorbent Material in the test sample

7. REPORT

Test fluid type
Organic Acid used in Test
Oil Type
Oil Viscosity
Ratio of the Test Fluid to Desiccant
Ratio of the Organic Acid to Oil
% Recovery
Starting and ending TAN
Time of test run
Temperature
Mass of adsorbent material and loose fill or commercial type
PAC
EAC_{100}

8. REFERENCES

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3. ASHRAE Standard 41.10, "Standard Methods for Refrigerant Flow Measurement Using Flowmeters," American Society of Heating, Refrigerating and Air-Conditioning Engineers, Inc. Atlanta, GA, 2013.

4. ASHRAE Standard 41.3, "Standard Methods for Pressure Measurement," American Society of Heating, Refrigerating and Air-Conditioning Engineers, Inc. Atlanta, GA, 2014.

5. Carter Stanfield and David Skaves, "Fundamentals of HAVCR" 2nd Ed., p. 480 - p. 498, Air-Conditioning, Heating, and Refrigeration Institute, Pearson, 2013.

6. William C. W., William M. J., John A. T., Eugene S., "Refrigeration & Air Conditioning Technology" 7th Ed., p. 624 - p. 626, Delmar, 2013.

7. Roger C. B., "Flow Measurement Handbook - Industrial Designs, Operating Principles, Performance, and Applications" Cambridge University Press, 2000.

8. Gunderson, D., Kauffman, R. (2002) Test Method for Organic Acid Removal by Adsorbents Used in Liquid Line Filter Driers. University of Dayton Research Institute, ASHRAE Project 1028-TRP.

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INFORMATIVE APPENDIX A – BIBLIOGRAPY

1. *ASHRAE Research Project Report RP-1028, Test Method for Organic Acid Removal by Adsorbents Used in Liquid Line Filter Driers,* University of Dayton Research Institute, Dayton, OH, March 2003.

2. *ARTI MCLR Final Report DOE/CE/23810-108, Equilibrium Point Dryness and Water Capacity of Desiccants with Alternative Refrigerants,* Air-Conditioning and Refrigeration Technology Institute, Arlington, VA, 2000.

3. *ASTM Standard A269 - 15a, Standard Specification for Seamless and Welded Austenitic Stainless Steel Tubing for General Service,* ASTM International, West Conshohocken, PA, 2015.

4. *ASTM Standard A403 - 16, Standard Specification for Wrought Austenitic Stainless Steel Piping Fittings,* ASTM International, West Conshohocken, PA, 2016.

(This appendix is part of this standard. It contains requirements necessary for conformance to the standard.)

NORMATIVE APPENDIX B – CALCULATIONS

B1. EXAMPLE CALCULATIONS

B1.1. Example Test Validation Calculation:

Material Added		
	Oil	54.00 grams
	Acid	4.08 grams
		Total = 58.08 grams
Material Recovered ($\%M_{oapt}$)		
Oil-acid liquid collected following the test after refrigerant evaporation		45.17 grams
Weight of filter-drier, start = 300g, Wt gain after driving off entrenched refrigerant		308.06 grams
Mass of sampled lubricant		3.06 grams
		Total Recovered = 56.29 grams
% Material Recovery = (56.29 / 58.08) * 100		96.9%
If the % of Oil Recovery is greater or equal to 95% mass then the test is validated. If the % of Oil Recovery is less than 95% mass, then the test is not valid		

B1.2 Example PAC (Preliminary Acid Capacity) Calculation

M_{ai} = Initial mass of acid added to the apparatus at the start of the test	4.08 grams
M_{tra} = Total mass of the acid remaining in the system after reaching equilibrium at the conclusion of the test.	0.06 grams
PAC = Preliminary Acid Capacity of the Adsorbent Material or Filter	4.02 grams

B1.3 Example EAC_{100} (Estimated Acid Capacity per 100g of adsorbent material) Calculation

PAC = Preliminary Acid Capacity of the Adsorbent Material or Filter	4.02 grams
M_{ad} = Mass of Adsorbent Material in the test sample	89 grams

$$EAC_{100} = \frac{4.02g \times 100}{89g}$$

$$EAC_{100} = 4.52$$