

BSR/ASHRAE Standard 41.4-2025RA

### **Publication Draft**

# Standard Method for Measuring the Proportion of Lubricant in Liquid Refrigerant

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### ANSI/ASHRAE Standard 41.4-2015, (RA ZOZX) Standard Method for Measuring the Proportion of Lubricant in Liquid Refrigerant

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NOTE

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#### FOREWORD

ASHRAE Standard 41.4 was last revised in 1996 (and reaffirmed in 2006), prior to the global commitment to use refrigerants with lower global warming potential that require the use of synthetic lubricants instead of mineral oils. The methods used in this revision apply to all lubricant-refrigerant combinations. This standard has been revised to comply with ASHRAE's mandatory language requirements and to more clearly specify the test requirements and procedures.

Safety is an important consideration for all procedures involving refrigerant—wear safety glasses and other personal protection equipment.

### 1. PURPOSE

This standard prescribes a method for measuring the proportion of lubricant in liquid refrigerant.

#### 2. SCOPE

**2.1** This standard uses the gravimetric method as the primary method, but alternative methods can be used if those methods are calibrated against the primary method.

**2.2** This standard does not apply to collected samples that contain less than 0.001 g (0.015 grains) of lubricant.

#### 3. DEFINITIONS AND SYMBOLS

#### 3.1 Definitions

*accuracy:* the degree of conformity of an indicated value to an accepted standard value, or true value. The degree of inaccuracy is known as *total measurement error* and is the sum of bias error and precision error.

*bias, fixed, or systematic error*: the difference between the true or actual value to be measured and the indicated value from the measuring system that persists and is a characteristic of the particular instrument or measurement technique.

*error:* the difference between the true value of the quantity measured and an observed value. Because the true value is not known, it is estimated by the mean. The difference between the mean and an observed value is called its *deviation*.

*lubricant circulation rate on the refrigerant basis:* the ratio of the mass of lubricant to the mass of refrigerant in the sample.

*lubricant circulation rate on the sample basis:* the ratio of the mass of lubricant to the mass of refrigerant and lubricant in the sample.

*mean:* the sum of measurement values divided by the number of measurements. Mean is considered the best approximation of the true value.

*packless all-metal diaphragm valves:* valve manufacturers use this term to describe diaphragm valves that have polytetrafluoroethylene (PTFE) gaskets but have no elastomeric seals.

*precision:* the closeness of agreement among repeated measurements of the same characteristic by the same method under the same conditions.

**random error (or precision error):** a statistical error that is caused by chance and is not recurring. There are two types of random error:

*additive errors:* errors that are independent of the magnitude of the observations.

*multiplicative errors:* errors that are dependent on the magnitude of the observations.

*resolution*: the minimum observable difference between two values of a measured characteristic.

*uncertainty:* a measure of the potential error in a measurement or experimental result that reflects the lack of confidence in the result to a specified level.

#### 3.2 Symbols, SI (I-P)

- $M_1 = \text{mass of an empty cylinder assembly, g (grains)}$
- $M_2$  = mass of a cylinder assembly plus the mass of the refrigerant-lubricant sample, g (grains)
- $M_3 = \text{mass of a cylinder assembly plus the lubricant in the sample, g (grains)}$
- $M_4 = \text{mass of an empty beaker, g (grains)}$
- $M_5 = \text{mass of a beaker plus the lubricant in the sample, g}$ (grains)
- $C_R$  = ratio of the mass of lubricant to the mass of refrigerant in the sample, dimensionless
- $C_S$  = ratio of the mass of lubricant to the mass of refrigerant and lubricant in the sample, dimensionless
- $\overline{C}$  = mean of the independent observations of the lubricant circulation rates ( $C_R$  or  $C_S$ ), dimensionless
- n = number of independent observations of lubricant circulation rates, dimensionless

#### 4. REQUIREMENTS

**4.1 Test Plan.** A test plan is a document or other form of communication that specifies the tests to be performed and the required measurement accuracy for each test. Sources of the test plan include, but are not limited to, (a) the person or the organization that authorized the tests to be performed, (b) a method-of-test standard, (c) a rating standard, or (d) a regulation or code.

#### 4.2 Values to Be Determined

**4.2.1** The mean of the lubricant circulation rate on the sample basis (percent).

**4.2.2** The lubricant circulation rate on the sample basis (percent) expressed as the mean plus or minus three times the standard deviation of the mean (SDM) if specified in the test plan.

**4.2.3** Measurement uncertainty, unless otherwise specified in the test plan.

#### FOREWORD

The 2024 edition of Standard 41.4 updates normative references. This standard was prepared under the auspices of ASHRAE. It may be used, in whole or in part, by an association or government agency with due credit to ASHRAE. Adherence is strictly on a voluntary basis and merely in the interests of obtaining uniform guidelines throughout the industry.

#### 5. INSTRUMENTS

**5.1** Instruments and data acquisition systems shall be selected to meet the accuracy limits specified in the test plan.

**5.2** Measurements from the instruments shall be traceable to primary or secondary standards calibrated by the National Institute of Standards and Technology (NIST) or to the Bureau International des Poids et Mesures (BIPM) if a National Metrology Institute (NMI) other than NIST is used. In either case, the indicated corrections shall be applied to meet the required uncertainty stated in subsequent sections. Instruments shall be recalibrated on a regular schedule that is appropriate for each instrument, and calibration records shall be maintained. All instruments shall be applied in a manner that ensures compliance with the accuracy specified in the test plan.

#### 6. EQUIPMENT REQUIRED

**6.1** Analytical Scale. Determine the estimated lubricant mass in a sample using Table 1. The selected analytical scale shall have a resolution that is equal to or greater than the minimum resolution calculated using Equation 1, and shall have an operating range that is capable of weighing the cylinder assembly filled with a sample:

Minimum Resolution =

$$\frac{\left(\frac{\text{Required Accuracy [\%]}}{100}\right)\left(\frac{\text{Sample Mass}}{2}\right) \qquad (1)$$

**6.2 Cylinder Assembly.** Figure 1 shows a schematic that identifies the components of the cylinder assembly and shows the cylinder assembly connected to the refrigerant liquid line access port during sampling. Select a cylinder that meets each of the requirements stated in Section 6.2.1 through 6.2.6.

**6.2.1 Cylinder Internal Volume.** The maximum cylinder assembly internal volume is 50 mL (0.013 gal) for refrigerant system capacities less than 70.4 kW (20 tons), and 500 mL (0.13 gal) for system capacities greater than or equal to 70.4 kW (20 tons). Determine the required minimum internal volume of the cylinder by following Steps 6.2.1(a) through 6.2.1(g).

- a. Use Table 1 to estimate the lubricant sample mass.
- b. Divide the estimated lubricant sample mass (Step 6.2.1[a]) by the expected lubricant circulation rate to obtain the corresponding estimated mass of refrigerant.
- c. Obtain the density of the saturated liquid refrigerant at 25°C (77°F) from REFPROP,<sup>1</sup> or from the refrigerant supplier if the refrigerant is not included in REFPROP.
- d. Divide the estimated mass of refrigerant (Step 6.2.1[b]) by the liquid refrigerant density (Step 6.2.1[c]) and by 0.8 to obtain a minimum cylinder assembly internal volume.
- e. Calculate the internal volume of the tubing and valves that are an integral part of the cylinder assembly (see Figure 1).
- f. Subtract out the internal volume of the tubing and valves (Step 6.2.1[e]) from the minimum cylinder assembly internal volume (Step 6.2.1[d]) to obtain the minimum cylinder internal volume.
- g. Select a cylinder that has an internal volume greater than the minimum cylinder internal volume (Step 6.2.1[f]).

**6.2.2** Cylinder Assembly Components. Figure 1 shows a schematic of a cylinder assembly. The cylinder assembly shall include a pressure relief valve or rupture disc (described in Section 6.2.5), three diaphragm valves, tube fittings, and connecting tubing. The diaphragm valves shall be manually operated all-metal packless diaphragm valves. Diaphragm valves of this type are commercially available with pressure ratings up to 241 bar (3500 psig).

**6.2.3 Cylinder Assembly Materials.** All cylinder assembly materials shall be corrosion resistant, chemically compatible, and pressure-rated in accordance with Section 6.2.4.

**6.2.4 Cylinder Assembly Pressure Rating.** The entire cylinder assembly shall have a working pressure no less than the refrigerant saturation pressure at  $85^{\circ}$ C ( $185^{\circ}$ F), or two times the maximum refrigerant system operating pressure, whichever is greater, in accordance with UL 207.<sup>2</sup>

**6.2.5 Cylinder Assembly Pressure Relief.** The cylinder assembly shall incorporate a relief valve or a rupture disc set at a pressure limit that does not exceed the stamped cylinder working pressure.

**6.2.6 Cylinder Assembly Sealing Materials.** Sealing materials (pipe thread sealant, o-ring seals, and valve packing) shall be chemically compatible with both the refrigerant and lubricant.

**6.3 Hot Plate.** A laboratory hot plate will be used to evaporate residual solvents from a beaker.

**6.4 Oven.** The oven shall be a ventilated forced-air oven that is capable of operating at  $150^{\circ}C$  ( $302^{\circ}F$ ).

**6.5 Beakers.** Beakers will be used to determine the lubricant sample mass where lubricant circulation rates are <1%.

6.6 Vacuum Pump and Vacuum Gauge. The vacuum pump and a vacuum gage shall be capable of creating and measuring static absolute pressures of 23.3 Pa ( $175 \mu m$  Hg).

6.7 Solvent. Select a solvent for dissolving the lubricant based upon the lubricant as prescribed by Section 6.7.1 or 6.7.2. The purity of the selected solvent shall be reagent grade or higher, and the selected solvent shall have a normal boiling temperature  $\leq 150^{\circ}$ C (302°F).

**6.7.1** Select hexane, pentane, dichlorofluoroethane for mineral oils, alkyl benzene (AB) lubricants, and polyalphaolefin (PAO) lubricants.

**6.7.2** Select acetone, refrigerant R245fa, or refrigerant R4310 for polyolester (POE) and polyalkylene glycol (PAG) lubricants.

**6.8 Refrigerant Recovery System.** A refrigerant recovery system shall be used to remove the refrigerant from the cylinder assembly and transfer that refrigerant into an approved storage container.

#### 7. TEST METHODS

**7.1 Primary Method**. This section provides an overview of the primary test method. Section 8 describes the detailed primary test procedures. The primary test method uses the overall steps listed in Sections 7.1.1 and 7.1.2.

Typical Applicati	ion	Domestic Refriger:	e ators	Residen Air Con	tial and L ditioners	ight Com	imercial	Superma Refriger Systems	arket ation	Building Air Conditioners and Large Chillers					
Refrigera capacity,	nt system kW (tons)	0.7 (0.2)	1.76 (0.5)	3.52 (1)	17.6 (5)	26.4 (7.5)	70.4 (20)	52.8 (15)	141 (40)	211 (60)	422 (120)	458 (130)	1408 (400)	1760 (500)	17600 (5000)
Approximate refrigerant charge, kg (lb)		0.20 (0.44)	0.45 (1.0)	1.12 (2.5)	5.67 (12.5)	8.44 (18.75)	22.70 (50)	17.00 (37.5)	45.4 (100)	68.1 (150)	136.2 (300)	147.55 (325)	454.00 (1000)	567.50 (1250)	5675 (12500)
Approxin Lubricant L (gal)	nate t Charge,	0.19 (0.05)	0.42 (0.11)	0.50 (0.13)	2.50 (0.66)	3.79 (1.00)	10.0 (2.64)	2.0 (0.53)	5.3 (1.40)	8.0 (2.12)	16.0 (4.25)	17.4 (4.6)	26.5 (7.0)	26.5 (7.0)	26.5 (7.0)
						Lubrica	nt in Sam	ple, g (gra	ains)						
ent)	0.10%	0.043 (0.662)	0.043 (0.662)	0.043 (0.662)	0.043 (0.662)	0.043 (0.662)	0.128 (1.971)	0.128 (1.971)	0.425 (6.545)	0.425 (6.545)	0.425 (6.545)	0.425 (6.545)	0.425 (6.545)	0.425 (6.545)	0.425 (6.545)
	0.20%	0.085 (1.309)	0.085 (1.309)	0.085 (1.309)	0.085 (1.309)	0.085 (1.309)	0.255 (3.927)	0.255 (3.927)	0.85 (13.09)	0.85 (13.09)	0.85 (13.09)	0.85 (13.09)	0.85 (13.09)	0.85 (13.09)	0.85 (13.09)
e (perc	0.50%	0.213 (3.28)	0.213 (3.28)	0.213 (3.28)	0.213 (3.28)	0.213 (3.28)	0.638 (9.825)	0.638 (9.825)	2.125 (32.73)	2.125 (32.73)	2.125 (32.73)	2.125 (32.73)	2.125 (32.73)	2.125 (32.73)	2.125 (32.73)
ion Rat	1.00%	0.425 (6.545)	0.425 (6.545)	0.425 (6.545)	0.425 (6.545)	0.425 (6.545)	1.275 (19.64)	1.275 (19.64)	4.25 (65.45)	4.25 (65.45)	4.25 (65.45)	4.25 (65.45)	4.25 (65.45)	4.25 (65.45)	4.25 (65.45)
irculat	2.00%	0.85 (13.09)	0.85 (13.09)	0.85 (13.09)	0.85 (13.09)	0.85 (13.09)	2.55 (39.27)	2.55 (39.27)	8.5 (130.9)	8.5 (130.9)	8.5 (130.9)	8.5 (130.9)	8.5 (130.9)	8.5 (130.9)	8.5 (130.9)
icant C	5.00%	2.125 (32.725)	2.125 (32.73)	2.125 (32.73)	2.125 (32.73)	2.125 (32.73)	6.375 (98.18)	6.375 (98.18)	21.25 (327.3)	21.25 (327.3)	21.25 (327.3)	21.25 (327.3)	21.25 (327.3)	21.25 (327.3)	21.25 (327.3)
Lubri	10.00%	4.25 (65.45)	4.25 (65.45)	4.25 (65.45)	4.25 (65.45)	4.25 (65.45)	12.75 (196.35)	12.75 (196.35)	42.5 (654.5)	42.5 (654.5)	42.5 (654.5)	42.5 (654.5)	42.5 (654.5)	42.5 (654.5)	42.5 (654.5)
	15.00%	6.375 (98.175)	6.375 (98.18)	6.375 (98.18)	6.375 (98.18)	6.375 (98.18)	19.13 (294.6)	19.13 (294.6)	63.75 (981.8)	63.75 (981.8)	63.75 (981.8)	63.75 (981.8)	63.75 (981.8)	63.75 (981.8)	63.75 (981.8)

TABLE 1 Estimated Lubricant In Sample (Informative)

Note: The shaded area indicates a required analytical scale resolution of 0.0001 g (0.0015 grains). The unshaded area indicates a required analytical scale resolution of 0.01 g (0.15 grains).

### 7.1.1 Overall Steps for Expected Lubricant Circulation Rates ≥1%

- a. Weigh a clean and evacuated cylinder assembly; record  $(M_1)$ .
- b. With the refrigerant system operating at steady-state conditions, collect a refrigerant and lubricant sample from an access port on the liquid line.
- c. Weigh the cylinder assembly containing the refrigerant and lubricant sample; record  $(M_2)$ .
- d. The mass of refrigerant and lubricant sample is  $(M_2 M_1)$ .
- e. Recover the refrigerant from the cylinder assembly using a refrigerant recovery system.
- f. Weigh the cylinder assembly containing only lubricant; record  $(M_3)$ .
- g. The mass of the lubricant sample is  $(M_3 M_1)$ .
- h. The mass of refrigerant collected into the cylinder assembly sample is  $(M_2 M_3)$ .
- i. The lubricant circulation rate on the sample basis is  $(M_3 M_1)/(M_2 M_1)$ , and the lubricant circulation rate on the refrigerant basis is  $(M_3 M_1)/(M_2 M_3)$ .

# 7.1.2 Overall Steps for Expected Lubricant Circulation Rates ${<}1\%$

a. Weigh a clean and evacuated cylinder assembly; record  $(M_1)$ .



FIGURE 1 Schematic of a cylinder assembly.

- b. With the refrigerant system operating at steady-state conditions, collect a refrigerant and lubricant sample from an access port on the liquid line.
- c. Weigh the cylinder assembly plus the refrigerant and lubricant sample; record  $(M_2)$ .
- d. The mass of refrigerant and lubricant sample is  $(M_2 M_1)$ .
- e. Recover the refrigerant from the cylinder assembly using a recovery system.
- f. Weigh a clean beaker; record  $(M_4)$ .
- g. Transfer the lubricant from the cylinder assembly to the beaker.
- h. Weigh the beaker plus lubricant; record  $(M_5)$ .
- i. The mass of the lubricant sample is  $(M_5 M_4)$ .
- j. The lubricant circulation rate on the sample basis is  $(M_5 M_4)/(M_2 M_1)$ , and the lubricant circulation rate on the refrigerant basis is  $(M_5 M_4)/(M_2 M_3)$ .

Three (3) independent determinations differing by no more than 5% are required unless otherwise specified in the test plan. The number of samples and the results for each shall be recorded in the test report as described in Section 11 unless otherwise specified in the test plan.

**7.2 Alternative Methods.** Alternative test methods are permitted provided that those methods are calibrated against the primary method. The calibration sheet shall be included in the test report.

#### 8. TEST PROCEDURES

**8.1 Clean the Cylinder Assembly.** Rinse the interior surfaces with solvent as prescribed in Section 6.7, dry in a vented forced-air oven for 15 minutes at 150°C (302°F), and then cool to room temperature.

8.2 Evacuate the Cylinder Assembly. Connect the vacuum pump and vacuum gage to Diaphragm Valve B shown in Figure 1. Close Diaphragm Valve A and open Diaphragm Valves B and C. Evacuate the cylinder assembly to an absolute static pressure less than 23.3 Pa (175  $\mu$ m Hg), release the vacuum, evacuate the cylinder assembly to an absolute static pressure less than 23.3 Pa (175  $\mu$ m Hg) a second time, and then close Diaphragm Valve B, leaving Diaphragm Valve A closed and Diaphragm Valve C open. Disconnect the vacuum pump and vacuum gage.

**8.3 Weigh Empty Cylinder Assembly.** Weigh the empty and evacuated cylinder assembly using an analytical scale with the resolution specified by Section 6.1, and then record the mass of the empty and evacuated cylinder assembly as  $(M_1)$ .

**8.4 Connect the Cylinder Assembly.** Connect the cylinder assembly to the refrigerant system as shown in Figure 1.

**8.5 Sampling.** With the refrigerant system operating at steady-state conditions, open the access valve if it is not a Schrader valve that was automatically opened when the cylinder assembly was connected in Section 8.4. Open Diaphragm Valve A to collect the refrigerant-lubricant sample. Close Diaphragm Valve A after pressure equalization is sensed

audibly or after 5 minutes, whichever occurs first. Disconnect the cylinder assembly.

8.6 Weigh the Cylinder Assembly and Sample. After removing all visible moisture from its surface, use an analytical scale with the resolution specified by Section 6.1 to weigh the cylinder assembly and sample, and then record the mass of the cylinder assembly and sample as  $(M_2)$ .

8.7 Recover the Refrigerant. Use a refrigerant recovery system to remove the refrigerant from the cylinder assembly. If the expected lubricant circulation rate is  $\geq 1\%$ , continue to Section 8.8. Otherwise, skip to Section 8.11.

**8.8 Weigh the Cylinder Assembly and Lubricant.** Use an analytical scale with the resolution specified in Section 6.1 to weigh the cylinder assembly and lubricant, and record the mass of the cylinder assembly and lubricant as  $(M_3)$ .

**8.9 Lubricant Mass.** The mass of lubricant in the sample is then determined by subtracting the mass of the cylinder assembly  $(M_1)$  from the mass of the cylinder assembly and lubricant  $(M_3)$ .

**8.10 Determine Lubricant Circulation Rate.** Calculate the lubricant circulation rate using Equations 2, 6, and 7 in Section 9 for the sample basis, or Equations 4, 6, and 7 for the refrigerant basis. Proceed to Section 10.

**8.11 Weigh a Beaker.** Weigh an empty and clean beaker using an analytical scale with the resolution specified in Section 6.1, and record the mass of the empty and evacuated cylinder assembly as  $(M_4)$ .

**8.12 Transfer Sample Lubricant to Beaker.** Transfer the lubricant from the cylinder assembly to the beaker. In a laboratory hood, rinse the interior surfaces of the cylinder assembly with a reagent-grade solvent (see Section 6.8) to ensure that all lubricants are removed from the cylinder assembly.

**8.13 Remove Solvents.** In the laboratory hood, the solvent shall be evaporated from the beaker using a heat source to simmer but not boil the solvent. Next, the beaker shall be transferred into a forced-air vented oven set at  $150^{\circ}C$  ( $302^{\circ}F$ ) for a minimum of two hours to remove any remaining trace of solvent. The beaker shall then be transferred to a desiccator until cool.

**8.14 Weigh the Beaker and Lubricant.** Weigh the beaker and lubricant using an analytical scale with the resolution specified in Section 6.1, and record the mass of the empty and evacuated cylinder assembly as  $(M_5)$ .

**8.15 Lubricant Mass.** The mass of lubricant in the sample is then determined by subtracting the mass of the beaker  $(M_5)$  from the mass of the cylinder assembly and lubricant  $(M_5)$ .

**8.16 Determine Lubricant Circulation Rate.** Calculate the lubricant circulation rate using Equations 3, 6, and 7 in Section 9 for the sample basis, or Equations 5, 6, and 7 for the refrigerant basis. Proceed to Section 10.

#### 9. CALCULATIONS

The lubricant circulation rate shall be calculated using Equations 2 through 7.

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TABLE 2 Example Test Record for Each Refrigerant-Lubrican	Sample
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Sample Source:		Identification Numb	er:
Technician: Date:	Cylinde	er Number:	Scale ID:
Run Number: Time Begin:	End:	Ambient Ter	mperature:
Data:	Mass, g	(grains):	
Empty cylinder assembly $(M_1)$			
Cylinder assembly + Refrigerant + Lubricant $(M_2)$			
Cylinder assembly + Residual lubricant $(M_3)$			
Empty beaker (M <sub>4</sub> )			
Beaker + Lubricant (Ms)			

**9.1 Lubricant Circulation Rate, Sample Basis.** Calculate  $C_S$  using Equation 2 if the lubricant circulation rate is  $\geq 1\%$ . Calculate  $C_S$  using Equation 3 if the lubricant circulation rate is <1%.

$$C_{S} = \frac{M_{3} - M_{1}}{M_{2} - M_{1}}$$
(2)

$$C_{S} = \frac{M_{5} - M_{4}}{M_{2} - M_{1}} \tag{3}$$

**9.2 Lubricant Circulation Rate, Refrigerant Basis.** Calculate  $C_R$  using Equation 4 if the lubricant circulation rate is  $\geq 1\%$ . Calculate  $C_R$  using Equation 5 if the lubricant circulation rate is < 1%.

$$C_R = \frac{M_3 - M_1}{M_2 - M_3} \tag{4}$$

$$C_R = \frac{M_5 - M_4}{M_2 - M_3}$$
(5)

**9.3 Mean Lubricant Circulation Rate.** Calculate the mean of the lubricant circulation rates using Equation 6.

$$\overline{C} = \frac{C_1 + C_2 + C_3 \dots C_n}{n}$$
 (6)

**9.4 Standard Deviation of the Mean.** Calculate the standard deviation of the mean (SDM) of the lubricant circulation rate using Equation 7.

SDM = 
$$\frac{\sqrt{(C_1 - \overline{C})^2 + (C_2 - \overline{C})^2 + (C_3 - \overline{C})^2 \dots}}{\sqrt{n^2 - n}}$$
 (7)

#### **10. UNCERTAINTY CALCULATIONS**

Accompanying the lubricant circulation rate measurement shall be an estimate of the uncertainty in the lubricant circulation rate measurement unless otherwise specified in the test plan. This is referred to as an *error analysis*, as outlined in ASHRAE Guideline 2.<sup>3</sup> The following terms have been copied from Guideline 2 and included in the definitions in Section 3: *accuracy; bias, fixed, or systematic error; error; mean; precision; random error (or precision error); resolution;* and *uncertainty.* Informative Annex B provides a recommended method to express uncertainty and describes uncertainty calculation procedures. Informative Annex D provides a detailed example of lubricant circulation rate uncertainty calculations.

#### **11. TEST REPORT**

Table 2 shows an example of the format and data that shall be recorded for each lubricant circulation rate sample and included in the test report. In addition to the data shown in Table 2, the test report shall include the data specified in Sections 11.1 through 11.5 unless otherwise specified in the test plan.

**11.1** The number of independent observations of lubricant circulation rate (n).

11.2 The lubricant circulation rate on the sample basis  $(C_S)$ .

**11.3** The lubricant circulation rate on the refrigerant basis  $(C_R)$ .

**11.4** The mean of the independent observations of lubricant circulation rate  $(\overline{C})$ .

**11.5** The standard deviation of the mean (SDM) of the lubricant circulation rate.

#### **12. NORMATIVE REFERENCES**

- 1. NIST. 2013. Thermodynamic Properties of Refrigerants and Refrigerant-Mixtures Database (REFPROP), version 9.1 National Institute of Science and Technology, Gaithersburg, Maryland.
- 2. UL. 2001. UL 207-2001, Standard for Safety: Refrigerant Containing Components and Accessories, Nonelectrical. Underwriters Laboratories, Northbrook, IL.
- 3. ASHRAE. 2014. ASHRAE Guideline 2-2010 (RA 2014), Engineering Analysis of Experimental Data. Atlanta: ASHRAE.

#### INFORMATIVE ANNEX A INFORMATIVE REFERENCES AND BIBLIOGRAPHY

A-1. ASHRAE. 2009. ASHRAE Handbook—Fundamentals, Chapter 29, "Refrigerants." Atlanta: ASHRAE. www.ashrae.org.

- A-2. ASHRAE. 2010. ANSI/ASHRAE Standard 23.1, Methods of Testing for Rating the Performance of Positive Displacement Refrigerant Compressors and Condensing Units That Operate at Subcritical Temperatures. Atlanta: ASHRAE.
- A-3. ASME. 2005. ANSI/ASME PTC 19.1, *Test Uncertainty*. New York: ASME. www.asme.org.
- A-4. ISO. 2005. ISO 17025/IEC, General Requirements for the Competence of Testing and Calibration Laboratories. Geneva, Switzerland: International Organization for Standardization. www.iso.org.
- A-5. NIST. 2012. NIST Handbook 44: Specifications, Tolerances, and other Technical Requirements for Weighing and Measuring Devices. Gaithersburg, MD: National Institute of Science and Technology. www.nist.gov.

#### INFORMATIVE ANNEX B METHODS FOR UNCERTAINTY

#### B1. RECOMMENDED METHOD TO EXPRESS UNCERTAINTY

All assumptions, parameters, and calculations used in estimating uncertainty shall be clearly documented prior to expressing any uncertainty values. Uncertainty shall then be expressed in the following form:  $v = m \pm w$ ; P percent

where

```
v = variable
```

- m = best value
- w = uncertainty estimate for the best value
- P = confidence level

For example, lubricant circulation rate =  $1.3\% \pm 0.12\%$ ; 95% states that the best value for the lubricant circulation rate is believed to be 1.3% with a 95% probability that the true value lies within  $\pm 0.12\%$  of this value.

#### **B2. UNCERTAINTY CALCULATION PROCEDURE**

ASHRAE Guideline 2-2010 (RA 2014)<sup>3</sup> and Section 4.2 of ANSI/ASME PTC 19.1-2005, *Test Uncertainty*<sup>A-3</sup> describe a thorough procedure to account for all items that may affect the uncertainty analysis. This procedure is applied to the example of uncertainty calculations in Informative Annex D.

#### INFORMATIVE ANNEX C GUIDANCE REGARDING LUBRICANT SAMPLING PROCEDURE

#### C1. STEPS

**C1.1 Step 1: Estimate the Refrigerant Charge in the System.** When intending to continue system operation after lubricant sampling, avoid removing more than 1% of the system charge to prevent an operating condition that could be detrimental to the equipment.

**C1.2 Step 2: Select a Sample Cylinder Size.** In accordance with Section 6.2.1, the maximum cylinder assembly size (internal volume) is 50 mL (0.013 gal) for refrigerant system

capacities less than 70.4 kW (20 tons), and 0.50 L (0.13 gal) for system capacities greater than or equal to 70.4 kW (20 tons).

The smallest cylinder size is dependent on the scale accuracy; the smallest practical size is 3 mL  $(7.93 \times 10^{-4})$  gal.

Section 6.2.1 provides guidance for selecting a cylinder size for a given application.

**C1.3 Step 3: Analytical Scale Resolution.** The shaded area in Table 1 indicates that an analytical is required to have a resolution of 0.0001 g (0.00154 grains).

For the unshaded area in Table 1, a top-loading scale with a resolution of 0.01 g (0.154 grains) is acceptable.

Some knowledge of the expected lubricant circulation rate is helpful:

- a. For small rotary compressors, lubricant circulation rates of 1.0% to 1.3% are typical.
- b. For reciprocating and scroll compressors, lubricant circulation rates of 0.5% to 1.0% are typical.
- c. For large chillers, lubricant circulation rates less than 1% are typical.

#### INFORMATIVE ANNEX D AN UNCERTAINTY ANALYSIS EXAMPLE FOR LUBRICANT CIRCULATION RATE ≥1%

The lubricant circulation rate is to be determined for a compressor being rated per ASHRAE Standard  $23.1^{A-2}$  with a desired uncertainty of less than 0.1%. Liquid line samples will be taken at a steady state condition on a large R410A system with 30°C (86°F) liquid temperature using 500 mL (0.132 gal) single-inlet sampling cylinders. It is expected that the circulation rate will be over 1%. Determine the expected measurement uncertainty in the lubricant circulation rate.

Lubricant circulation rate is a derived measurement consisting of single sampled data for each liquid sample that is taken. The empty cylinder mass,  $M_1$ , is a stationary measurement, as the mass of the cylinder does not change between uses if it is completely cleaned before each use. Total cylinder mass,  $M_2$ , and cylinder plus remaining lubricant mass,  $M_3$ , are also stationary measurements once the sample is taken; these masses will not change during subsequent measurements. Based on this, the propagation of errors may be performed as shown in Section A.4.1 of ASHRAE Guideline 2.<sup>3</sup>

The following sections show an example step-by-step analysis as outlined in Section 5.1 of ASHRAE Guideline 2.

# D1. IDENTIFY EXPERIMENTAL GOALS AND ACCEPTABLE ACCURACY

The goal is to determine an estimate of uncertainty in determining a lubricant circulation rate on an existing refrigeration system. Three individual samples will be taken and averaged for the final reported value. Desired uncertainty for the lubricant rate is to be less than 0.1%.

### D2. IDENTIFY THE IMPORTANT VARIABLES AND APPROPRIATE RELATIONSHIPS

Three masses are used in the calculations when the lubricant circulation rate on the sample basis is >1% per Equation D-1 (Equation 2 in Section 9):

$$C_{S} = \frac{M_{3} - M_{1}}{M_{2} - M_{1}} \tag{D-1}$$

#### D3. ESTABLISH THE QUANTITIES THAT MUST BE MEASURED AND THEIR EXPECTED RANGE OF VARIATION

The estimated masses are as follows:

- $M_1$  = Empty cylinder mass ~ 675 g (10,416.8 grains)
- $M_2$  = Cylinder + lubricant + refrigerant mass ~ 1085 g (16,744.1 grains)
- $M_3$  = Cylinder + residual lubricant mass ~ 680 g (10,494.0 grains)

Additionally, the sample size must be checked to verify that the sampling cylinder will not be overfilled, which could cause unexpected venting of the sample, which would invalidate the measurement and create a safety problem if the relief venting device fails to relieve. The sample cylinder is one that is good for CO<sub>2</sub> service, so the pressure rating is well above what is expected for the R410A used in this example. Table D-1 summarizes the maximum fill capacity for a 500 mL (0.132 gal) cylinder at the maximum cylinder temperature of 60°C (140°F) and the actual test temperature of 30°C (86°F). If the sample will weigh approximately 410 g (6327.3 grains), care must be taken to make sure that the cylinder is not warmed up prior to removing the refrigerant. This should not occur as the testing is being performed in a temperature controlled area that is maintained at 22°C (71.6°F). Uncertainty can be minimized using the largest refrigerant and lubricant sample that can be safely contained in the sampling cylinder.

#### D4. TENTATIVELY SELECT SENSORS/ INSTRUMENTATION APPROPRIATE FOR THE TASK

The masses will be recorded using a digital analytical scale with at least a 0.2 g (3.0865 grains) minimum resolution, as shown in Equation D-2 (Equation 1 in Section 6).

Minimum Resolution =

$$\left(\frac{0.1 \ [\%]}{100}\right)\left(\frac{413.1 \ \text{g} \ [6375.1 \ \text{grains}]}{2}\right) = 0.2 \ \text{g} \ [3.086 \ \text{grains}]$$
(D-2)

#### D5. DOCUMENT UNCERTAINTY OF EACH MEASURED VARIABLE

This analytical scale was calibrated within the last six months, which is the manufacturer's suggested calibration cycle. The latest calibration data is shown in Table D-2. The calibration was performed by an ISO  $17025^{A-4}$  certified testing lab using secondary traceable NIST standard masses that are certified to be accurate to within 0.001% of the true mass. Per the manufacturer, the uncertainty of this scale is 0.025% of the reading.

Items to note from the data in Table D-2:

- a. The analytical scale is within the manufacturer's specifications.
- b. The scale being used has a resolution of 0.1 g (1.543 grains), which will yield a minimum resolution of 0.015% for the  $M_1$  and  $M_3$  measurements (= 0.1 g [1.543 grains]/ 680 g [10,494 grains]).
- c. No hysteresis of the scale was measured during the calibration.
- d. The calibration uncertainty for the  $M_1$  and  $M_3$  measurements will be 0.028% if the 0.3 g (4.63 grains) calibration uncertainty is not accounted for (= 0.3 g [4.63 grains]/ 1085 g [16,744.1 grains]).

Additionally, the tap point for drawing these samples is initially planned to include a trapped volume that is a section of piping that is approximately 6.35 mm (0.25 in.) diameter and 50.8 mm (2.00 in.) long. This estimated trapped mass may have fluid that is not homogeneous with that of the main flow. The total trapped mass is calculated to be 1.66 g (25.6 grains) as shown in Table D-3.

### D6. PERFORM A PRELIMINARY UNCERTAINTY ANALYSIS

#### TABLE D-1 Maximum Sample Size Calculation

Cylinder Volume, mL (gal)	R410A Density @ 60°C, kg/m <sup>3</sup> (140°F, lbm/ft <sup>3</sup> )	100% Full Cylinder, g (grains)	80% Full Cylinder, g (grains)
500 (0.132)	815.48 (50.91)	407.8 (6293.3)	326.2 (5034)
	R410A Density @ 30°C (86°F, lbm/ft <sup>3</sup> )		
500 (0.132)	1032.71 (64.47)	516.4 (7969.3)	413.1 (6375.1)

#### TABLE D-2 Digital Scale Calibration Record (Example Scale Measured in Grams Only)

Scale Info:	M/N: C01276	S/N: 12987651
Capacity: 5100	<b>Division Size: 0.1</b>	Units of Measure: grams
Scale Class*: II		

\*Note: Weighing device accuracy class as determined from NIST Handbook 44, A-5 Table 3. Unless otherwise specified, scale tolerances are per NIST Handbook 44, Table 6.

Mass, g	As Found, g	Equipment Tolerance, g	As Left, g	Estimated Uncertainty, g
500	499.8	499.8 to 500.2	499.8	0.125
1000	999.8	999.7 to 1000.3	999.8	0.25
2000	1999.7	1999.5 to 2000.5	1999.7	0.50
4000	3999.9	3999.0 to 4001.0	3999.9	1.00

**TABLE D-3 Trapped Sampling Volume** 

Pipe Diameter, mm (in.)	Pipe Length, mm (in.)	Volume, mm <sup>3</sup> (in. <sup>3</sup> )	Density, kg/m <sup>3</sup> (grains/in. <sup>3</sup> )	Mass, g (grains)
6.35	50.8	1608.8	1032.7	1.66
(0.25)	(2.00)	(0.0982)	(261.2)	(25.6)

Since these three data points are stationary, the propagation of error may be made as is shown in Section A.4.1 of Guideline 2. This will yield the following relationships:

$$C_{S} = \frac{M_{3} - M_{1}}{M_{2} - M_{1}} \tag{D-3}$$

Substituting  $X_1 = M_3 - M_1$  and  $X_2 = M_2 - M_1$  leads to the following:

$$C_{S} = \frac{(X_{1} \pm S_{X1})}{X_{2} \pm S_{X2}}$$
(D-4)

and

$$S_{Xs} = \frac{X_1}{X_2} \sqrt{\left(\frac{S_{X1}}{X_1}\right)^2 + \left(\frac{S_{X2}}{X_2}\right)^2}$$
 (D-5)

Obtaining the uncertainty Equation A-11 from the Section 4 of ASHRAE Guideline 2,

$$S_{X1} = \sqrt{S_{M3}^2 + S_{M1}^2}, \qquad S_{X2} = \sqrt{S_{M2}^2 + S_{M1}^2}$$
 (D-6)

Per Equation A-9 of Guideline 2 where

$$S_X = \frac{M_3 - M_1}{M_2 - M_1} \sqrt{\left(\frac{\sqrt{S_{M3}^2 + S_{M1}^2}}{M_3 - M_1}\right)^2 + \left(\frac{\sqrt{S_{M2}^2 + S_{M1}^2}}{M_2 - M_1}\right)^2} \quad (D-7)$$

Expanding Equation D-7,

$$S_{\chi} = \frac{M_3 - M_1}{M_2 - M_1} \sqrt{\frac{S_{W3}^2 + S_{W1}^2}{(M_3 - M_1)^2} + \frac{S_{W2}^2 + S_{W1}^2}{(M_2 - M_1)^2}}$$
(D-8)

Substituting the estimated numbers into the previous equations:

SI

$$C_s = \frac{M_3 - M_1}{M_2 - M_1} = \frac{(680 - 675)}{(1085 - 675)} = \frac{5}{410} = 1.22\%$$
 (D-9)

I-P

$$C_{s} = \frac{M_{3} - M_{1}}{M_{2} - M_{1}} = \frac{(10,494 - 10,416.8)}{(16,744.1 - 10,416.8)} = \frac{77.2}{6327.3} = 1.22\%$$

The component uncertainties are now estimated.

 $S_{M1}$  standard uncertainty is assumed to be made up of the analytical scale uncertainty and resolution of the scale:

$$S_{M1} = \sqrt{(\text{Scale Uncertainty})^2 + (\text{Resolution})^2}$$
  
=  $\sqrt{(0.025\% \times 675)^2 + 0.1^2}$  (D-10)

I-P

$$S_{M1} = \sqrt{(\text{Scale Uncertainty})^2 + (\text{Resolution})^2}$$
  
=  $\sqrt{(0.025\% \times 10,416.8)^2 + 1.54^2}$ 

so it follows that

SI

$$S_{M1} = \sqrt{0.0285 + 0.01} = 0.196 \text{ g}$$
 (D-11)

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TAI	BLE	D-4	Experimental	Result	Summar	v
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Mass	Sample #1, g (grains)	Sample #2, g (grains)	Sample #3, g (grains)
Mi	674.3 (10,406)	670.5 (10,347.4)	666.5 (10,285.7)
<i>M</i> <sub>2</sub>	1083.9 (16,727.1)	1079.3 (16,656.1)	1075.7 (16,600.6)
<i>M</i> <sub>3</sub>	679.5 (10,486.3)	675.4 (10,423)	671.3 (10,359.7)
Lubricant concentration on a sample basis, $C_S$	0.0127	0.0120	0.0117
Lubricant circulation rate, %	1.27	1.20	1.17
Arithmetic mean of lubricant concentration		0.0121	
Arithmetic mean lubricant circulation rate,%		$1.21 \pm 0.0500$	

I-P

$$S_{M1} = \sqrt{6.782 + 2.372} = 3.0255$$
 grains

 $S_{W2}$  standard uncertainty is assumed to have the same components as  $S_{M1}$ :

SI

$$S_{M2} = \sqrt{(\text{Scale Uncertainty})^2 + (\text{Resolution})^2}$$
  
=  $\sqrt{(0.025\% \times 1085)^2 + 0.1^2}$  (D-12)

I-P

$$S_{M2} = \sqrt{(\text{Scale Uncertainty})^2 + (\text{Resolution})^2}$$
  
=  $\sqrt{(0.025\% \times 16,744.1)^2 + 1.54^2}$ 

so it follows that

SI

$$S_{M2} = \sqrt{0.0736 + 0.01} = 0.289 \text{ g}$$
 (D-13)

I-P

 $S_{M2} = \sqrt{17.523 + 2.372} = 4.46$  grains

 $S_{M3}$  is assumed to have the same basic components as  $S_{M1}$  and  $S_{M2}$ . Additionally, this term will contain any sampling error that may exist if a nonhomogeneous sample is taken. In this case, it is assumed that the mass calculated in Table D-3 is a 50% rich refrigerant and lubricant mixture.

$$S_{M3} = \sqrt{\frac{(\text{Scale Uncertainty})^2 + (\text{Resolution})^2}{+ (\text{Sample Error})^2}} \quad (D-14)$$

so it follows that

SI

$$S_{M3} = \sqrt{(0.025\% \times 680)^2 + 0.1^2 + (1.66 \times 0.5)^2}$$
 (D-15)

I-P

$$S_{M3} = \sqrt{(0.025\% \times 10,494)^2 + 1.54^2 + (25.6 \times 0.5)^2}$$

and

$$S_{M3} = \sqrt{0.0289 + 0.01 + 0.6901} = 0.854 \text{ g}$$
 (D-16)

I-P

$$S_{M3} = \sqrt{6.883 + 2.37 + 164.35} = 13.176$$
 grains

Combining these terms into the uncertainty equation,

$$\frac{S_C}{C_S} = \sqrt{\frac{S_{M3}^2 + S_{M1}^2}{(M_3 - M_1)^2} + \frac{S_{M2}^2 + S_{M1}^2}{(M_2 - M_1)^2}}$$
(D-17)

$$\frac{S_C}{C_S} = \sqrt{\frac{0.854^2 + 0.196^2}{(5^2)} + \frac{0.289^2 + 0.196^2}{(410^2)}}$$
(D-18)

I-P

$$\frac{S_C}{C_S} = \sqrt{\frac{13.176^2 + 3.0271^2}{(77.2^2)} + \frac{4.46^2 + 3.0271^2}{(6327.3^2)}}$$

SI

$$\frac{S_C}{C_S} = \sqrt{\frac{0.729 + 0.038}{25} + \frac{0.084 + 0.038}{168,100}}$$
(D-19)

I-P

$$\frac{S_C}{C_S} = \sqrt{\frac{173.6 + 9.136}{5953.9} + \frac{19.89 + 9.163}{40,034,195}}$$

$$\frac{S_C}{C_S} = \sqrt{\frac{0.767}{25} + \frac{0.122}{168,100}}$$
(D-20)

I-P

SI

$$\frac{S_C}{C_S} = \sqrt{\frac{182.77}{5953.9} + \frac{29.058}{40,034,195}}$$
$$\frac{S_C}{C_S} = \sqrt{0.0307 + 7.261E - 07} = \pm 0.175 \qquad (D-21)$$

The overall lubricant circulation measurement is estimated to be  $1.22\% \pm 0.175\%$  at a 95% confidence level.

#### D7. STUDY UNCERTAINTY RESULTS AND REASSESS THE ABILITY OF THE MEASUREMENT METHODS AND INSTRUMENTATION TO MEET ACCEPTABLE ACCURACY

Since the initial goal was to measure lubricant circulation rate to within 0.1% of the actual value, this system will not meet that goal, as the initial estimated uncertainty was 0.175%. The

#### POLICY STATEMENT DEFINING ASHRAE'S CONCERN FOR THE ENVIRONMENTAL IMPACT OF ITS ACTIVITIES

ASHRAE is concerned with the impact of its members' activities on both the indoor and outdoor environment. ASHRAE's members will strive to minimize any possible deleterious effect on the indoor and outdoor environment of the systems and components in their responsibility while maximizing the beneficial effects these systems provide, consistent with accepted Standards and the practical state of the art.

ASHRAE's short-range goal is to ensure that the systems and components within its scope do not impact the indoor and outdoor environment to a greater extent than specified by the Standards and Guidelines as established by itself and other responsible bodies.

As an ongoing goal, ASHRAE will, through its Standards Committee and extensive Technical Committee structure, continue to generate up-to-date Standards and Guidelines where appropriate and adopt, recommend, and promote those new and revised Standards developed by other responsible organizations.

Through its Handbook, appropriate chapters will contain up-to-date Standards and design considerations as the material is systematically revised.

ASHRAE will take the lead with respect to dissemination of environmental information of its primary interest and will seek out and disseminate information from other responsible organizations that is pertinent, as guides to updating Standards and Guidelines.

The effects of the design and selection of equipment and systems will be considered within the scope of the system's intended use and expected misuse. The disposal of hazardous materials, if any, will also be considered.

ASHRAE's primary concern for environmental impact will be at the site where equipment within ASHRAE's scope operates. However, energy source selection and the possible environmental impact due to the energy source and energy transportation will be considered where possible. Recommendations concerning energy source selection should be made by its members.



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