

Appendix C to AHRI Standard 700-2014 (formerly Appendix C to AHRI Standard 700-2012)

2008 Appendix C for Analytical Procedures For AHRI Standard 700-2014 – Normative



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FOREWORD

The intent of this normative appendix is to establish definitive test procedures for determining the quality of new, reclaimed and/or repackaged refrigerants for use in new and existing equipment within the scope of AHRI.

These test methods are established as referee test methods. If alternate test methods are employed, the user must be able to demonstrate that the results of these procedures are equivalent to the specified referee test methods (see Section 7 of each test method for information concerning the sensitivity, precision and accuracy of that test method).

Note:

This Appendix supersedes Appendix C-1999 to ARI Standard 700.

TABLE OF CONTENTS

PARTS	PAGE
Part 1	Determination of Acidity in New and Reclaimed Refrigerants by Titration 1
Part 2	Determination of Water in New and Reclaimed Refrigerants by Karl Fischer Coulometric Titration 5
Part 3	Determination of High Boiling Residue in New and Reclaimed Refrigerants by Volumetric and/or Gravimetric Measurement and Determination of Particulate Residue by Visual Indication 11
Part 4	Determination of Chloride in New and Reclaimed Refrigerants by Silver Chloride Precipitation 16
Part 5	Determination of Non-Condensable Gas in New and Reclaimed Refrigerants by Gas Chromatography 19
Part 6	Determination of Purity of New and Reclaimed R-11 by Gas Chromatography 25
Part 7	Determination of Purity of New and Reclaimed R-12, R-13, R-22, R-23, R-114, R-115, R-116, R-124, R-125, R-143a, R-152a, R-218, R-290, R-600, and R-600a by Packed Column Gas Chromatography 32
Part 8	Determination of Purity of New and Reclaimed R-123 by Capillary and Packed Column Gas Chromatography 41
Part 9	Determination of Purity of New and Reclaimed R-22, R-32, R-113, R-134a, R-141b, R-142b, and R-245fa by Capillary Column Gas Chromatography 50
Part 10	Determination of Composition of New and Reclaimed 400 Series and 500 Series Refrigerant Blends by Gas Chromatography 62

TABLES

PARTS		PAGE
Part 2	Table C2-1 Single Operator Method Precision.....	9
	Table C2-2 Testing for Percent Water Recovery	10
Part 3	Table C3-1 Densities of Some Common Liquid Refrigerants at 25°C [77 °F]	15
Part 5	Table C5-1 K _i Values for Refrigerants at 24.0°C [75.2°F].....	23
Part 6	Table C6-1 Retention Time Data for Identified Impurities Not Normally Observed.....	29
	Table C6-2 Component Statistical Parameters	29
	Table C6-3 Primary Calibration Standard Components	30
Part 7	Table C7-1 GC Operating Conditions... ..	34
	Table C7-2 Component Statistical Parameters	38
	Table C7-3 Primary Calibration Standard Components	39
Part 8	Table C8-1 Component Statistical Parameters.....	46
	Table C8-2 Additional Impurities Observed in R-123, Quantitation by Effective Carbon Number Method.....	47
	Table C8-3 Primary Calibration Standard Components	48
Part 9	Table C9-1 GC Operating Conditions.....	52
	Table C9-2 Additional Impurities Observed in R-134a, Quantitation by Effective Carbon Number Method.....	58
	Table C9-3 Component Statistical Parameters	59
	Table C9-4 Primary Calibration Standard Components	60
	Table C9-5 Primary Calibration Standard Liquid Impurities	60

FIGURES

PARTS		PAGE
Part 2	Figure C2-1 Needle Attachment Assembly for Cylinder Sampling	7
	Figure C2-2 Example of Moisture Analyzer Report Print-out	10
Part 5	Figure C5-1 Evacuated System Method of Introducing Vapor Sample into Gas Chromatograph	24
Part 6	Figure C6-1 Apparatus Used for Calibration Standard Preparation.....	31
Part 7	Figure C7-1 Apparatus Used for Calibration Standard Preparation And for Cylinder Sampling	40
Part 8	Figure C8-1 Apparatus Used for Calibration Standard Preparation.....	49
Part 9	Figure C9-1 Apparatus Used for Calibration Standard Preparation And for Cylinder Sampling.....	61
Part 10	Figure C10-1 Apparatus Used for Calibration Standard Preparation And for Cylinder Sampling.....	68

PART 1

DETERMINATION OF ACIDITY IN NEW AND RECLAIMED REFRIGERANTS BY TITRATION

Section C1-1. Purpose

The purpose of this test method is to determine the amount of acidity in new and reclaimed refrigerants.

Section C1-2. Scope

This test method is for use with low, medium and high pressure fluorocarbon refrigerants.

Section C1-3. Definitions

Definitions for this part are identical to those of ARI Standards 700 and 740.

Section C1-4. Principle

A known quantity of a liquid refrigerant sample is added to, or bubbled through, an extraction solvent that is a mixture of toluene, isopropanol and water to which bromothymol blue indicator has been added. The acidity imparted to the extraction solvent by the sample quantity is titrated with standardized potassium hydroxide to the indicator endpoint. The acidity is reported in ppm as HCl.

Section C1-5. Applicability

This method is applicable to the routine quantitative determination of acidity in low, medium and high pressure refrigerants.

Section C1-6. Limitations and Interferences

None of the refrigerants tested interfere with the acidity determination. The test must be performed quickly after the indicator solution is brought to its blue/green end point to avoid interferences from atmospheric carbon dioxide.

Section C1-7. Sensitivity, Precision and Accuracy

7.1 *Sensitivity.* The sensitivity of the acidity test using 50 g of sample in 100 g of extraction solvent is 0.1 ppm. Care must be taken in sample handling and in avoiding cross contamination when performing this test.

7.2 *Precision.* (Data is not available)

7.3 *Accuracy.* (Data is not available)

Section C1-8. Special Apparatus and Reagents

1. Capillary tubing 1/16 inch x 0.007 inch tetrafluoroethylene
2. Top loading balance, 1000 g with 0.1 g resolution
3. 100 mL stainless steel double-ended, 1/4 inch FNPT cylinder (1800 psig)
4. Two 1/4 inch stainless steel valves with MNPT fittings
5. Two 1/4 inch FNPT x 1/4 inch flare fittings
6. 1/16 inch x 1/4 inch stainless steel tube compression fitting reducing union
7. 1/4 inch compression x 1/4 inch flare AN female adaptor
8. 1/4 inch x 1/4 inch copper flare connector
9. 1/4 inch inlet MNPT x 1/4 inch outlet FNPT pressure relief valve (350 to 400) psig
10. 250 mL Erlenmeyer flask
11. Bromothymol blue sodium salt endpoint indicator
12. Reagent grade isopropanol
13. Reagent grade toluene
14. 0.1 N Potassium hydroxide in methanol
15. 0.1 N Sulfuric acid
16. Absolute methanol (Anhydrous, reagent grade)
17. Stir plate/stir bar
18. Glass distilled water
19. Buret (10 mL with 0.05 mL graduation)

Section C1-9. Procedure

9.1 *Capillary Tubing Connector.* Take 1/16 inch x 0.007 inch tetrafluoroethylene tubing and "swage" on a 1/16 inch nut and ferrule. Connect this to a 1/16 inch x 1/4 inch compression fitting reducing union and then connect it to the 1/4 inch compression fitting x 1/4 inch flare adaptor. The 1/4 inch flare adaptor can then be connected to the 1/4 inch flare fitting on the cylinder assembly just before each acidity determination.

9.2 *Cylinder Assembly.* The cylinder assembly is used as the sampling apparatus for each acidity determination. Before assembly, all pipe fittings must be taped with tetrafluoroethylene to ensure a proper seal at each joint.

Attach the pressure relief valve to the 100 mL stainless steel cylinder. Attach one of the 1/4 inch MNPT x 1/4 inch MNPT stainless steel valves to the pressure relief valve. Connect a 1/4 inch FNPT x 1/4 inch flare fitting to the 1/4 inch MNPT valve. To the other side of the 100 mL cylinder, attach another 1/4 inch

MNPT x 1/4 inch MNPT valve. Sampling should occur from the side of the 100 mL cylinder that does not have the pressure relief valve.

9.3 *Reagent Preparation.*

9.3.1 *0.01 N KOH Solution.* Pipet 100 mL of 0.1 N KOH solution into a 1000 mL volumetric flask. Dilute to the mark with absolute methanol and mix thoroughly.

9.3.2 *0.01 N Sulfuric Acid Solution.* Pipet 100 mL of 0.1 N sulfuric acid solution into a 1000 mL volumetric flask. Dilute to the mark with distilled water and mix thoroughly.

9.3.3 *Extraction Solvent.* Add 495 mL of toluene to 495 mL of isopropanol. Add 10 mL of water to the toluene/isopropanol solution and mix thoroughly.

9.3.4 *Bromothymol Blue Indicator Solution.* Dissolve 1 g of bromothymol blue sodium salt in 100 mL of methanol. Mix thoroughly and store in a dropper bottle.

9.4 *Sample Analysis.*

9.4.1 Thoroughly clean the 100 mL stainless steel cylinder, the valve, the capillary tube, the copper connector and the 250 mL Erlenmeyer flask before initiating testing. Oven heat all of the components to 110°C [230°F] and pull a vacuum.

9.4.2 Weigh the cylinder assembly to the nearest 0.1 g and designate this weight as "X."

9.4.3 Attach the 1/4 inch copper fitting to the vapor valve of the sample cylinder and to the cylinder assembly. Open the vapor valve; loosen the connector and quickly tighten the fitting. This will purge the 1/4 inch connector of air.

9.4.4 Invert the sample cylinder with the attached cylinder assembly. Open the sample cylinder valve and then the cylinder assembly valve. Allow the refrigerant to be introduced into the cylinder assembly until 50 g to 75 g of refrigerant is sampled.

9.4.5 Close the cylinder assembly valve and set the sample cylinder upright. Close the sample cylinder valve, loosen the 1/4 inch connector and remove the cylinder assembly.

9.4.6 Reweigh the cylinder assembly with the refrigerant and designate this value as "Y." The weight of the refrigerant is given by $Y - X$ = grams of refrigerant sampled. (Value for X is in 9.4.2 above).

Note: For the low pressure refrigerants, weigh the sample cylinder before sampling and then add the refrigerant directly to the extraction solvent. Reweigh the sample cylinder and subtract the initial weight from the final weight to obtain the total weight of the refrigerant sampled.

9.4.7 Add 100 mL of the prepared extraction solvent to a 250 mL Erlenmeyer flask. Add a clean magnetic stirring bar. Add 6 drops of the indicator solution to the extraction solvent and initiate moderate stirring.

9.4.8 If the extraction solvent/indicator solution is yellow, add 0.01 N potassium hydroxide through the buret until a just noticeable difference of blue/green is seen in the extraction solvent. Half drops from the buret may be necessary to achieve the real end point.

9.4.9 If the extraction solvent/indicator solution is green or blue, add 0.01 N sulfuric acid through the buret dropwise until the solution is yellow and then proceed as in 9.4.8 immediately above.

9.4.10 Attach the cleaned capillary connector to the cylinder assembly containing the refrigerant sample and slowly introduce the entire sample into the extraction solvent/indicator solution by gradually opening the cylinder valve until it is fully open. The cylinder assembly should be clamped to a ring stand throughout the procedure.

9.4.11 Add all of the refrigerant sample to the extraction solvent. If the color of the solution is green or blue, the result is reported as "non-detect." If the solution is yellow, record the buret volume to the nearest 0.01 mL (designate this value as V_1) and add 0.01 N KOH dropwise until the green endpoint is reached. Record the final buret volume to the nearest 0.01 mL (designate this value as V_2).

Final volume (designate this value as V_f) of 0.01 N KOH added:

$$V_f = V_2 - V_1 \quad \text{C1.1}$$

9.4.12 Calculation of total acidity expressed in ppm as HCl is given by:

$$\text{ppm as HCl} = \frac{V_f \cdot \text{normality KOH} \cdot 36,500}{\text{refrigerant weight sampled}} \quad \text{C1.2}$$

Note: The value 36,500 is the equivalent weight of HCl (36.5×10^3).

PART 2

DETERMINATION OF WATER IN NEW AND RECLAIMED REFRIGERANTS BY KARL FISCHER COULOMETRIC TITRATION

Section C2-1. Purpose

The purpose of this test method is to determine moisture in new and reclaimed refrigerants by the Karl Fischer coulometric titration method.

Section C2-2. Scope

This test method is for use with low, medium and high pressure refrigerants.

Section C2-3. Definitions

Definitions for this part are identical to those of ARI Standards 700 and 740.

Section C2-4. Principle

Karl Fischer (KF) titrimetry is based upon the redox reaction of water, iodine and sulfur dioxide:



The solvent is typically a mixture of methanol and a weak organic base (imidazole, pyridine, etc.) with the base serving to neutralize the reaction products. In coulometric KF titrimetry, iodine is generated at the anode in direct proportion to the amount of water introduced, and the end point is detected bi-amperometrically as the first appearance of excess free I_2 . The added refrigerant eventually evaporates; hence, the solvent can be used repeatedly until either the SO_2 or the base solution is consumed.

Section C2-5. Applicability

This method is applicable to the routine quantitative determination of small amounts of water in low, medium, and high pressure refrigerants.

Section C2-6. Limitations and Interferences

None of the refrigerants tested interfere with the titration. Oxidizing agents such as MnO_4^- , $\text{Cr}_2\text{O}_7^{2-}$, H_2O_2 , Fe (III), Cu (II) and reducing agents such as S^{2-} , thiosulphates and Sn (II) will interfere. Also, certain compounds such as basic oxides and salts of weak acids (NaHCO_3 , for example) can form water with the KF reagent. None of these interferences are normally present in new or reclaimed refrigerants.

Section C2-7. Sensitivity, Precision and Accuracy

7.1 Sensitivity. The sensitivity of the analyzer in this method using a 10 g sample is 1 ppm. Extreme care must be used in sample handling in order to achieve this sensitivity.

7.2 Precision and Accuracy. The mean of the analysis ($\bar{X}$), standard deviation (σ) and 95% confidence limits (CL) established for the single operator precision of this method are shown in Table C2-1.

The data in Table C2-1 were calculated from 17 replicate analyses of one sample (approximately 10 g) performed by one analyst over a period of two days.

The samples in Table C2-2 were tested for total percent recovery. They were prepared by analyzing R-12 and R-22 to 4.8 ppm and 7.1 ppm, respectively, and then contaminating the refrigerants with known amounts of water. Both samples were then mixed for a period of 24 hours before analyzing. Results are shown in Table C2-2.

The total percent recovery for each sample was 99.3% for R-12 and 99.7% recovery for R-22.

Section C2-8. Special Apparatus and Reagents

1. KF coulometric titrator system (contains a drying tube for venting refrigerant, anode and cathode solutions, septum, and water vaporizer)
2. Drierite, 20-40 mesh
3. Desiccator, containing Drierite
4. Refrigerant sample cylinder, e.g. 50 mL, 500 mL, 1000 mL stainless steel double ended 1/4 inch FNPT cylinders (1800 psig), steel cylinder, 2.2 lb, single 9 gauge valve, 3/8 inch pipeneck, disposable can, 17 oz, or other suitable cylinder
5. Stainless steel integral bonnet non-rotating stem valve, 1/4 inch MNPT x 1/4 inch FNPT
6. Brass screwed-bonnet needle valve, 1/4 inch MNPT
7. Male Luer lock 10-32 standard thread needle connector, Cut threads back 1/8 inch (threads are too long as received)
8. Needle, 19 gage Luer lock, 4-1/2 inch length
9. 1/4 inch compression fitting to 1/4 inch AN female flare adaptor
10. Quick Seal Flare Cap, No. NFT5-4, 1/4 inch tubular seal gasket
11. Sample Injection Needle and Valve Attachment (see Figure C2-1). Remove the inner gasket-then drill and tap for a 10/32 inch standard thread through the center of the flare cap. Coat the threads with epoxy-then screw the needle connector (item No. 7) into the hole until snug, then allow the epoxy to set overnight. The needle is attached to the connector and the assembly then screwed onto the needle valve AN female flare adapter
12. Syringe, 10 mL, gas tight
13. Syringe needle, 19 gage-4 inch (deflected point)

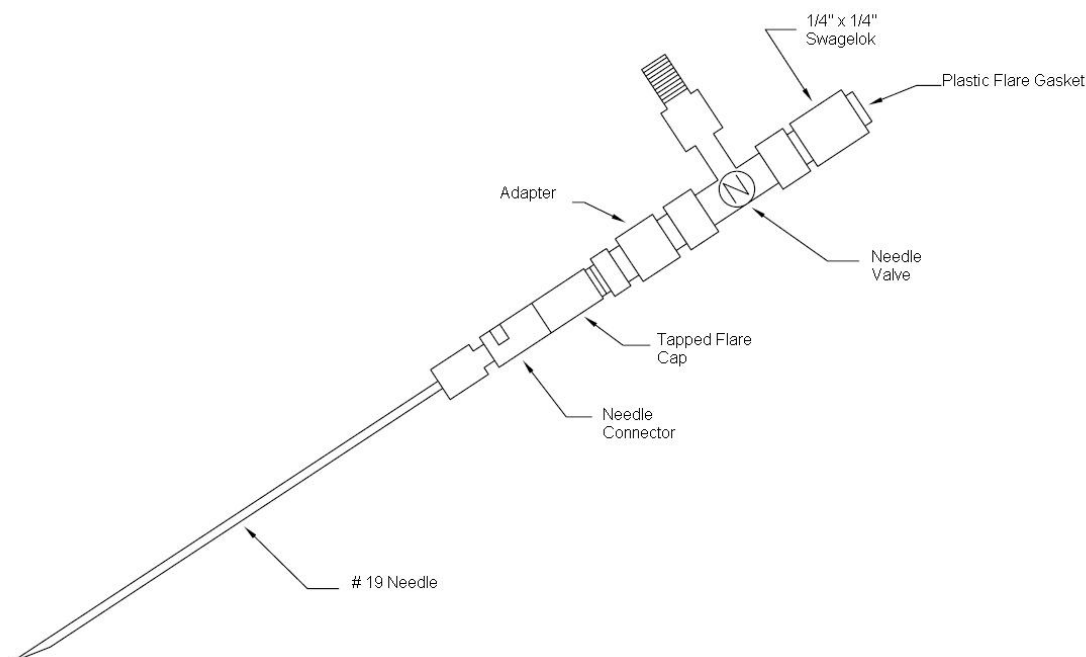


Figure C2-1. Needle Attachment Assembly for Cylinder Sampling

Section C2-9. Procedure

9.1 Verification. Verify that the instrument is operating accurately by injecting quantified moisture standard prior to sample testing.

9.2 Sample Analysis.

Note: To minimize contamination from moisture, the sample should be introduced directly from the refrigerant sample cylinder into the coulometric titrator, i.e., avoid a secondary container transfer, whenever possible. Also, the effects of moisture contamination and phase distribution will be minimized if the sample container is 60% to 80% liquid filled with refrigerant. If the sample is a very high pressure refrigerant, cool the cylinder to approximately 14 K [25.0°F] below critical temperature (T_C) of the refrigerant and allow 30 minutes for equilibrium to be established before starting the analysis.

9.2.1 Refer to the instruction manual for moisture analyzer installation and operation. Instrument sensitivity should be set at 0.10 and a new septum should be attached.

9.2.2 Turn-on the analyzer and magnetic stirrer and wait until the background current ($\mu\text{g H}_2\text{O}$ per second) has reached a low, steady level. It may be necessary to "shake" the titration vessel to contact (wash down) any water mist on the upper inside walls with the anode solution. Optimum levels are below 0.10 microgram of water per second (normally 0.02 to 0.05).

Note: If after 15 minutes a low ($< 0.1 \mu\text{g}$ per second) background current is not obtained or if the cathode solution turns a dark reddish brown color, turn off the moisture meter and, using a small funnel, renew both anode and cathode solutions. Also, should a negative background reading persist (free I_2 in the anode solution), introduce a drop of R-113 or methanol-water wetting solution into the vessel to eliminate the free I_2 via reaction with water and produce a positive background. This wetting solution can be made by adding a small amount of water (typically less than 500 ppm) to methanol or R-113.

9.2.3 Using a heat gun, dry-off the valve threaded end of the sample cylinder valve that contains a pressure relief valve (350 psi to 400 psi) and cylinder stem valve (1/4 inch MNPT x 1/4 inch FNPT).

9.2.4 Remove the Needle/Needle Valve attachment (see Figure C2-1) from the oven or desiccator and immediately attach to the sample cylinder valve.

9.2.5 Open the refrigerant sample cylinder valve, then slowly open the needle valve and purge a small amount of sample liquid phase to flush the air from the needle (1 second to 2 second purge). Close both valves.

9.2.6 Using a heat gun (high position), carefully dry the needle for 20 seconds to 30 seconds.

9.2.7 Weigh the refrigerant sample cylinder plus attachment on a top loader balance (nearest 0.1 g) and record on a work sheet.

9.2.8 Using a clamp (or clamps) and weighted ring stand, invert and position the sample cylinder such that the needle punctures the septum and is immersed to the hub of the needle. The needle should be submerged about one inch below the KF solution surface.

Note: The background current will rise after inserting the needle, then return back to the normal low value.

9.2.9 At this juncture it is assumed that the instrument has been turned on, preset for a 5 minute titration start delay, verified, and that the background current is at a low (0.02 μg to 0.05 μg) value.

Notes:

1. Do not initiate the titration unless and until the background current has stabilized at a low μg value.

2. The coulometric titrator background signal (given as $\mu\text{g H}_2\text{O}$ per second) is subtracted from the analyzed result and represents the background moisture presumably accumulated during the time taken to introduce and to titrate the sample. The background value subtracted is the final value read just before sample addition begins. An artificially elevated background value will result in an erroneously low result (i.e. negatively biased). Hence, it is important that the background value be as small (but correct) as possible. Often, it is necessary to physically "swirl" the titration cell as to rinse moisture accumulated on the inner walls into the KF solution. This operation normally speeds up the process of reaching a low background signal.

9.2.10 Ensure that the desiccant tube is clear of obstructions.

9.2.11 Enter the gross cylinder weight (W_1) from 9.2.7 into the moisture meter, if applicable, or record the initial weight of the cylinder to the nearest 0.1 g on a worksheet.

9.2.12 If applicable, remove any prior number displayed for the second weight.

9.2.13 When the moisture meter is stable (maintains a low background current), initiate a run, slowly open the needle valve and introduce sample at a moderate rate such that no foaming is observed on the KF solution surface. Add at a rate such that 15 g to 20 g of the sample is added over an approximate 10 minute period. Use the sample addition count down (delay) if available.

9.2.14 Normally, a 20 g sample is desirable for best accuracy. Observe the cell potential reading or microgram reading. If during sample addition this number climbs rapidly to a comparatively

large value (range: 200 to 300), this means the sample contains high moisture, and a smaller than normal sample size (5 g to 10 g) is sufficient. .

9.2.15 After the proper sample size has been added, initiate the titration, or after the count down (delay) period ends the coulometer will begin the titration.

Note: If the sample contains high moisture, the rate of titration may never exceed the rate of H₂O addition and the titration must be terminated (closing off the needle valve) before too much sample is added. Conversely, if the sample added is small (4 g to 5 g) and the moisture level also is small (5 ppm to 10 ppm), to achieve better accuracy, the sample should be reanalyzed using a longer sample addition delay (10 minutes for example).

9.2.16 Remove and reweigh the sample cylinder/assembly to the nearest 0.1 g (W₂).

Note: If a small sample size is used, a more accurate balance is recommended and weights should be recorded to the nearest 0.01 g.

9.2.17 Enter the weight from 9.2.16 into the moisture meter if applicable, or record the final weight of the cylinder to the nearest 0.1 g on a worksheet.

9.2.18 Calculate and print-out the ppm or microgram water result. See Figure C2-2 for a sample print-out.

9.2.19 Calculation.

$$\text{Moisture Concentration, ppm} = \frac{\text{micrograms of H}_2\text{O}}{\text{grams of sample (W}_1 - \text{W}_2)} \quad \text{C2.2}$$

Report all results to the nearest 1 ppm. If results are < 2 ppm, report < 2 ppm.

Note: Experience has demonstrated that erratic and out-of-specification moisture results are almost always the result of poor and/or improper sampling. Also, be advised that moisture contamination occurs more readily when the relative humidity is high and particular care is required during these times.

Table C2-1. Single Operator Method Precision

	Mean ($\bar{X}$)	Standard Deviation (σ)	95% Confidence Limit
Water (R-12), ppm by weight	10.6	0.11	0.26
Water (R-22), ppm by weight	28.1	0.29	0.77

Table C2-2. Testing for Percent Water Recovery					
	Original Value	Amount Contaminated	Calculated Total	Recovered Total	Percent Recovery
Water (R-12), ppm by weight	6.8	6.9	13.7	13.6	99.3
Water (R-22), ppm by weight	19.2	19.0	38.2	38.1	99.7

No. : 1-14 F=1
CONC: 4.02 ppm
FNo1: (M-B) / (W-w)
M: 83.6 µg
B.G. : 0.08 µg/S
TIME: 5
SENS: 0.10
VA-T: 100
VA-P:
PRNT: 3
CALC: 1
IDNo: 1-14
W: 503.5
W: 482.7
W-w: 20.800000 g
B: 0
TIME: 1:43

Figure C2-2. Example of Moisture Analyzer Report Print-Out

PART 3

DETERMINATION OF HIGH BOILING RESIDUE IN NEW AND RECLAIMED REFRIGERANTS BY VOLUMETRIC AND/OR GRAVIMETRIC MEASUREMENT AND DETERMINATION OF PARTICULATE RESIDUE BY VISUAL INDICATION

Section C3-1. Purpose

The purpose of this test method is to determine high boiling residue and visible particulates in new and reclaimed refrigerants.

Section C3-2. Scope

This test method is for use with low, medium and high pressure refrigerants.

Section C3-3. Definitions

Definitions for this part are identical to those of ARI Standards 700 and 740.

Section C3-4. Principle

High boiling residue (HBR), also called non-volatile residue, is determined by evaporating a known amount of refrigerant in a Goetz bulb at an ambient or elevated temperature. The remaining residue is then visually measured or weighed. If greater than specification volume is observed, the bulb is placed in a 60.0°C [140°F] oven for 30 minutes and, after cooling, the volume of residue is again measured. For gravimetric determination, the residue is redissolved in a suitable high purity solvent (e.g. R-141b) and quantitatively transferred into a small, tared aluminum pan. The solvent is removed by evaporation and the pan reweighed to obtain the weight of residue.

Prior to evaporation, the measured volume of liquid refrigerant is visually examined for the presence of insoluble materials such as packing fibers, rust, dirt, etc. The residue from high pressure samples is redissolved in a clean solvent, swirled, and then visually examined for any insoluble particulates.

Section C3-5. Applicability

This method is applicable to the routine quantitative determination of HBR and visible evidence of particulates in all low, medium and high pressure new and reclaimed refrigerants. The method was developed to measure HBR and particulates in compliance with AHRI Standard 700 specifications for HBR and particulates.

Section C3-6. Limitations and Interferences

In order to achieve the statistical parameters stated for this method, at least 100 mL of refrigerant sample is required. There are no known interferences to this method.

Section C3-7. Sensitivity, Precision and Accuracy

7.1 Sensitivity. Based upon a 100 mL volume of sample, the method will detect 0.01 mL of HBR, which is the first mark on the Goetz bulb buret. This 0.01% value is the AHRI Standard 700 specification for most refrigerants. The detection limit by weight is generally $< 0.01\%$ due to the sensitivity of the analytical balance and because 0.01 mL of residue (usually oil) weighs < 0.01 g. Also, except for very high pressure refrigerants, the weight of 100 mL of liquid refrigerant weighs > 100 g.

7.2 Precision. The precision for the HBR determination at 0.03 volume percent was found to be ± 0.005 at the 95% confidence limit. This was based upon an analysis of R-11 by two analysts each of whom used silicone oil as the residue.

7.3 Accuracy. The Relative Mean Error at the 0.03% volume level was found to be 3.3%.

Note: These statistical parameters are not applicable to visual observations of particulates.

Section C3-8. Special Apparatus and Reagents

1. Goetz graduated centrifuge tube: 100 mL
2. Boileezers, carborundum crystal
3. Disposable aluminum dish

Section C3-9. Procedure

9.1 Calibration. For the HBR procedure, a calibration solution of 0.03% by weight of silicone oil in R-11 may be prepared by weighing 0.220 g of silicone oil and dissolving in 500 mL (738 g) of high purity R-11, mixing thoroughly, labeling and storing in a screw-capped glass bottle in a refrigerator. Alternatively, weigh 0.187 g of oil and dissolve in 500 mL of R-141b or other appropriate solvent.

9.2 Sample Analysis, HBR Volume Percent Measurement and Particulates.

9.2.1 Measure 100 mL of refrigerant sample into the Goetz bulb as follows:

9.2.1.1 For low pressure refrigerants (R-11, R-113, R-123): add 100 mL of liquid refrigerant from a glass graduate into the Goetz bulb. Alternatively, add liquid refrigerant from the sample container to the 100 mL mark of the Goetz bulb.

9.2.1.2 While firmly holding the bulb, gently swirl the sample solution and then position the bulb in front of a source light, window, etc. and visually examine for the presence of particulate matter. Record as "pass" if particulates are not observed. Proceed to 9.2.2.

9.2.1.3 For medium and high pressure refrigerants: tare the sample cylinder (to the nearest 0.1 g), invert the cylinder and, by positioning the valve opening just inside the neck of the Goetz bulb, carefully open the valve to allow the liquid phase to discharge inside the bulb. Except for very high pressure refrigerants (R-503, for example), liquid refrigerant will begin to accumulate. Continue to add sample until 60 mL to 75 mL of

liquid has been collected. Turn off the sample valve. Reweigh the sample cylinder and record the difference as the weight of sample added.

9.2.1.4 Repeat 9.2.1.2 above. Use isopropyl alcohol, "thumb," or paper towel to remove frost from the outside of the bulb (create a window) to facilitate the visual observation.

9.2.1.5 For very high pressure refrigerants (R-503, R-13, R-23), the sample cylinder is precooled to approximately 4.4°C [40°F] in ice water (especially on hot days) before carefully flashing the liquid phase into the Goetz bulb. Continue to add liquid phase until the sample cylinder weigh-back shows that between 100 g and 130 g of refrigerant has been flashed into the bulb. At this point, little or no liquid phase refrigerant will have accumulated in the bulb. Record this weight as the "g" of sample added. Add 100 mL of a high purity solvent (e.g. R-141b) to the bulb, put stopper in bulb, swirl to dissolve any residue on the inner walls of the bulb, remove the stopper.

Note: Do not allow any stopcock grease to be present on the glass stopper or on the neck of the bulb.

9.2.1.6 Repeat 9.2.1.2 above.

9.2.2 Add one small boileezer and place the Goetz bulb in a 45.0°C [113°F] constant temperature water bath (60.0°C [140°F] for R-113). Position the bulb such that it is immersed in the bath to about the 20 mL to 25 mL mark. Do not remove the bulb from the bath until all the refrigerant has completely evaporated (determined by observing the disappearance of refrigerant condensation around the neck of the bulb).

9.2.3 Remove the Goetz bulb from the bath, wipe the outside dry and visually measure the mL residue (if any) at the bottom of the buret (ignore the boileezer). Measure to the nearest 0.005 mL.

9.2.4 If the observed residue is ≤ 0.01 mL, proceed to the calculation section below. If the observed residue is > 0.01 mL, proceed to 9.2.5.

9.2.5 Place the Goetz bulb upright in a 60.0°C [140°F] oven for 30 minutes, remove, cool, then measure and record the volume of residue (to the nearest 0.005 mL) in the buret as above. To measure weight percent, save the Goetz bulb-residue for 9.3.

9.2.6 Calculation.

$$\text{HBR Volume \%} = \frac{A \cdot 100}{B} \quad \text{C3.1}$$

where:

A = volume of residue (mL) in buret.

B = mL of sample added to bulb (9.2.1 above).

Note: To calculate the mL of high and very high pressure refrigerant samples, divide the weight of the sample by the liquid density of the refrigerant at the ambient sample temperature (see Table C3-1).

Report all results to the nearest 0.01% volume. If results are $< 0.01\%$, report as " $< 0.01\%$ volume."

9.3 *Sample Analysis, Weight Percent Measurement.*

9.3.1 Prepare an aluminum pan by rinsing it in acetone and placing it in a 60.0°C [140°F] oven for at least 30 minutes. Remove (use tweezers) and place in a desiccator until cool (usually 15 minutes to 20 minutes).

9.3.2 Using tweezers, remove the pan from the desiccator and determine the tare weight (to the nearest 0.0001 g).

9.3.3 Add 20 mL of a high purity solvent (e.g. R-141b) to the Goetz bulb saved from 9.2.5. Stopper the bulb and shake to redissolve the residue and/or to resuspend the particulates (if present) in the solvent.

9.3.4 Carefully pour the solution from the Goetz bulb into the pan. Use two approximately 8 mL portions of the solvent to complete the quantitative transfer of residue. Do not permit the boiler to fall into the aluminum pan; but, if that should occur, carefully remove using metal tweezers.

9.3.5 Carefully place the aluminum pan inside a hood and allow the high purity solvent to evaporate (alternatively, the pan may be placed on the hot water bath).

9.3.6 Place the pan in the 60.0°C [140°F] oven for 30 minutes, remove and place in the desiccator until cool (20 minutes to 30 minutes).

9.3.7 Using tweezers, remove the pan, reweigh and record the difference in weight (from 9.3.2 above) as the weight of residue.

9.3.8 Calculation.

$$\text{HBR weight \%} = \frac{A \cdot 100}{B} \qquad \text{C3.2}$$

where:

A = grams of residue from 9.3.7 above.

B = grams of sample taken from 9.2.1.

Note: To determine the g of a low pressure sample refrigerant (R-11, R-113, R-123, etc.), multiply the volume taken times the density (see Table C3-1).

Report results to the nearest 0.01% weight. If results are <0.01% weight, report as "<0.01% weight."

Table C3-1. Densities of Some Common Liquid Refrigerants at 25°C [77°F]	
Refrigerant	Density (g/mL)
R-11	1.476
R-12	1.311
R-13	0.907
R-13B1	1.538
R-22	1.194
R-32	0.961
R-113	1.565
R-114	1.456
R-115	1.291
R-123	1.468
R-124	1.364
R-125	1.190
R-134a	1.210
R-141b	1.244
R-142b	1.114
R-143a	0.946
R-152a	0.899
R-290	0.492
R-401A	1.188
R-401B	1.188
R-402A	1.151
R-402B	1.156
R-403B	1.150
R-404A	1.167
R-405A	1.173
R-407A	1.142
R-407B	1.166
R-407C	1.134
R-408A	1.062
R-409A	1.223
R-410A	1.031
R-500	1.168
R-502	1.217
R-503	0.795
R-507	1.170

PART 4

DETERMINATION OF CHLORIDE IN NEW AND RECLAIMED REFRIGERANTS BY SILVER CHLORIDE PRECIPITATION

Section C4-1. Purpose

The purpose of this test method is to qualitatively determine the presence of chloride in new and reclaimed refrigerants.

Section C4-2. Scope

This test method is for use with low, medium and high pressure refrigerants.

Section C4-3. Definitions

Definitions for this part are identical to those of ARI Standards 700 and 740.

Section C4-4. Principle

The qualitative determination of chloride in refrigerants is based on precipitation of the chloride anion as silver chloride:



The refrigerant is added to a solution of silver nitrate in methanol. Visual turbidity indicates the presence of chloride and is reported as "fail." If no turbidity is observed, chloride is within acceptable limits and reported as "pass."

Section C4-5. Applicability

This method is applicable to the routine qualitative determination of chloride in low, medium and high pressure refrigerants.

Section C4-6. Limitations and Interferences

None of the refrigerants tested interfere with the chloride determination. Anions of weak acids can be an interference in the determination, but these interferences are not normally present in new or reclaimed refrigerants. Samples containing insoluble lubricants and oils may show a visual haze or slight turbidity, however, such levels of lubricant or oil necessary to show such visual turbidity are not normally present in new or reclaimed refrigerants.

Section C4-7. Sensitivity, Precision and Accuracy

7.1 *Sensitivity.* The sensitivity of the chloride turbidity test using 5 mL of sample in 5 mL of methanol containing three drops of saturated AgNO_3 is approximately 3 ppm. Care must be taken in sample handling to avoid cross contamination when performing this test.

7.2 *Precision.* Data is not available

7.3 *Accuracy.* Data is not available

Section C4-8. Special Apparatus and Reagents

1. Stainless steel capillary tubing
2. Top loading balance, 1000 g with 0.1 g resolution
3. Methanol anhydrous reagent
4. Silver nitrate
5. 75 mL stainless steel double ended 1/4 inch FNPT cylinder
6. Two 1/4 inch stainless steel valves with MNPT fittings
7. Two 1/4 inch FNPT x 1/4 inch flare fittings
8. 1/16 inch x 1/4 inch stainless steel tube compression fitting reducing union
9. 1/4 inch compression fitting x 1/4 inch flare AN female adaptor
10. 1/4 inch x 1/4 inch copper flare connector
11. 1/4 inch inlet MNPT x 1/4 inch outlet FNPT pressure relief valve

Section C4-9. Procedure

9.1 *Stainless Steel Capillary Tubing Connector.* Take 1/16 inch x 0.007 inch stainless steel tubing and attach a 1/16" nut and ferrule. Connect this to a 1/16 inch x 1/4 inch compression fitting reducing union and then connect it to the 1/4 inch compression fitting x 1/4 inch flare adaptor. The 1/4 inch flare adaptor can then be connected to the 1/4 inch flare fitting on the cylinder assembly just before each chloride determination.

9.2 *Cylinder Assembly.* The cylinder assembly is used as the sampling apparatus for chloride determination of medium and high pressure refrigerants. In order to complete this assembly, all pipe fittings must be tetrafluoroethylene taped to ensure a proper seal at each joint.

Attach the pressure relief valve to the 75 mL stainless steel cylinder. Attach one of the 1/4 inch MNPT x 1/4 inch MNPT stainless steel valves to the pressure relief valve. Connect a 1/4 inch FNPT x 1/4 inch flare fitting to the 1/4 inch MNPT valve. To the other side of the 75 mL cylinder, attach another 1/4 inch MNPT x 1/4 inch MNPT valve. Sampling shall always occur from the side of the 75 mL cylinder which does not employ the pressure relief valve.