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Development and Consolidation of Gas Chromatography Methods for Appendix C to ARI 700

Final Report
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Abstract

New Gas Chromatography methods for determining the composition of zeotropic and azeotropic refrigerant blends are reported. The new procedures provide a single column to determine the blend composition of R-401, R-402, R-403B, R-404A, R-406A, R-407, R-408A, R-409, R-410, R-411, R-412A, R-500, R-502, R-503, R-507, R-509.
This project also revised and generalized the format of each previously developed GC purity method. These methods originally appeared in Appendix C to ARI 700-95 as well as those set forth in a previous MCLR-funded project; namely, *Methods Development for Organic Contaminant Determination in Fluorocarbon Refrigerant Azeotropes and Blends*.

Background

It has been almost 12 years since the inception of ARI Standard 700, *Specifications for Fluorocarbon and Other Refrigerants.* This standard, in conjunction with its Appendix, *Analytical Procedures for ARI Standard 700*, is widely recognized as the technical specification which must be met by new and reclaimed refrigerants throughout the refrigeration and air-conditioning industry.

The current Gas Chromatographic (GC) procedures for organic purity and blend compositions given in Appendix C to ARI Standard 700-95 are summarized in Tables 1 and 2. As evidenced, these GC methods contain significant procedural differences from one refrigerant to the next. In 1988, these procedural differences were largely overcome because there were only nine refrigerants in the initial version of ARI Standard 700. By customizing each chromatographic method to a specific refrigerant, near-ideal separation and quantitation of the broadest possible range of manufacturing impurities was achieved. At that time a laboratory could analyze all nine refrigerants with only three different GC columns, and no sub-ambient temperature instrumentation was required.

Justification

What served well in 1988 has become complicated in the year 2000. In the 1995 Standard 700 revision, the number of refrigerants requiring analysis quadrupled and now stands at 36. Eleven different GC columns are now required (See Table 2). Many columns have no technical specificity in laboratories that analyze more than one refrigerant. For example, the 105 meter DB1301 column required in the analysis of R-113 is unnecessary as the 120 meter DB1301 column required in the analysis of R-22, R32, and R134a can also be adapted to the R-113 analysis. Similarly, the 8 and 16 foot Carbopack B columns used in the analysis of R-32, R-408A, and R-409A may be replaced by one 24 foot Carbopack column. As Table 1 shows, ARI Standard 700 presently mandates 13 separate GC methods for the determination of organic purity. Table 1 also shows 11 separate GC methods for determining the major component compositions of zeotropic and azeotropic blends with six blend composition methods still to be added.

Because of these complications, the industry has requested simplified, technically appropriate GC test methods and/or method revisions for determining the organic purity and blend compositions of these refrigerants. The demand is especially great in the international standards community, as many parties have been reluctant to support refrigerant testing standards that are perceived as unnecessarily complex. Also, the investigators of this project have been in direct contact with several members of the ARI Standard 700 Engineering Subcommittee and with ISO TC 86/SC 8/WG 3 for a consensus as to what work is required. Therefore, in order to safeguard domestic acceptance of ARI 700 and to facilitate international trade, it was most important to revise, simplify, and to consolidate all of the current methods.

Table 1. Refrigerant Type-Dependent GC Test Methods for ARI Standard 700

Refrigerant	**Purity Method** (1)	**Composition Method (blends only)**(1)	**Detector**	**Column(s)** (2)	**Temperature Program(s)** (3)
11	Part 6		FID	K	W
12	Part 7		FID	K	N
13	Part 8		TCD	K	N
22	Part 9		FID	E & K	K & N
23	Part 10		TCD	H	Q
32	Part 11		FID	E & J	A & S
113	Part 12		FID	D	I
114	Part 13		FID	K	R
123	Part 14		FID	F & K	C & V
124	Part 15		FID	K	O
125	Part 16		FID	K	L
134a	Part 17		FID	A & E	B & P
143a	Part 18		FID	A & K	H & P
401	∅	Part 19	TCD	G	M
402	∅	Part 20	TCD	G	M
404A	∅	Part 21	TCD	G	G
405	∅	∅			
406	∅	∅			
407	∅	Part 24	TCD	K	T
408A	∅	Part 25	FID	I	E
409A	∅	Part 26	TCD	I	D
410	∅	Part 27	TCD	B	J
411	∅	∅			
412	∅	∅			
500	∅	Part 30	TCD	C	T
502	∅	Part 31	TCD	C	F
503	∅	Part 32	FID	K	U
507	∅	Part 33	TCD	B	J
508	∅	∅			
509	∅	∅			

1. ∅ = Appendix does not contain any test method.
2. A through K = See Table 2 for column information.
3. A through W = See Table 3 for temperature program.

As a result, this project introduces a general test method using just one GC column that determines the blend compositions of refrigerants 401 through 412, and for refrigerants 500, 502, 503, 507, 508, and 509. This project also revised and generalized the format of each previously developed GC purity method. These methods originally appeared in Appendix C to ARI 700-95 as well as those set forth in a previous MCLR-funded project; namely, *Methods Development for Organic Contaminant Determination in Fluorocarbon Refrigerant Azeotropes and Blends*.

The completion of this work has produced new and revised test methods in a format similar to Appendix C to ARI Standard 700. These methods can be advanced for consideration in the next edition of ARI 700, ISO 12810, and other refrigerant standards after the appropriate review and revision processes of the respective committees have taken place.

Table 2. Columns for Organic Purity and Blend Composition Testing for ARI 700-95

Column	Description (details in ARI 700 Appendix)
A	Bentone / Krytox / SP1000 / Porapak-T, connected in series
B	Carbowax / Dibutyl Maleate / SP1000, connected in series
C	DC200
D	DB1301 (105 meter)
E	DB1301 (120 meter)
F	DB1701 / SPB5, connected in series
G	Krytox
H	Porapak-T
I	SP1000 (8 foot) on Carbopack-B
J	SP1000 (16 foot) on Carbopack-B
K	SP1000 (24 foot) on Carbopack-B

Table 3. Temperature Programs for Organic Purity and Blend Composition Testing for ARI 700-95

Program	Initial Temp., ° C	Initial Time, min.	Ramp, ° C / min.	Final Temp., ° C
A	–28*	10	5	40
B	–20*	10	8	150
C	15*	10	7	60
D	30	3.75	15	100
E	30	3.75	70	110
F	35	isothermal		
G	35	7	20	75
H	35	7	10	150
I	35	10	8	160
J	40	isothermal		
K	40	10	8	50
L	40	6	5	130
M	40	6	30	150
N	40	6	10	160
O	40	10	10	160
P	40	6	10	165
Q	40	3	18	175
R	40	6	10	175
S	45	8	8	150
T	50	isothermal		
U	60	isothermal		
V	125	isothermal		
W	125	4	10	180

*Subambient capability is necessary for temperature programs A–C.

Objective

This work has been sponsored to develop gas chromatographic methods for determining the composition of refrigerants 401 through 412, 500, 502, 503, 507, 508, and 509 using a single column. A second task, involving the same refrigerants, entailed rewriting each previously developed method in simplified form. This second task was expanded to include refrigerants 11, 12, 13, 22, 23, 32, 113, 114, 123, 124, 125, 134a, and 143a that were not part of the original proposal.

The following goals were sought during the development and consolidation of the Gas Chromatography methods included herein:

- The new gas chromatographic procedures for determining the composition of refrigerant blends and azeotropes should all employ the same column.

- The new methods should allow any laboratory skilled in the art to readily use the methods to determine the composition of refrigerant azeotropes and blends.

- The gas chromatographic methods currently appearing in Appendix C to ARI 700-95 and in the previously funded MCLR project: *Methods Development for Organic Contaminant Determination in Fluorocarbon Refrigerant Azeotropes and Blends*, should be replaced with generalized procedures and GC Method Data Sheets to allow for a reduction of redundancy currently existing in the standard.

- The simplified methods should provide all of the detail included in the previous methods for which they are intended to replace. To the greatest extent possible, all of the generalized methods should employ calibration standard preparation, sample analysis, calculations (statistical and otherwise) and techniques consistent with those already appearing in Appendix C to ARI 700-95.

Principle Features of the Resulting Methods

The new methods appear at the end of this report. Each of the 50 resulting procedures has no more than 5 pages of text and in many cases 2 pages. The following new features are added over those previously published in ARI 700-95:

1. The new gas chromatographic procedures for the analysis of the composition of blends do in fact employ a single column (See Table 4). The column utilized for each composition analysis is a Supelco 1% SP-1000 on Carbopack B. The method describing the new generalized procedure for blend composition determination appears in Part 15 of this report with the gas chromatographic methods appearing in Parts 16-32.

Table 4. Summary of Refrigerant Testing Procedures

Refrigerant Testing Procedure	Pages in Current ARI 700	Pages in Consolidated Procedure	Comment on New Methods
General procedure for organic purity	∅	10	Universal method
General procedure for blend composition	∅	5	Universal method
General procedure for blend purity	∅	5	Universal method
R-11	7	3	Consolidated
R-12	9	4	Consolidated
R-13	8	3	Consolidated
R-22	10	5	Consolidated
R-23	8	3	Consolidated
R-32	10	4	Consolidated
R-113	7	3	Consolidated
R-114	9	3	Consolidated
R-123	10	5	Consolidated
R-124	9	4	Consolidated
R-125	8	4	Consolidated
R-134a	11	4	Consolidated
R-143a	10	5	Consolidated
R-401 (Composition)	5	2	New Single Column
R-402 (Composition)	5	2	New Single Column
R-403 (Composition)	∅	2	New Single Column
R-404 (Composition)	6	2	New Single Column
R-406 (Composition)	∅	2	New Single Column
R-407 (Composition)	7	2	New Single Column
R-408 (Composition)	6	2	New Single Column
R-409 (Composition)	6	2	New Single Column
R-410 (Composition)	5	2	New Single Column
R-411 (Composition)	5	2	New Single Column
R-412 (Composition)	5	2	New Single Column
R-500 (Composition)	5	2	New Single Column
R-502 (Composition)	5	2	New Single Column
R-503 (Composition)	5	2	New Single Column
R-507 (Composition)	5	2	New Single Column
R-508 (Composition)	5	2	New Single Column
R-509 (Composition)	5	2	New Single Column
R-401 (Purity)	∅	3	Single Column
R-402 (Purity)	∅	3	Single Column
R-404 (Purity)	∅	3	Single Column
R-405 (Purity)	∅	3	Single Column
R-406 (Purity)	∅	3	Single Column
R-407 (Purity)	∅	3	Single Column
R-408 (Purity)	∅	3	Single Column
R-409 (Purity)	∅	3	Single Column
R-410 (Purity)	∅	3	Single Column
R-412 (Purity)	∅	3	Single Column
R-500 (Purity)	∅	3	Single Column
R-502 (Purity)	∅	3	Single Column
R-503 (Purity)	∅	3	Single Column
R-507 (Purity)	∅	3	Single Column
R-508 (Purity)	∅	3	Single Column
R-509 (Purity)	∅	3	Single Column

2. The composition methods presently appearing in Appendix C to ARI 700 were developed at different laboratories and employ some variety in both materials and laboratory practice. This has imposed a considerable financial, procedural and training burden on laboratories that must employ the procedures of the standard or establish procedures of their own and demonstrate equivalence (as currently required by law).

   The new methods developed in the course of this study standardize, to the greatest extent possible, analytical procedures, equipment, calibration standard preparation and sampling methods. The methods reported herein are considered readily usable by any refrigerant-testing laboratory skilled in the art.

3. The GC Method Data Sheet, found in Parts 2-14, 16-32, and 34-50, can stand alone if used in conjunction with its respective Generalized Procedure found in Part 1, Part 15 or in Part 33. This simplification has allowed for a needed reduction in verbiage in the current edition of ARI 700-95 while maintaining its technical integrity.

4. All of the generalized methods employ similar calibration standard preparation techniques, sample analysis, gas chromatographic equations, refrigerant transfer procedures and sampling techniques that are consistent with those already appearing in ARI 700. Statistical parameters, definitions and calculations are those used in ARI 700 and while not detailed in this report, are readily known and available to all personnel who would employ the procedures.

5. All of the methods appearing in this report are ready for review from all appropriate domestic and international committees for which they were originally written.

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METHODS

DEVELOPMENT AND CONSOLIDATION
OF GAS CHROMATOGRAPHY METHODS
FOR APPENDIX C TO ARI 700

TABLE OF CONTENTS

PAGE

TABLE OF CONTENTS (CONTINUED)

PAGE

TABLE OF CONTENTS (CONTINUED)

PAGE

LIST OF FIGURES

LIST OF FIGURES (CONTINUED)

LIST OF FIGURES (CONTINUED)

LIST OF TABLES

LIST OF TABLES (CONTINUED)

LIST OF TABLES (CONTINUED)

LIST OF TABLES (CONTINUED)

LIST OF TABLES (CONTINUED)

LIST OF TABLES (CONTINUED)

PART 1

GENERAL PROCEDURE FOR THE DETERMINATION OF PURITY OF NEW AND RECLAIMED REFRIGERANTS BY GAS CHROMATOGRAPHY

Section 1. Purpose

The purpose of this test method is to determine the purity of new and reclaimed refrigerants by gas chromatography (GC).

Section 2. Scope

This test method is for use in conjunction with the GC Method Data Sheets for all refrigerants as listed in section 12 of this method.

Section 3. Definitions

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

Section 4. Principle

The organic purity of new and reclaimed refrigerants is determined by programmed temperature gas chromatography using either a packed or capillary GC column and either a flame ionization detector (FID) or thermal conductivity detector (TCD). Component peak areas are integrated electronically and quantified by the area normalization-response factor method by reference to a suitable calibration standard.

Section 5. Applicability

This method is applicable for the determination of the organic impurities typically present in new and reclaimed refrigerant. These impurities are listed in the GC Method Data Sheet for each respective refrigerant.

Section 6. Limitations and Interferences

This method will not detect impurities that coelute within the large peak matrix of the refrigerant being analyzed. Additional limitations and interferences (if any) appear in the GC Method Data Sheet for each refrigerant.

Section 7. Sensitivity, Precision, and Accuracy

Values for these statistical parameters are given in Table 1 of the respective GC Method Data Sheet for each refrigerant.

Section 8. Special Apparatus and Reagents

NOTE: Equivalents may be substituted.

1. Gas chromatograph: Model 5890, equipped with FID, TCD and with capillary column split injection, subambient temperature valve and packed column capability as specified in each refrigerant’s GC Method Data Sheet, Hewlett Packard, Wilmington, DE.
2. Electronic integrator: Model# 3396, Hewlett Packard, Wilmington, DE.
3. Gas chromatography columns specific to each refrigerant are given in Section 3 of the appropriate GC Method Data Sheet.
4. Glass collecting tubes: 500 mL, 250 mL and 125 mL, LG-8601, Lab Glass Inc., Vineland, NJ. (Enlarge side outlet opening to accommodate a crimp-on 2-cm septum. Apply fiberglass tape outside for protection from breakage)
5. Steel cylinder: 1L, with a single #9 valve (#1014-C, Superior Valve), 3/8" pipe neck, E. F. Britten Co., Cranford, NJ.
6. Deflected point needles: Cat# 7174, #22, Popper and Sons, Inc., New Hyde Park, NY.
7. Swivel union: US44, United Refrig. Inc., Philadelphia, PA.
8. Serum bottle: 125 mL, (Note: Bottle holds 160 mL when liquid full.) Cat# 223748, Wheaton Glass, Vineland, NJ.
9. Impurities for calibration standard preparation: The fluorochemicals may be purchased from Lancaster, Windham, NH and Synquest, Inc., Alachua, FL. The hydrocarbons may be purchased from Scott Specialty Gases, Inc., Plumbsteadville, PA. All other impurities may be purchased from Aldrich, Milwaukee, WI. See Table 2 of the GC Method Data sheet for the specific impurities required for each refrigerant. The purity of each calibration component must be predetermined by gas chromatography and, if necessary, by GC/Mass Spectroscopy (GC-MS).
10. Stainless steel cylinder: 1L, 304L-WDF4-1000, 1/4" pipe, Whitey Co., Highland Heights, OH.

Section 9. Procedure for Low Pressure Refrigerants

NOTE: The following procedure is generalized for the determination of purity for low-pressure refrigerants, R-11, R-113, and R-123. Each respective GC Method Data Sheet contains the detector type, the column(s), the chromatographic conditions, and the impurities to be added for calibration standard preparation required to perform the procedure stated herein.

9.1 *Calibration Standard Preparation for Low Pressure Refrigerants*

a. Obtain a stock of the highest purity refrigerant for the desired calibration standard preparation.

b. Determine the tare weight (to the nearest 0.01g) of a 125 ml serum bottle with septum and cap loosely attached, then fill with stock refrigerant within about 1.5 cm of the top. Crimp on the septum.

c. Reweigh and subtract the tare weight in step b to obtain the grams of stock refrigerant added.

d. Individually, and in turn, add the volumes of each calibration component indicated in Table 2 of the corresponding GC Method Data Sheet through the septum and below the surface of the stock refrigerant already in the bottle. Use an appropriately sized milliliter gas tight syringe with deflected point needles for gases and a liquid microliter syringe for liquids (care should be taken to account for syringe needle volumes). Shake the bottle to mix after the addition of each component.

e. Total the µg added column and combine this weight with that of step c to obtain the total weight (to the nearest 0.01g) of the calibration standard in the bottle.

f. Calculate the ppm added (to the nearest 1 ppm) for each component by dividing the µg added by the total weight of standard in the serum bottle. (step e)

g. Calculate the ppm present for each component by combining the ppm present in the stock refrigerant (if any) and the ppm component added (refer to Section 5, Note 1 in the GC Method Data Sheet). The ppm component present values are those used for determining the method response factors.

h. Place the serum bottle standard in an ice bath and, after it is ice cold, remove and immediately replace with a new septum.

i. On the label, write the ppm present values for each component, date of preparation, gross weight and total grams of calibration sample. Store in a refrigerator. Discard and prepare a new standard when the sample weight falls below 60% of the original weight.

NOTE: For long term storage, the standard is transferred to a steel cylinder of appropriate volume.

9.2 *Determination of Component Response Factors for Low Pressure Refrigerants (See Section 9.6, Note 3)*

a. Set up the electronic integrator for an area normalization response factor calibration.

b. Analyze the calibration standard solution in triplicate using the chromatographic conditions described in Section 3 of the respective refrigerant's GC Method Data Sheet.

c. Using the matrix of the primary low pressure refrigerant (R-11, R-113, R-123) as the reference peak, perform the necessary functions to have the integrator determine each component's Relative Response Factor (RRF_i) which is then stored. Response Factors are calculated as follows:

$$ARF_i = \frac{\text{Wt\% i in Cal. Std.}}{A_i} \qquad ARF_x = \frac{100.0000 - S}{A_x}$$

A_i = peak area of component i. (average of 3 runs).
ARF_i = Absolute Response Factor of contaminant i.
A_x = peak area of primary refrigerant (average of 3 runs).
ARF_x = Absolute Response Factor of primary refrigerant.
S = Weight % sum of all impurities present.

Then, using the primary refrigerant as the reference peak:

$$RRF_i = \frac{ARF_i}{ARF_x} \qquad RRF_x = \frac{ARF_x}{ARF_x} = 1.0$$

RRF_i values are computed to the nearest 0.0001 unit.

9.3 *Sampling*

Submitted samples should be in either metal cylinders or in glass or plastic bottles such that the containers are at least 80% liquid full.

9.4 *Sample Analysis*

Analyze the sample using the chromatographic conditions described in Section 3 of the GC Method Data Sheet for each refrigerant. The sample and syringe are pre-cooled (refrigerator or ice bath) to 10°C before sampling. This is to simplify loading the sample into the microliter syringe. Use component spiking and/or GC-MS to identify questionable peaks.

9.5 *Calculations*

a. The weight percentage of each component is calculated as follows:

$$W_i = \frac{100 \text{ x } RRF_i \text{ x } A_i}{\sum (A_i \text{ x } RRF_i)}$$

Where:

W_i = weight percentage of component i.
RRF_i = relative response factor for component i.
A_i = peak area of component i.
Σ…= sum of all component peak areas times their respective relative response factors.

9.6 *Notes for Low Pressure Refrigerants*

1. The purest refrigerant will contain some of the impurities listed in Table 1 of the GC Method Data Sheet. The ppm amounts of impurities already in the primary refrigerant are determined via the method of Standards Addition. Individual impurity peak areas in the stock are increased in the calibration standard by the ppm amount of the corresponding impurity added. The ppm already present is combined with the ppm added to give the total ppm component present in the calibration standard.

2. To preserve the stock of calibration component, it is suggested to load a small evacuated 125 mL gas collecting tube to 101.325 kPa from the liquid phase as illustrated in Figure 1. The appropriate volume is then withdrawn and injected into the serum bottle containing the primary refrigerant. For impurities that are liquid at room temperature, inject the indicated µl volumes of each respective component into the serum bottle.

3. Depending upon the electronic integrator used, it is often more desirable to convert the ppm values to wt % for response factor calculations and for reporting purposes.

4. Typically, commercially available R-123 contains from 1 to 7% R-123a isomer and 300 to 600 ppm R-123b isomer. The concentration of R-123a in the stock is determined separately by the method of Standards Addition (adding percent amounts of R-123a to the stock R-123 and chromatographing as in Section 9.1). The calculated RRF_{123a} value is also assigned to the R-123b isomer, as R-123b is not commercially available for separate calibration. The amounts present are added to Table 2 in the GC Method Data Sheet; the R-123a isomer shown as percent present.

Section 10. Procedure for High Pressure Refrigerants

NOTE: The following procedure is generalized for the determination of purity for high-pressure refrigerants, R-12, R-22, R-32, R-114, R-124, R-125, R-134a, and R-143a. Each respective GC Method Data Sheet contains the detector type, the column, the chromatographic conditions, and the impurities necessary for the following calibration standard preparation.

10.1 *Primary Calibration Standard Preparation and Analysis for High Pressure Refrigerants*

a. Crimp-on the septum, then determine the internal volume of the 500 ml gas bulb by weighing the bulb empty, then filled to maximum capacity with water. Record the grams of water as ml volume capacity on the outside of the bulb (to the nearest 0.1ml).

b. Thoroughly dry the gas bulb, then assemble the apparatus as illustrated in Figure 1.

c. Attach a cylinder of the refrigerant for which a calibration standard is desired (hereafter referred to as the primary refrigerant). Make sure that the refrigerant used is of high purity. (Refer to Note 1 in Section 10.7)

d. With valve "A" closed, open all other valves and evacuate to 0.133 kPa.

e. Close valve "D" and monitor the gauge for several minutes to ensure that the system is not leaking.

f. Close metering valve "E". Open valve "A", then slowly open valve "E" and flash liquid phase refrigerant to bring the system to 101.325 kPa. Close Valve "A".

g. Repeat steps d through f.

h. Close valves "B" and "C" and remove the bulb from the vacuum/sampling apparatus.

i. Calculate the grams of primary refrigerant added to the bulb as follows:

$$\textit{grams added} = \frac{\underline{\textit{MW Ref. x Internal Volume of Bulb (mL)}}}{24{,}450}$$

Where:

MW Ref. = Molecular weight of the primary refrigerant
24,450 = Volume occupied by 1 mole of the primary refrigerant at 25°C and 101.325 kPa.

NOTE: Alternatively, the grams added may be determined by weighing (0.0001g) the dried, evacuated bulb (step d above) and then reweighing at step h.

j. Individually, and in turn, add the volumes of each gaseous calibration component indicated in Table 2 of the corresponding GC Method Data Sheet to the calibration bulb. Use an appropriately sized microliter or milliliter gas tight syringe with deflected point needles. (See Note 2 in Section 10.7 of this method)

k. Into a 30 ml serum bottle that has been capped and crimped with a septum, add the exact volumes of the liquid impurities in the order given in Table 3 (if any) of each GC Method Data Sheet. Add each impurity by syringe injection through the septum using a #22 needle (or smaller) as a vent. After addition, shake vigorously to mix. Label, date and store in a refrigerator.

NOTE: For refrigerants which have no liquid impurities listed in Table 4 of the GC Method Data Sheet skip steps k-n and proceed to step o.

l. Evacuate a 125 ml gas sampling bulb (refer to Figure 1) with its internal volume pre-measured, and fill to 101.325 kPa with the primary refrigerant for which a calibration standard is desired.

NOTE: For R-114, a 250-mL gas sampling bulb is used.

m. Accurately withdraw and inject exactly 10 microliters of solution from the 30-mL serum bottle into the 125-mL bulb. Allow to equilibrate for 30 minutes.

NOTE: For R-114, 20 µL is added instead of 10 µL and for R-134a, 5 µL is added instead of 10 µL.

n. Using a gas tight syringe, withdraw vapor from the 125-mL bulb and inject the exact volume as listed in Table 1 below into the 500-mL calibration bulb. The µg of each component thus added is calculated as follows and is added to the fourth column of Table 2 of the appropriate GC Method Data Sheet as follows:

$$\mu g \text{ of component i added} = \frac{g_i \times D}{32 \times A}$$

Where:

g_i = g from step k and found in Table 4 of the GC Method Data Sheet.
A = internal mL of the gas sampling bulb.
32 = total (approximate) mL of solution in step k/Table 3.
D = dilution ratio as listed in Table 1 below.

o. Total the µg added column in Table 2 (refer to the GC Method Data Sheet) and combine this weight with that of step i to obtain the total weight of the sample (to the nearest 0.0001 g) in the bulb.

p. Calculate the ppm added (to the nearest 1 ppm) for each component by dividing the µg added by the total weight of sample in the gas bulb (step o).

Table 1. Vapor Volume and Dilution Ratio (D) for Liquid Impurities Used for Primary Calibration Standard

Refrigerant	Vapor Volume (used in step n)	Dilution Ratio (D)
R-12	5.0 mL	50,000
R-22	5.0 mL	50,000
R-32	None added	None
R-114	10.0 mL	200,000
R-124	5.0 mL	50,000
R-125	2.0 mL	20,000
R-134a	5.0 mL	25,000
R-143a	None added	None

q. Calculate the ppm present for each component by combining the ppm present in the primary refrigerant (if any) and the ppm component added (See Note 1 in Section 10.7 of this method). The ppm component present values are those used for determining the method response factors.

r. Allow the gas calibration bulb to stand for 20-30 minutes to equilibrate. The standard will be stable for 3 days.

10.2 *Determination of Component Response Factors (Refer to Section 10.7, Note 3)*

a. Set up the electronic integrator for an area normalization-response factor calibration.

b. Analyze the calibration standard bulb in triplicate using the chromatographic conditions described in Section 3 of the GC Method Data Sheet.

c. Using the primary refrigerant as the reference peak, perform the necessary functions to have the integrator determine each component's Relative Response Factor (RRF_i) which is then stored. Response factors are calculated as follows:

$$ARF_i = \frac{\text{Wt\% i in Cal. Std.}}{A_i} \qquad ARF_x = \frac{100.0000 - S}{A_x}$$

Where:

ARF_i = Absolute Response Factor of component i.
A_i = peak area of component i (average of 3 runs)
A_x = peak area of primary refrigerant (average of 3 runs).
ARF_x = Absolute Response Factor of Primary Refrigerant.
S = Weight % sum of all impurities present.

Then, using the primary refrigerant as the reference peak:

$$RRF_i = \frac{ARF_i}{ARF_x} \qquad RRF_x = \frac{ARF_x}{ARF_x} = 1.0$$

RRF_i values are computed to the nearest 0.0001 unit.

Refer to Note 5 in Section 10.7 for R-32 and refer to Note 6 in Section 10.7 for R-134a.

10.3 *Secondary Calibration Standard Preparation*

NOTE: A secondary calibration standard is prepared in much larger quantity due to the comparatively short lifetime of the primary bulb standard. The primary bulb standard is necessary initially because of inherent phase distribution of added components if simply preparing and calibrating a standard such as described here. The secondary standard is analyzed as a sample against the primary standard and then used subsequently as the daily calibration standard.

a. Evacuate a 1L steel cylinder and determine the tare weight to the 0.1g.

b. Attach a Swagelok nut and septum to the valve and then externally cool the cylinder in ice water. Open the cylinder valve.

c. While keeping the cylinder in ice water, individually and in turn add the volume of each gaseous component given in Table 2 of the GC Method Data Sheet multiplied by the factor given in Table 2 below for each refrigerant to the cylinder by syringe injection through the septum. Similarly, add the volume of liquid refrigerant impurities from Section 10.1 k (if any) in the quantity specified in Table 2 below. Close the cylinder valve and remove the Swagelok nut and septum.

Table 2. Secondary Calibration Standard Gaseous and Liquid Impurity Addition Information

Refrigerant	Gaseous Impurities Factor[1]	Liquid Refrigerant Impurities (mL)
R-12	400	0.15
R-22	500	0.20
R-32	700	None
R-114	400	0.30
R-124	400	0.15
R-125	400	0.15
R-134a	500	0.10
R-143a	350	None

[1] Multiply each gaseous impurity in Table 2 of the GC Method Data Sheet by the factor given above to give the total volume of impurity to be added for the secondary calibration standard.

d. Evacuate a second clean, dry 1L steel cylinder and determine the tare weight to the nearest 0.1g.

e. Cool the cylinder in ice water and attach a short (up to 61 cm) section of flex line from the primary refrigerant's supply cylinder. Purge a small amount of the primary refrigerant through the flex line before immediately attaching to the 1L cylinder.

f. Open the 1L cylinder valve, then open the refrigerant cylinder of the primary refrigerant for which it is desired to constitute a secondary calibration standard. While keeping the 1L cylinder in ice water, fill the 1L cylinder with the amount of liquid phase of the primary refrigerant listed in Table 3 below. If significantly more than the prescribed weight of the primary refrigerant is added, vent the cylinder to give approximately the weight desired in Table 3 (See Note 4 in Section 10.7 of this method). Remove the 1L cylinder from the ice bath and allow it to warm to ambient temperature.

g. Place the 1L secondary calibration standard cylinder (the cylinder mentioned in steps a, b, and c) in the ice bath and cool for 30 minutes.

h. Using a short double female swivel coupler, invert the 1L cylinder containing the primary refrigerant in step f, and connect to the secondary calibration standard cylinder. Open the valve slightly and purge some of the primary refrigerant vapor to sweep the coupler before connecting to the secondary calibration standard cylinder. Warm, but do not overheat, the cylinder containing the primary refrigerant with a heat gun.

Table 3. Approximate Amount of Primary Refrigerant to Add to Secondary Calibration Standard

Refrigerant	Weight of Primary Refrigerant (g)
R-12	1200
R-22	1100
R-32	900
R-114	1300
R-124	1200
R-125	1100
R-134a	1100
R-143a	820

i. Open the valves on both cylinders and allow all of the primary refrigerant to transfer into the calibration standard cylinder. Close the cylinder valves.

j. Remove the calibration cylinder from the ice bath and allow the cylinder to reach ambient laboratory temperature before the final weighing. Dry off the cylinder then reweigh it to the nearest 0.1g.

k. Subtract the tare weight (from step a) from the total weight (step j) to obtain the total grams of standard in the cylinder. Record this weight together with the cylinder tare weight and the date of preparation on the cylinder label.

l. Roll the cylinder for at least 4 hours to thoroughly mix.

m. Analyze the cylinder contents in triplicate as described in Section 3 of the GC Method Data Sheet, loading first into an evacuated gas bulb as shown in Figure 1.

n. Average the results calculated electronically (see Section 10.6, Calculations) and tabulate to the nearest 1ppm. List each component on the cylinder label with the ppm amount for each. This cylinder is used henceforth as the calibration standard until the loss of standard weight indicates that the internal volume of liquid phase is less than 60% of the total internal volume of the cylinder. Liquid densities of the primary refrigerants are listed in Table 4 below.

Table 4. Liquid Densities of Refrigerants

Refrigerant	Density at 25°C (g/mL)
R-12	1.311
R-13	0.907
R-22	1.194
R-23	0.670
R-32	1.100
R-114	1.456
R-124	1.364
R-125	1.250
R-134a	1.202
R-143a	0.946

10.4 *Sampling*

Submitted sample cylinders must contain sufficient liquid phase (80% liquid full is recommended) for analysis.

10.5 *Sample Analysis*

Analyze the sample using the chromatographic conditions specified in Section 3 of the respective GC Method Data Sheet. Load the sample as illustrated in Figure 1 by flashing the liquid phase into an evacuated gas bulb and bringing to 101.325 kPa pressure.

NOTE: Alternatively, the sample liquid phase may be flashed into a Tedlar bag (1L recommended) and the sample for GC analysis is withdrawn from the bag.

10.6 *Calculations*

a. The weight percentage of each component is calculated as follows:

$$W_i = \frac{100 \text{ x } RRF_i \text{ x } A_i}{\sum (A_i \text{ x } RRF_i)}$$

Where:

W_i = weight percentage of component i.
RRF_i = relative response factor for component i.
A_i = peak area of component i.
$\Sigma\ldots$= sum of all component peak areas times their respective relative response factors.

b. Report sample component concentrations to the nearest 0.0001% (or to the nearest 1 ppm). If the results are less than the individual detection limits (see Table 1), then report < the detection limit (DL) value given.

10.7 *Notes for High Pressure Refrigerants*

1. The purest refrigerant will contain some of the impurities listed in Table 1 of the GC Method Data Sheet. The ppm amounts of impurities already in the primary refrigerant are determined via the method of Standards Addition. Individual impurity peak areas in the stock are increased in the calibration standard by the ppm amount of the corresponding impurity added. The ppm already present is combined with the ppm added to give the total ppm component present in the calibration standard.

2. To preserve the stock of calibration component, it is suggested to load a small evacuated 125 mL gas collecting tube to 101.325 kPa from the liquid phase as illustrated in Figure 1. The appropriate volume is then withdrawn and injected into the 500-mL calibration bulb.

3. Depending upon the electronic integrator used, it is often more desirable to convert the ppm values to wt % for response factor calculations and for reporting purposes.

4. During the primary refrigerant's addition to the 1L cylinder (secondary standard preparation), it is unnecessary to bring the cylinder to ambient temperature between weighings as only an approximate weight is required.

5. To determine the R-125 packed column response in the presence of HCC-40 while analyzing R-32, it is necessary to first establish an RRF ratio for HCC-40 between the two columns. This is done by analyzing the standard on both columns and determining the RRF_{40} on the packed column

(i.e., $RRF_{40\text{-P}}$) by the method of Standards Addition. Having established this ratio R (i.e., $R = RRF_{40\text{-c}}/RRF_{40\text{-P}}$), the area in the 125/40 combination peak attributed to HCC-40 is:

$$A_{40\text{-}P} = \frac{R \text{ x Weight \% R-40}}{RRF_{40\text{-}C} \text{ x } ARF_{32\text{-P}}}$$

Then: $A_{125} = \text{Comb. Peak Area} - A_{40\text{-}P}$

And: $$ARF_{125} = \frac{\text{Weight \% R-125}}{A_{125}}$$

The same equations are used to determine the weight % of R-125 present when analyzing samples. The R-value should be checked periodically in conformance to standard quality control practice.

6. While analyzing R-134a, in order to separate 31 and 1140 (which coelutes on the capillary column), repeat the capillary column analysis exactly as given in Section 3 of the GC Method Data Sheet except that the column temperature is held at 50°C (isothermal) throughout. The two components will be resolved at about 15 minutes retention time with the 31 peak eluting 0.8 minutes before the 1140 peak.

11. Procedure for Very High Pressure Refrigerants

NOTE: The following procedure is generalized for the determination of purity for very high-pressure refrigerants, R-13 and R-23. Each respective GC Method Data Sheet contains the detector type, the column, the chromatographic conditions, and the impurities necessary for the following calibration standard preparation.

11.1 *Calibration Standard Preparation and Analysis*

a. Attach a Swagelok® nut and septum to one end of a clean dry stainless steel cylinder and a vacuum pump line to the other. Evacuate the cylinder to full vacuum, or 0.133 kPa.

b. Referring to Table 2, Calibration Standard Components, of the appropriate GC Method Data Sheet, and, using the appropriately sized milliliter gastight syringe, carefully add the specified volume of each calibration component to the cylinder via the Swagelok® nut/septum. Also, refer to Section 11.6, Note 2.

c. Remove the Swagelok® nut/septum, and then tare weigh the 1L cylinder to the nearest 0.1g.

d. Position the cylinder inside a cold bath, either alcohol or dry ice, and allow to cool while performing subsequent steps e through i.

e. Obtain a stock cylinder of the VHP refrigerant under preparation whose purity has been previously established by either GC analysis or by the Method of Standards Addition (See Section 11.6 Note 1). This cylinder must be precooled either in a refrigerator or external ice bath to at least 20°C below the critical temperature (t_c) of the refrigerant.

f. Evacuate a second clean, dry 1L stainless steel cylinder (as in step a above). Tare weigh this cylinder to the nearest 0.1g.

g. Place this second 1L cylinder into a dry ice or cold (alcohol) bath and cool for 10-15 minutes.

h. Using a Swagelok® steel mesh Teflon® line (flexline), invert the VHP source cylinder and connect it to the second 1L cylinder. Initially purge the flexline with VHP gas (flashed liquid phase) so as to purge air from the line before the final connection is made.

i. Transfer the specified amount of VHP stock refrigerant liquid phase into the 2nd 1L cylinder as specified in Table 5 below. If too much gas has been added, then slowly vent until the weight is about that specified.

NOTE: It is not necessary here to weigh back at room (ambient) temperature as only an approximate grams of VHP stock is necessary.

Table 5. Approximate Amount of Liquid VHP Refrigerant to Add to Calibration Standard

Refrigerant	Weight of VHP Refrigerant (g)	Corresponding Pressure (kPa)
R-13	150	1140
R-23	200	2516

j. Remove the second 1L cylinder from the cold bath, and, using the Teflon® flexline, connect it to the 1L Calibration Standard cylinder still in the cold bath (step d).

k. Using a heat gun, warm the second 1L cylinder and, when warmed to ambient temperature, transfer the entire contents into the 1L Calibration Standard cylinder. Close the cylinder valve, remove the flexline, and remove the cylinder from the cold bath.

l. Position the cylinder on a roller mill, and roll to mix while allowing the cylinder to slowly warm to ambient (room) temperature. Note that the liquid phase will slowly vaporize as the VHP gas warms to room temperature.

m. When equilibrated at room temperature, weigh the cylinder (to the nearest 0.1g). Subtract the tare weight (step c) and record the difference as the grams of stock VHP refrigerant added.

n. Total the grams of all added impurities (step b) and add this sum to the grams of stock from step m above. This amount represents the total weight of calibration sample present.

o. Divide the grams of each added impurity multiplied by 10^6 by the total weight of calibration sample and record these results in Table 2 of the GC Method Data Sheet as ppm component added. Correct any amount for the purity of the added component as previously established (Section 8, 9)

p. Add the ppm amount of any impurity component already present in the VHP stock refrigerant to the ppm amount of impurity added, and complete Table 2 by recording each respective value in the Total ppm Present column (see Section 11.6, Note 1)

q. Record the individual impurities and ppm amounts present on the Standard Cylinder, the gross weight, and the date of preparation. Because the standard mixture is all vapor, the mixture is stable indefinitely, or until all is consumed.

11.2 *Determination of Component Response Factors (Refer to Section 11.6, Note 3)*

a. Set up the electronic integrator for an area normalization-response factor calibration.

b. Analyze the calibration standard cylinder in triplicate using the chromatographic conditions described in Section 3 of the GC Method Data Sheet. Refer to Figure 1 for loading into the gas sampling bulb.

c. Using the primary refrigerant as the reference peak, perform the necessary functions to have the integrator determine each component's Relative Response Factor (RRF_i) which is then stored. Response factors are calculated as follows:

$$ARF_i = \frac{\text{Wt\% i in Cal. Std.}}{A_i} \qquad ARF_x = \frac{100.0000 - S}{A_x}$$

Where:

ARF_i = Absolute Response Factor of component i.
A_i = Peak Area of component i (average of 3 runs).
A_x = Peak Area of primary refrigerant (average of 3 runs).
ARF_x = Absolute Response Factor of Primary Refrigerant.
S = Wt% sum of all impurities present.

Then, using the primary refrigerant as the reference peak:

$$RRF_i = \frac{ARF_i}{ARF_x} \qquad RRF_x = \frac{ARF_x}{ARF_x} = 1.0$$

RRF_i values are computed to the nearest 0.0001 unit.

$$ARF_i = \frac{\text{Wt\% i in Cal. Std.}}{A_i} \qquad ARF_x = \frac{100.0000 - S}{A_x}$$

Where:

ARF_i = Absolute Response Factor of component i.
A_i = Peak Area of component i (average of 3 runs).
A_x = Peak Area of primary refrigerant (average of 3 runs).
ARF_x = Absolute Response Factor of Primary Refrigerant.
S = Wt% sum of all impurities present.

Then, using the primary refrigerant as the reference peak:

$$RRF_i = \frac{ARF_i}{ARF_x} \qquad RRF_x = \frac{ARF_x}{ARF_x} = 1.0$$

RRF_i values are computed to the nearest 0.0001 unit.

11.3 *Sampling*

Submitted sample cylinders must be stainless steel, either 500 mL or 1L volume. Source cylinders or containers are sampled above the critical temperature (T_c) of the VHP refrigerant so as to ensure the refrigerant is all vapor phase (refer to Note 4).

11.4 *Sample Analysis*

Analyze the sample using the chromatographic conditions specified in Section 3 of each GC Method Data Sheet. Load the vapor sample into the gas bulb as illustrated in Figure 1. Be certain that metering valve "E" is rated above the pressure of the VHP refrigerant gas in the sample cylinder. Alternatively, the sample may be slowly purged through Tygon® tubing and, after purging air from the tubing, the GC sample is withdrawn through the tubing in proximity to the cylinder valve.

NOTE: Alternatively, the sample liquid phase may be flashed into a Tedlar bag (1L recommended) and the vapor then sampled for GC analysis.

11.5 *Calculations*

a. The weight percentage of each component is calculated as follows:

$$W_i = \frac{100 \text{ x } RRF_i \text{ x } A_i}{\sum (A_i \text{ x } RRF_i)}$$

Where:

W_i = weight percentage of component i.
RRF_i = relative response factor for component i.
A_i = peak area of component i.
Σ…= sum of all component peak areas times their respective relative response factors.

b. Report sample component concentrations to the nearest 0.0001% (or to the nearest 1 ppm). If the results are less than the individual detection limits (see Table 1), then report < the detection limit (DL) value given.

11.6 *Notes for Very High Pressure Refrigerants*

1. The purest refrigerant will contain some of the impurities listed in Table 1 of the GC Method Data Sheet. The ppm amounts of impurities already in the primary refrigerant are determined via the method of Standards Addition. Individual impurity peak areas in the stock are increased in the calibration standard by the ppm amount of the corresponding impurity added. The ppm already present is combined with the ppm added to give the total ppm component present in the calibration standard. The ppm added amounts for calibration should be greater than the usual amounts present in typical samples. This is because the peak response of the TCD becomes increasingly non-linear as impurity concentration levels increase.

2. To preserve the stock of calibration component, it is suggested to load an evacuated 250-mL gas collecting tube to 110 kPa from the liquid phase as illustrated in Figure 1. The appropriate volume is then withdrawn and injected into the 1L stainless steel cylinder.

3. Depending upon the electronic integrator used, it is often more desirable to convert the ppm values to wt % for response factor calculations and for reporting purposes.

4. VHP refrigerants all have T_c values either near or somewhat below room temperature. Because ARI Standard 700 specifies that the liquid phase of refrigerants is sampled for purity, et. al., the only way to form sufficient liquid phase for these analyses is to cool the VHP sample to at least 20°C below $T_{c.}$ In practice, consistent impurity phase distributions are difficult to control because the relative amounts of sample liquid and vapor phases are strongly dependent upon the temperature of the refrigerant at the time of analytical sampling and analysis. For this reason, the decision was made to ensure sample homogeneity by converting all source refrigerants to vapor phase before analytical GC sampling. In cold weather, the sampling of very large containers (outdoors) is accomplished by sampling the liquid phase and then warming the sample cylinder and contents to above T_c before GC analysis.

5. A 32-fold increase in peak sensitivity is possible by operating the chromatographic TCD at the high sensitivity position. However, at higher sensitivity, the useful lifetime of the detector (hot wires) is diminished, the baseline noise and background is often intolerable and peak area reproducibility is generally degraded. For these reasons, the lower sensitivity position was chosen for routine applications.

Section 12. GC Method Data Sheets

The GC Method Data Sheet for each respective refrigerant can be found in the following Part:

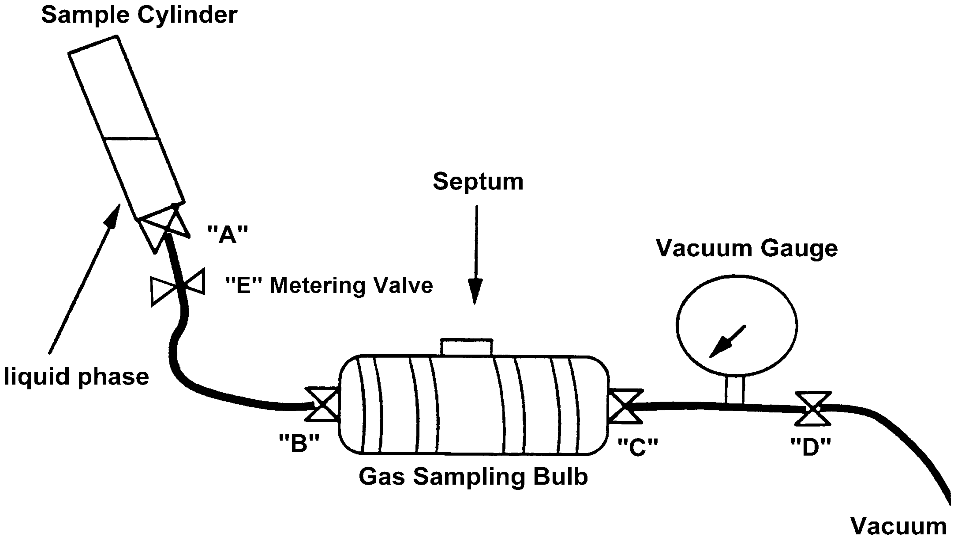
Table 6. GC Method Data Sheets

Refrigerant	Procedure for Calibration Standard Preparation	GC Method Data Sheet Part Number
R-11	Part 1, Section 9	Part 2
R-12	Part 1, Section 10	Part 3
R-13	Part 1, Section 11	Part 4
R-22	Part 1, Section 10	Part 5
R-23	Part 1, Section 11	Part 6
R-32	Part 1, Section 10	Part 7
R-113	Part 1, Section 9	Part 8
R-114	Part 1, Section 10	Part 9
R-123	Part 1, Section 9	Part 10
R-124	Part 1, Section 10	Part 11
R-125	Part 1, Section 10	Part 12
R-134a	Part 1, Section 10	Part 13
R-143a	Part 1, Section 10	Part 14

Section 13. References

1. Air-Conditioning and Refrigeration Institute, *Appendix C to ARI Standard 700-95: Analytical Procedures for ARI Standard 700-95*, 4301 North Fairfax Drive, Arlington, Virginia 22203.

Figure 1. Apparatus Used for Sampling Calibration Standards and Samples



Part 2

R-11 GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Section 9 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-11.

Section 2. Limitations and Interferences

This method is applicable and calibrated for only those impurities found in Table 2. Other impurities that have been detected on occasion are listed (with retention times) in Table 3. This method will not detect any impurities that may elute within the comparatively large R-11 peak matrix.

Section 3. Gas Chromatographic Analysis Equipment and Conditions

Packed column: 7.32 m x 3.17 mm OD stainless steel, 1% SP-1000 on Carbopack B, 60/80 mesh, Supelco, Bellefonte, PA.

Detector	FID
Carrier Gas	30 mL He/Min.
Initial Column Temp.	125°C
Initial Hold	4 Min.
Program	10 K/Min.
Final Column Temp.	180°C
Post Hold	14 Min.
Sample	1 µL (liquid syringe)
Detector Temp.	250°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Section 4. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1. The data was obtained by analyzing an R-11 calibration mixture 7 times during one day by one operator.

Section 5. Tables

The following tables are to be used in conjunction with Section 9 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography.

RT (Min.)	Comp.
2,20	23
2,65	13
3,03	152a
3,22	22
3,97	115
4,63	12
5,98	133a
6,35	21
7,00	30
7,41	114
8,83	11
9,79	123a
10,32	123
11,67	20
12,56	113
14,96	140a
15,64	10
19,79	1120
22,09	112

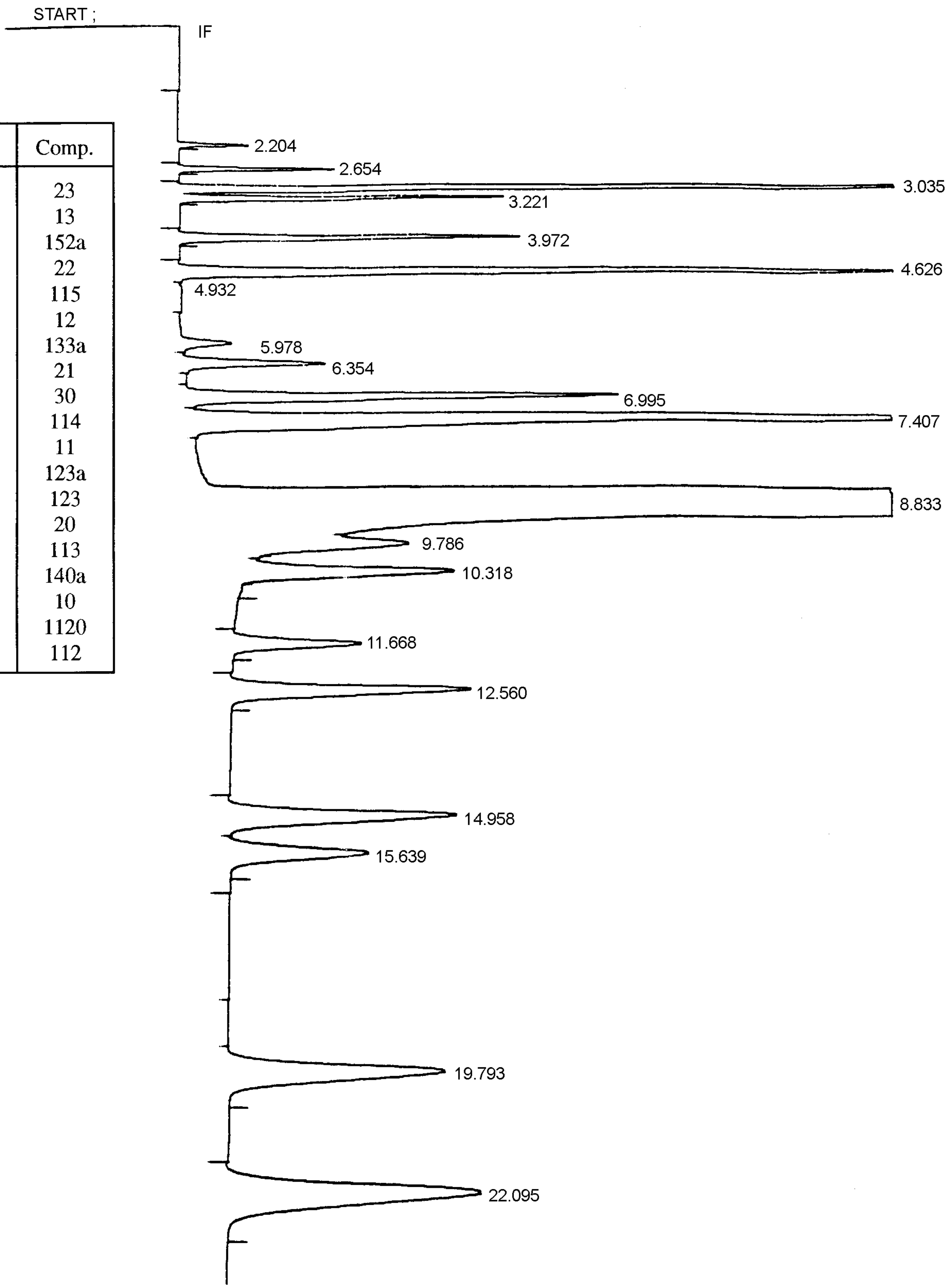


Figure 1. Gas Chromatogram of R-11

Table 1. Component Statistical Parameters

Component	Detection Limit, ppm	Concentration Investigated, ppm	Precision at 95% Confidence Limit, ppm	Relative Mean Error, %
23	2	15	0.37	-2.8
13	3	20	0.53	-3.1
152a	1	30	0.47	1.7
22	2	50	0.98	-0.8
115	2	30	0.80	0.7
12	2	60	1.10	1.1
133a	1	25	0.33	-2.5
21	2	30	0.67	1.2
30	2	25	0.33	-2.5
114	2	40	1.91	-2.7
123a	3	25	2.70	-4.8
123	2	50	1.33	3.3
20	2	25	0.73	0.7
113	2	60	2.31	2.2
10	2	25	1.70	-3.3
1120	2	25	1.77	1.8

Table 2. Primary Calibration Standard Components

Component	Mol. Wt.	Vol. Added, mL	µg Added (1)	ppm Added (2)	Total ppm Present (3)
23(4)	70	1.2	3436	15	
13(4)	105	1.0	4274	19	
152a(4)	66	2.5	6748	30	
22(4)	86	3.2	11321	50	
115(4)	136	1.2	6650	29	
12(4)	121	2.8	13845	61	
133a(4)	118	1.1	5332	24	
21(4)	103	1.6	6740	30	
30	85	5.0µL	6680	29	
114(4)	170	1.3	9061	40	
123a	153	5.0µL	7490	33	
123	153	10.0µL	14750	64	
20	120	5.0µL	7445	33	
113	188	10.0µL	15650	68	
10	154	10.0µL	15950	70	
1120	132	5.0µL	7278	32	

(1) If necessary, correct the µg added for the purity of the calibration component previously established.
(2) Values shown are for illustration; exact values are determined in Section 9.1 step f of the General Procedure.
(3) Column to be filled in (Section 9.1 step g of the General Procedure) after determining ppm present in stock R-11 (see Note 1 in Section 9.6 of the General Procedure).
(4) These impurities are gases at ambient room temperature; the others are liquids with low boiling points.

Table 3. Retention Time Data for Identified Impurities Not Normally Observed

Impurity	Retention Time (Min.)
32(1)	2.37
1114	4.10
C_3H_8	8.00

(1)Coelutes with R-23. To separate, attach 30.5 cm section of Porapak-T column to detector end of column and chromatograph (R-23 elutes first).

Part 3

R-12 GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Section 10 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography. This GC Method Data Sheet is for use with R-12.

Section 2. Limitations and Interferences

This method is applicable and calibrated for only those impurities found in Table 2 and Table 3. Other impurities that have been detected on occasion are listed (with retention times) in Table 4. This method will not detect any impurities that may elute within the comparatively large R-12 peak matrix.

Section 3. Gas Chromatographic Analysis Equipment and Conditions

Packed column: 7.32 m x 3.17 mm OD stainless steel, 1% SP-1000 on Carbopack B, 60/80 mesh, Supelco, Bellefonte, PA.

Detector	FID
Carrier Gas	30 mL He/Min.
Initial Column Temp.	40°C
Initial Hold	6 Min.
Program	10K/Min.
Final Column Temp.	160°C
Post Hold	18 Min.
Sample	0.50 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	150°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Section 4. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1. The data was obtained by analyzing an R-12 calibration mixture 7 times during one day by one operator.

Section 5. Tables

The following tables are to be used in conjunction with Section 10 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography.

RT (Min.)	Comp.
2,40	CH_4
3,19	23
3,92	C_2H_4
4,62	C_2H_6
5,36	13
6,27	143a
7,23	152a
7,49	40
7,97	134a
8,60	22
10,91	C_3H_6
11,19	115
12,92	12
13,61	142b
15,17	124
15,77	133a
16,31	21
16,69	ISO-C_4H_{10}
17,58	114
18,19	n-C_4H_{10}
18,94	2-C_4H_8
19,54	11
20,85	123a
21,63	123
24,18	MEK
24,65	113
25,34	2-butanol
26,34	C_5H_{12}

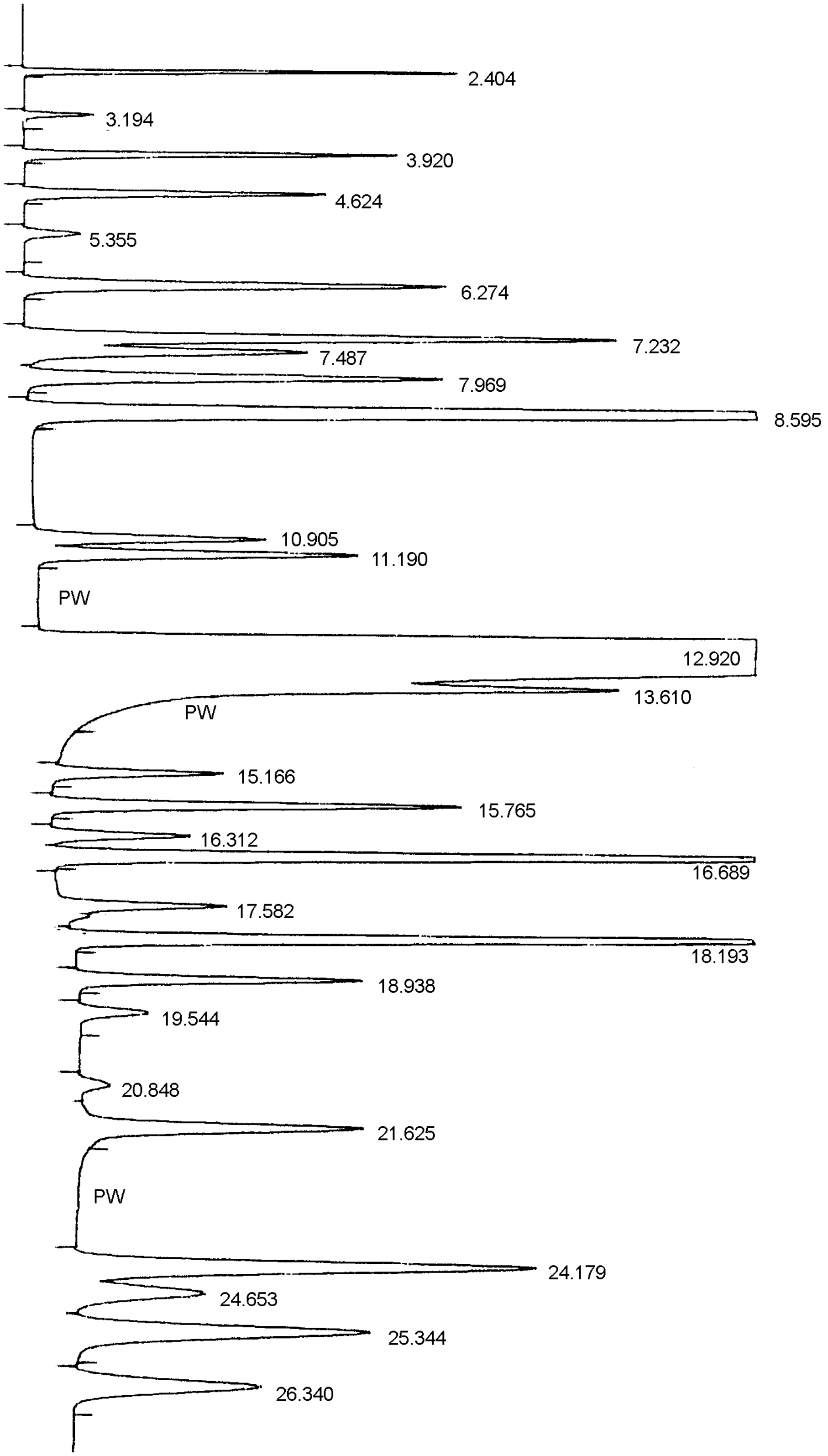


Figure 1. Gas Chromatogram of R-12

Table 1. Component Statistical Parameters

Component	Detection Limit, ppm	Concentration Investigated, ppm	Precision at 95% Confidence Limit, ppm	Relative Mean Error, %
Methane	0.5	5	0.07	4.00
23	2	25	0.54	-2.30
C_2H_4	0.5	5	0.13	-5.60
C_2H_6	0.5	5	0.10	-4.10
13	3	30	0.47	-3.80
143a	1	25	0.30	3.30
152a	1	30	0.63	1.67
140	1	20	0.37	2.30
134a	1	45	0.27	-3.30
22	2	65	1.75	2.73
C_3H_6	0.5	5	0.10	3.37
115	2	115	1.67	1.80
142b	1	20	0.23	-1.33
124	1	25	0.37	1.83
133a	1	35	0.23	1.83
21	2	50	0.83	1.80
isobutane	0.5	20	0.23	-2.77
114	2	50	0.83	2.03
n-butane	0.5	20	0.18	-3.33
2-butene-T	0.5	5	0.06	-3.80
11	4	40	0.87	1.05
123	2	35	1.05	-4.73
2-butanol	2	20	0.33	1.60
MEK	2	25	0.47	-2.33
113	2	30	0.87	-4.00
n-pentane	0.5	5	0.25	-3.73

Table 2. Primary Calibration Standard Components

Component	Mol. Wt.	Vol. Added, µL	µg Added (1)	ppm Added (2)	Total ppm present (3)
Methane	16	20	13.1	5	
23	70	22	63.0	23	
C_2H_4	28	12	13.7	5	
C_2H_6	30	11	13.5	5	
13	104	20	85.4	31	
143a	84	20	68.8	25	
152a	66	30	81.0	30	
40	50	28	57.8	21	
134a	102	30	125.1	46	
22	86	50	176.9	64	
C_3H_6	42	8	13.7	5	
115	154	50	315.9	115	
142b	100	15	61.7	22	
124	136	12	67.0	24	
133a	118	20	97.0	35	
21	103	32	134.7	49	
isobutane	58	25	59.3	22	
114	170	20	139.8	51	
n-butane	58	25	59.3	22	
2-butene-T	56	6	13.7	5	
11(4)	137		(5)	57	
123(4)	153		(5)	38	
MEK(4)	72		(5)	17	
113(4)	188		(5)	27	
2-butanol(4)	74		(5)	21	
n-pentane(4)	72		(5)	5	

Table 2 Notes

(1) If necessary, correct the µg added for the purity of the calibration component previously established.
(2) Values shown are for illustration; exact values are determined in Section 10, step p of the General Procedure.
(3) Column to be filled in (Section 10.1, step q of the General Procedure) after determining the ppm present in stock R-12 (see Note 1 in Section 10.7).
(4) These components are liquids at ambient laboratory temperature and are added to the 500 mL bulb as described in Section 10.1 of the General Procedure, steps k through n.
(5) From Section 10.1 step n.

Table 3. Liquid Impurities For Calibration Standard Preparation

Component	Vol. Added, mL	Density at 20°C	g
2-butanol	6.0	0.808	4.848
MEK	5.0	0.805	4.025
113	4.0	1.565	6.260
n-pentane	2.0	0.626	1.252
123	6.0	1.470	8.820
11	9.0	1.487	13.383

Table 4. Retention Time Data for Identified Impurities Not Normally Observed

Impurity	Retention Time (Min.)
32(1)	3.45
1114	6.00
C_3H_8	11.60
30	16.93
2, 2-Dimethylpropane	19.80
Isopentane	24.30

(1) Coelutes with R-23. To separate, attach 1 ft. (30.5 cm) section of Porapak-T column to detector end of column and re-chromatograph (23 elutes first).

Part 4

R-13 GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Section 11 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography. This GC Method Data Sheet is for use with R-13.

Section 2. Limitations and Interferences

This method is applicable and calibrated for only those impurities found in Table 2. This method will not detect any impurities that may elute within the comparatively large R-13 peak matrix.

Section 3. Gas Chromatographic Analysis Equipment and Conditions

Packed column: 7.32 m x 3.17 mm OD stainless steel, 1% SP-1000 on Carbopack B, 60/80 mesh, Supelco, Bellefonte, PA.

Detector	TCD, Low Sensitivity*
Carrier Gas	30 mL He/Min.
Initial Column Temp.	40°C
Initial Hold	6 Min.
Program	10K/Min.
Final Column Temp.	160°C
Post Hold	4 Min.
Sample	2.0 mL (gas syringe)
Detector Temp.	200°C
Inj. Port Temp.	150°C
Max Safe Column Temp.	225°C (for conditioning purposes)

*See Note 5 in Section 11.6.

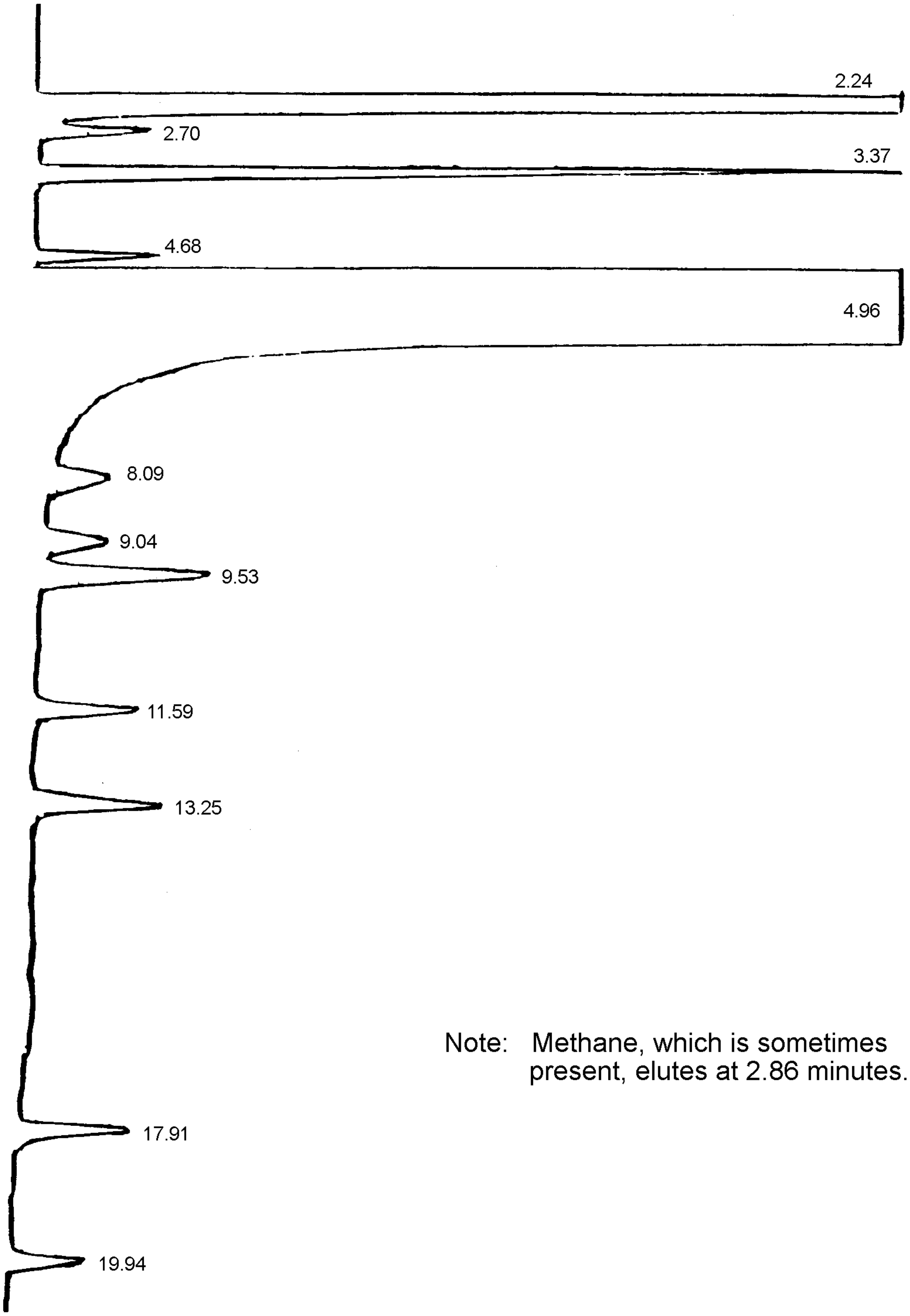
Section 4. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1. The data was obtained by analyzing an R-13 calibration mixture 7 times during one day by one operator.

Section 5. Tables

The following tables are to be used in conjunction with Section 11 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography.

RT (Min.)	Comp.
2,24	Air
2,70	14
3,37	23
4,68	116
4,96	13
8,09	125
9,04	22
9,53	31
11,59	115
13,25	12
17,91	114
19,94	11



Note: Methane, which is sometimes present, elutes at 2.86 minutes.

Figure 1. Gas Chromatogram of R-13

Table 1. Component Statistical Parameters

Component	Detection Limit, ppm	Concentration Investigated, ppm	Precision at 95% Confidence Limit, ppm	Relative Mean Error, %
14	12	300	7.2	1.7
23	10	200	2.7	-1.0
116	14	250	3.1	1.3
125	12	150	6.6	-2.0
22	10	175	5.3	2.3
31	10	100	2.6	2.0
115	12	200	4.7	-2.0
12	10	200	3.6	2.5
114	12	100	6.3	4.0
11	12	100	3.1	-2.0

Table 2. Primary Calibration Standard Components

Component	Mol. Wt.	Vol. Added, mL	grams Added (1)	ppm Added (2)	Total ppm Present (3)
14	88	10.0	0.0340	227	
23	70	10.0	0.0286	191	
116	138	5.0	0.0282	188	
125	120	5.0	0.0245	163	
22	86.5	10.0	0.0354	236	
31	68	5.0	0.0139	93	
115	154.5	5.0	0.0316	211	
12	121	8.0	0.0396	264	
114	171	3.0	0.0210	140	
11 (4)	137.4	4.0	0.0225	150	

(1) If necessary, correct the grams added for the purity of the calibration component previously established.
(2) Values shown are for illustration; exact values are determined in Section 11.1 step o of the General Procedure.
(3) Column to be filled in (Section 11.1, step p of the General Procedure) after determining ppm present in stock R-13 (see Note 1 in Section 11.6).
(4) Added by warming an uncapped vial of the liquid component to enrich the headspace vapor, capping, cooling, then removing headspace vapor via a gas syringe.

Part 5

R-22 GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Section 10 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-22.

Section 2. Limitations and Interferences

This method is applicable and calibrated for only those impurities found in Table 2 and Table 3. Other impurities that have been detected on occasion are listed (with retention times) in Table 4. This method will not detect any impurities that may elute within the comparatively large R-22 peak matrix.

Section 3. Gas Chromatographic Analysis Equipment and Conditions

3.1 *Chromatographic Equipment and Conditions, Packed Column*

Packed column: 7.32 m x 3.17 mm OD stainless steel, 1% SP-1000 on Carbopack B, 60/80 mesh, Supelco, Bellefonte, PA.

Detector	FID
Carrier Gas	30 mL He/Min.
Initial Column Temp.	40°C
Initial Hold	6 Min.
Program	10K/Min.
Final Column Temp.	160°C
Post Hold	18 Min.
Sample	0.50 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	150°C
Max Safe Column Temp.	225°C (for conditioning purposes)

3.2 *Chromatographic Equipment and Conditions, Capillary Column*

Capillary Column: 120m (or 2 60m) DB-1301, .25mm,
1µ, J&W Scientific Co.,Folsom, CA.

Detector	FID
Carrier Gas	approx. 1mL He/Min
Injection Port Temp	150°C
Detector Temp.	250°C
Sample	1.0 mL
Max. Safe Column Temp.	280°C
Initial Col. Temp	40°C
Initial Hold	10 Min.
Program	8K/Min.
Final Column Temp.	50°C
Post Hold	18 Min.
Split Ratio	30:1
Pressure	240 kPa

Section 4. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1. The data was obtained by analyzing an R-22 calibration mixture 7 times during one day by one operator.

Section 5. Tables

The following tables are to be used in conjunction with Section 10 of the General Procedure.

RT (Min.)	Comp.
2,40	CH_4
3,19	23
3,90	C_2H_4
4,60	C_2H_6
5,33	13
6,25	143a
7,29	152a
7,66	40
7,96	134a
8,29	22
10,91	C_3H_6
11,20	115
12,89	12
13,58	142b
15,16	124
15,75	133a
16,30	21
16,68	iso C_4H_{10}
17,57	114
18,18	C_4H_{10}
18,92	2-C_4H_8
19,52	11
20,82	123a
21,60	123
24,62	113

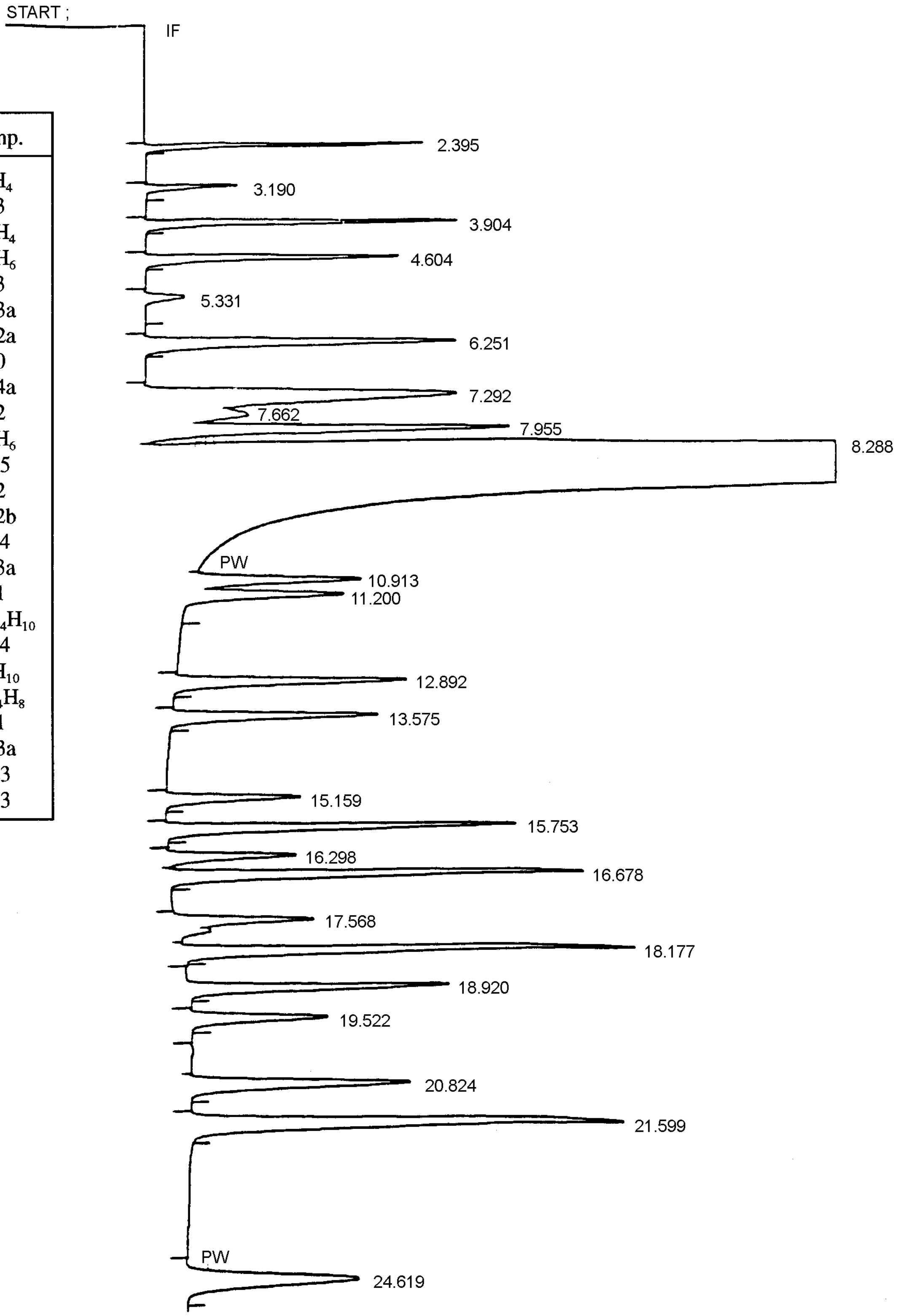


Figure 1. Gas Chromatogram of R-22

RT (Min.)	Comp.
12,81	22
13,91	isobutane
14,66	31
14,85	n-butane

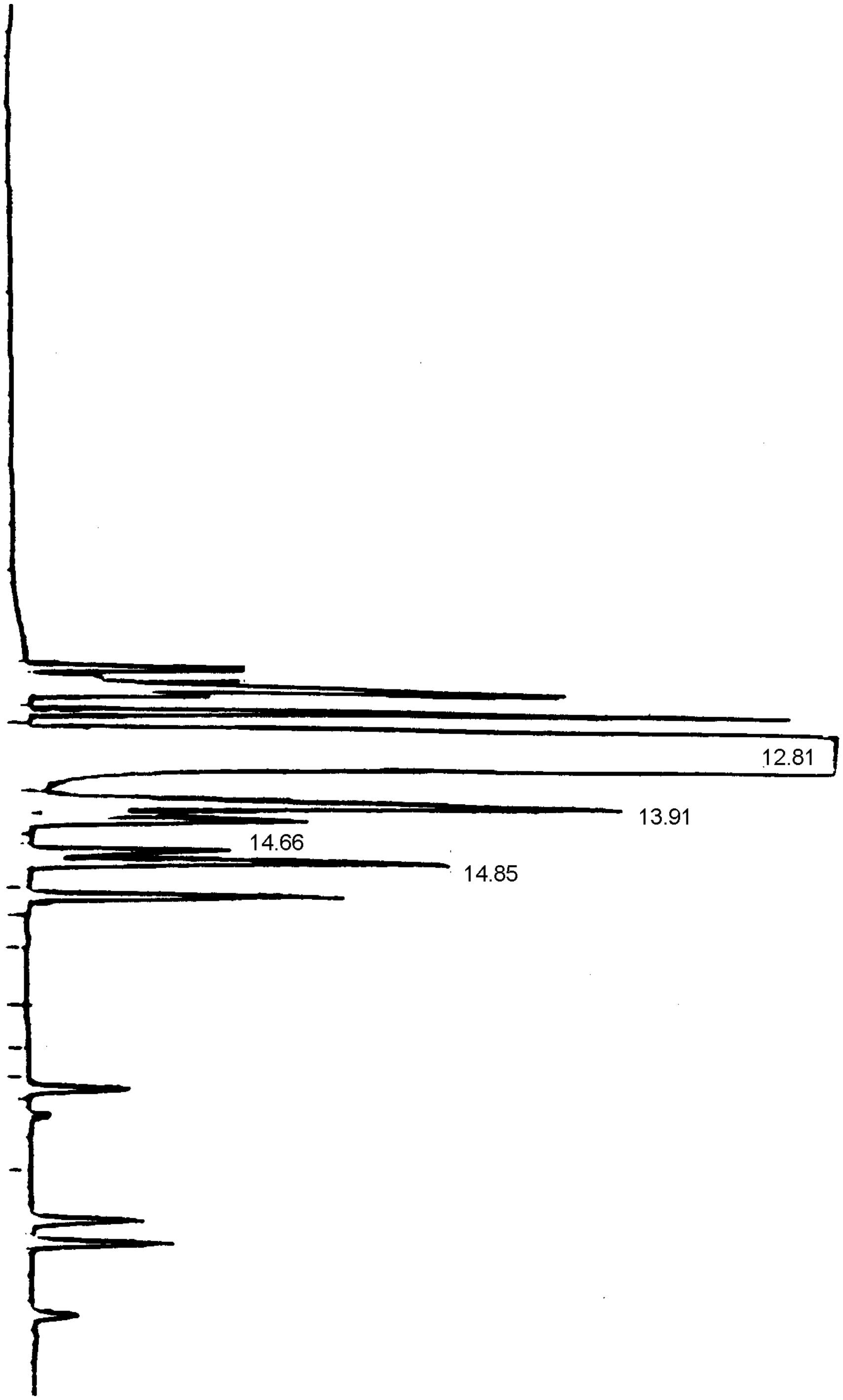


Figure 2. Capillary Column Gas Chromatogram of R-22 for Determination of R-31 Impurity

Table 1. Component Statistical Parameters

Component	Detection Limit, ppm	Concentration Investigated, ppm	Precision at 95% Confidence Limit, ppm	Relative Mean Error, %
CH_4	0.5	5	0.10	4.7
23	2	20	0.88	1.7
C_2H_4	0.5	5	0.08	-6.2
C_2H_6	0.5	5	0.05	-5.77
13	3	30	0.47	-3.33
143a	1	25	0.67	1.7
152a	1	40	0.83	-0.67
40	1	25	0.87	-2.45
134a	1	50	0.50	-1.07
31(1)	2	40	3.33	-4.67
C_3H_6	0.5	5	0.13	5.7
115	2	80	3.05	-0.8
12	2	100	2.88	2.4
142b	1	20	0.17	1.07
124	1	30	0.45	2.07
133a	1	35	0.83	1.87
21	2	60	1.33	-3.8
isobutane	0.5	10	0.13	-4.3
114	2	35	1.33	1.8
n-butane	0.5	10	0.13	-4.3
2-butene-T	0.5	5	0.05	-5.3
11	4	40	1.67	2.87
123a	2	15	1.33	-3.67
123	2	30	1.67	-2.33
113	2	25	1.33	1.87

(1) In absence of n-butane.

Table 2. Primary Calibration Standard Components

Component	Mol. Wt.	Vol. Added, µL	µg Added (1)	ppm Added (2)	Total ppm Present (3)
Methane	16	15	9.8	5	
23	70	15	43.0	22	
C_2H_4	28	10	11.4	6	
C_2H_6	30	10	12.3	6	
13	104	15	64.0	33	
143a	84	15	51.6	26	
152a	66	30	81.0	41	
40	50	25	51.6	26	
134a	102	25	104.3	53	
31	68	28	78.5	40	
C_3H_6	42	5	8.6	4	
115	154	25	158.0	80	
12	121	40	197.8	100	
142b	100	10	41.1	21	
124	136	10	55.8	28	
133a	118	15	72.8	37	
21	103	30	126.3	64	
isobutane	58	10	23.7	12	
114	170	10	69.9	36	
n-butane	58	10	23.7	12	
2-butene-T	56	5	11.4	6	
11(4)	137		(5)	133	
123a(4)	153		(5)	36	
123(4)	153		(5)	70	
113(4)	188		(5)	47	

See Table 2 Notes below.

Table 2 Notes

(1) If necessary, correct the μg added for the purity of the calibration component previously established.
(2) Values shown are for illustration; exact values are determined in Section 10, step p of the General Procedure.
(3) Column to be filled in (Section 10, step q of the General Procedure) after determining the ppm present in stock R-22 (see Note 1 in Section 10).
(4) These components are liquids at ambient laboratory temperature and are added to the 500-mL bulb as described in Section 10.1 of the General Procedure, steps k through n.
(5) From Section 10.1 step n.

Table 3. Liquid Impurities for Calibration Standard Preparation

Component	Volume Added, mL	Density At 20°C	g
113	5	1.565	7.825
123	8	1.470	11.76
123a	4	1.492	5.968
11	15	1.487	22.305

Table 4. Additional Impurities Observed in R-22

Impurity	Packed Column Retention Time (Min.)
32(1)	2.7
1114	6.0
Propane	11.7
124a	14.8
30	17.0
CCl_4	26.8
pentane	27.0

(1) Coelutes with R-23 on packed column; to separate, attach 1 foot (30.5 cm) section of Porapak-T column to detector end of column and re-chromatograph (23 elutes first).

Part 6

R-23 GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Section 11 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-23.

Section 2. Limitations and Interferences

This method is applicable and calibrated for only those impurities found in Table 2. This method will not detect any impurities that may elute within the comparatively large R-23 peak matrix.

Section 3. Gas Chromatographic Analysis Equipment and Conditions

Packed column: 6.09 m x 3.17 mm OD stainless steel, Porapak-T, 80/100 mesh, Supelco, Bellefonte, PA.

Detector	TCD, Low Sensitivity*
Carrier Gas	15 mL He/Min.
Initial Column Temp.	40°C
Initial Hold	3 Min.
Program	18 K/Min.
Final Column Temp.	175°C
Post Hold	10 Min.
Sample	2.0 mL (gas syringe)
Detector Temp.	200°C
Inj. Port Temp.	150°C
Max Safe Column Temp.	190°C (for conditioning purposes)

* See Note 5 in Section 11.6 of the General Procedure.

Section 4. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1. The data was obtained by analyzing an R-23 calibration mixture 7 times during one day by one operator.

Section 5. Tables

The following tables are to be used in conjunction with Section 11 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography.

RT (Min.)	Comp.
1,40	Air
1,85	40
3,64	116
4,93	CO_2
5,35	13
5,82	23
7,90	32
8,24	13B1
10,02	12
10,62	22
12,28	31
12,77	H_2O

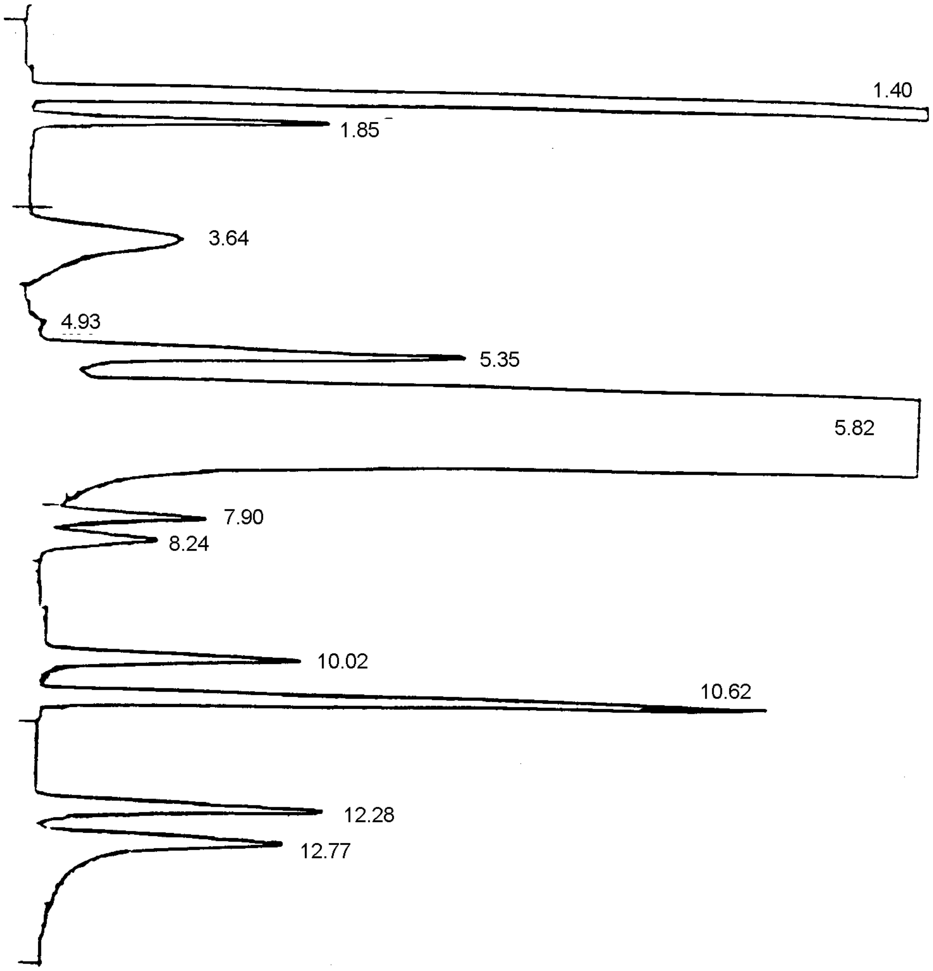


Figure 1. Gas Chromatogram of R-23

Table 1. Component Statistical Parameters

Component	Detection Limit, ppm	Concentration Investigated, ppm	Precision at 95% Confidence Limit, ppm	Relative Mean Error, %
14	10	200	1.77	-0.40
116	15	350	5.80	-0.85
13	10	400	5.10	0.60
32	5	150	1.93	1.40
13B1	10	120	5.87	-1.05
12	5	160	3.67	0.88
22	5	450	3.90	0.32
31	5	175	1.60	-1.37

Table 2. Calibration Standard Components

Component	Mol. Wt.	Vol. Added, mL	µg Added (1)	ppm Added (2)	Total ppm Present (3)
14	88	10.0	0.0360	180	
116	138	10.0	0.0564	282	
13	104	20.0	0.0851	426	
22	86.5	20.0	0.0709	355	
32	52	10.0	0.0213	107	
31	68	10.0	0.0278	139	
12	121	6.0	0.0297	149	
13B1	149	5.0	0.0305	153	

(1) If necessary, correct the grams added for the purity of the calibration component previously established.
(2) Values shown are for illustration; exact values are determined at Section 11.1 step o of the General Procedure.
(3) Column to be filled in (Section 11.1, step p of the General Procedure) after determining the ppm present in stock R-23 (see Note 1 of Section 11.6 of the General Procedure).

Part 7

R-32 GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Section 10 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-32.

Section 2. Limitations and Interferences

This method is applicable and calibrated for only those impurities found in Table 2. Other impurities that have been detected on occasion are listed (with retention times) in Table 4. This method will not detect any impurities that may elute within the comparatively large R-32 peak matrix.

Section 3. Gas Chromatographic Analysis Equipment and Conditions

3.1 *Chromatographic Equipment and Conditions, Packed Column*

Packed column: 4.88 m x 3.17 mm OD stainless steel, 1% SP-1000 on Carbopack B, 60/80 mesh, Supelco, Bellefonte, PA.

Detector	FID
Carrier Gas	20 mL He/Min.
Initial Column Temp.	45°C
Initial Hold	8 Min.
Program	8 K/Min.
Final Column Temp.	150°C
Post Hold	15 Min.
Sample	1.0 mL (gas syringe)
Detector Temp.	200°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	225°C (for conditioning purposes)

3.2 *Chromatographic Equipment and Conditions, Capillary Column*

Capillary Column: 120m (or 2-60m) DB-1301, 0.25mm, 1µ, J&W Scientific Co.,Folsom, CA.

Detector	FID
Carrier Gas	approx. 1mL He/Min
Injection Port Temp	150°C
Detector Temp.	250°C
Sample	1.5 mL
Max. Safe Column Temp.	280°C
Initial Col. Temp	-28°C
Initial Hold	10 Min.
Program	5K/Min.
Final Column Temp.	40°C
Post Hold	5 Min.
Split Ratio	30:1
Pressure	200 kPa
Makeup Gas	30 mL He/Min.

See Note 5 of Section 10.7 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography.

Section 4. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1. The data was obtained by analyzing an R-32 calibration mixture 7 times during one day by one operator.

Section 5. Tables

The following tables are to be used in conjunction with Section 10 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography.

RT (Min.)	Comp.
8,70	41
9,16	32
17,20	143a
19,70	125/40
22,40	31
25,90	115
29,06	12
32,70	124
38,10	114a

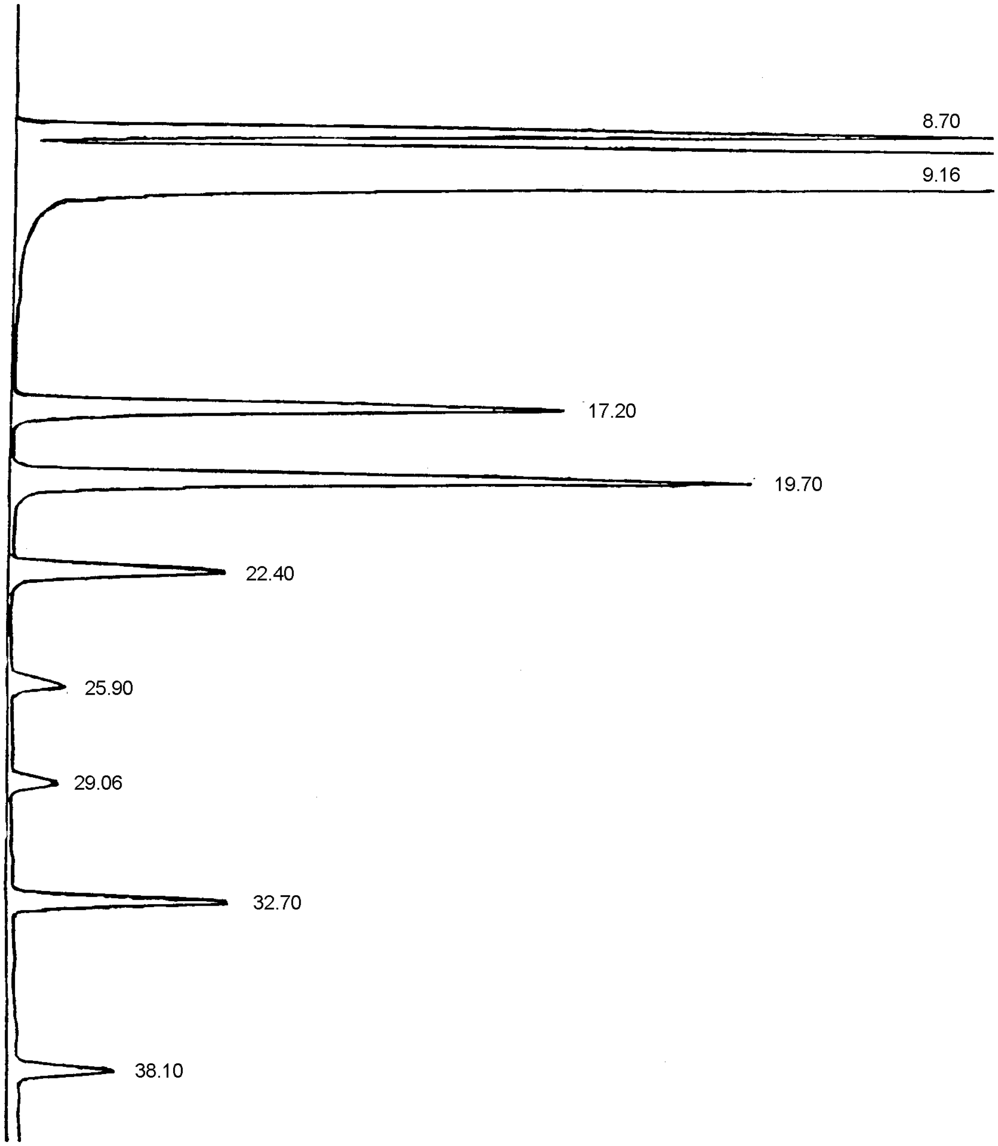


Figure 1. Packed Column Gas Chromatogram of R-32

RT (Min.)	Comp.
9,55	23
9,86	143a
10,30	32
21,88	40

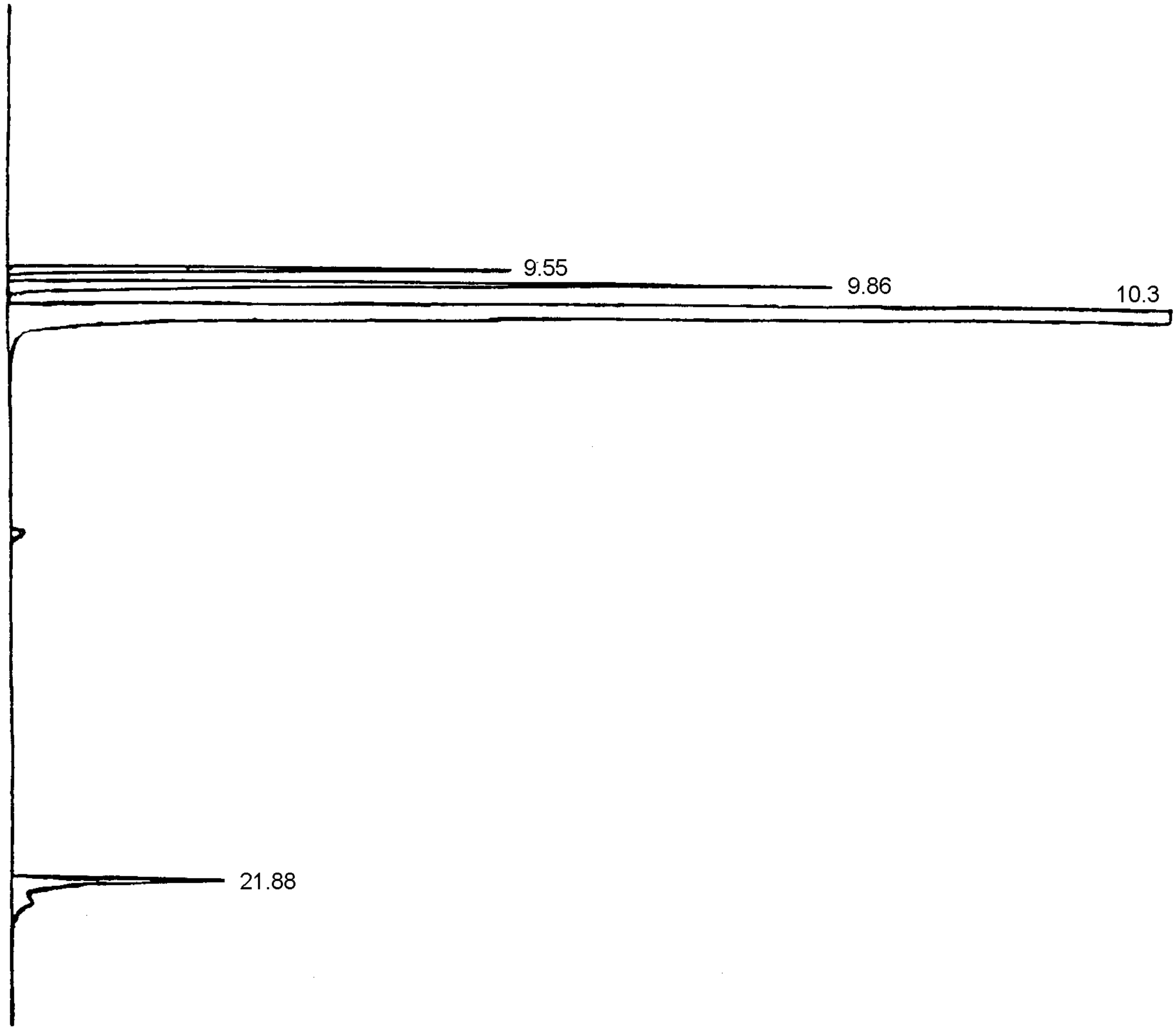


Figure 2. Capillary Gas Chromatogram of R-32

Table 1. Component Statistical Parameters

Component	Detection Limit, ppm	Concentration Investigated, ppm	Precision at 95% Confidence Limit, ppm	Relative Mean Error, %
41	3	100	4.33	-2.1
143a	1	100	0.80	-1.1
125	1	100	4.10	-0.8
40	1	100	2.67	1.4
31	1	100	1.45	1.3
115	2	100	1.88	2.1
12	2	100	2.33	-1.0
124	1	100	1.01	0.7
23	2	100	1.73	-3.0
114a	2	100	3.53	2.8

Table 2. Primary Calibration Standard Components

Component	Mol. Wt.	Vol. Added, µL	µg Added (1)	ppm Added (2)	Total ppm Present (3)
41	34	75	104.3	88.4	
143a	84	30	103.2	87.4	
125	120	25	122.9	104.0	
40	50	50	103.3	87.5	
31	68	40	112.1	95.0	
115	154	20	126.4	107.0	
12	121	25	123.6	104.7	
124	136	20	111.8	94.8	
23	70	40	114.6	97.1	
114a	171	15	104.8	88.8	

(1) If necessary, correct the µg added for the purity of the calibration component previously established.
(2) Values shown are for illustration; exact values are determined at step p.
(3) Column to be filled in (step q) after determining ppm present in stock R-32 (see Note 1 in Section 10.7 in the GC Method Data Sheet).

Table 3. Liquid Impurities for Calibration Standard Preparation

Component	Volume Added, mL	Density At 20°C	g
None Added			

Table 4. Additional Impurities Observed in R-32

The following impurities have been observed occasionally in samples of R-32 on the packed column:

Impurity	Packed Col. Retention Time (Min.)
13	15.0
22	24.2
142b	30.0
133a	34.8
21	35.3
11(1)	40
123(1)	42.3

(1) Need to extend final hold time if presence of these impurities is suspected.

Part 8

R-113 GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Section 9 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-113.

Section 2. Limitations and Interferences

This method is applicable and calibrated for only those impurities found in Table 2. This method will not detect any impurities that may elute within the comparatively large R-113 peak matrix.

Section 3. Gas Chromatographic Analysis Equipment and Conditions

Capillary column: 105m x 0.25 mm, 1.0µ, Rtx-1301, Restek Corp., 110 Benner Circle, Bellefonte, PA.

Detector	FID
Carrier Gas	approx. 1 mL He/Min.
Initial Column Temp.	35°C
Initial Hold	10 Min.
Program	8 K/Min.
Final Column Temp.	160°C
Post Hold	8 Min.
Sample	2 µL (liquid syringe)
Detector Temp.	250°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	280°C (for conditioning purposes)
Split Ratio	30:1
Aux. Flow	30 mL/Min.
Pressure	200 kPa

Section 4. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1. The data was obtained by analyzing an R-113 calibration mixture 7 times during one day by one operator.

Section 5. Tables

The following tables are to be used in conjunction with Section 9 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography.

RT (Min.)	Comp.
9,60	115
10,24	1113
10,41	12
10,54	22
11,10	114
12,41	216ba
12,65	133a
13,72	1112a
14,51	318mbb
15,06	11
16,00	C-316bb
16,35	123a
16,59	123
17,30	113
18,51	225
23,26	10
23,49	122
24,68	112a
24,81	112
25,06	1120
28,76	1110

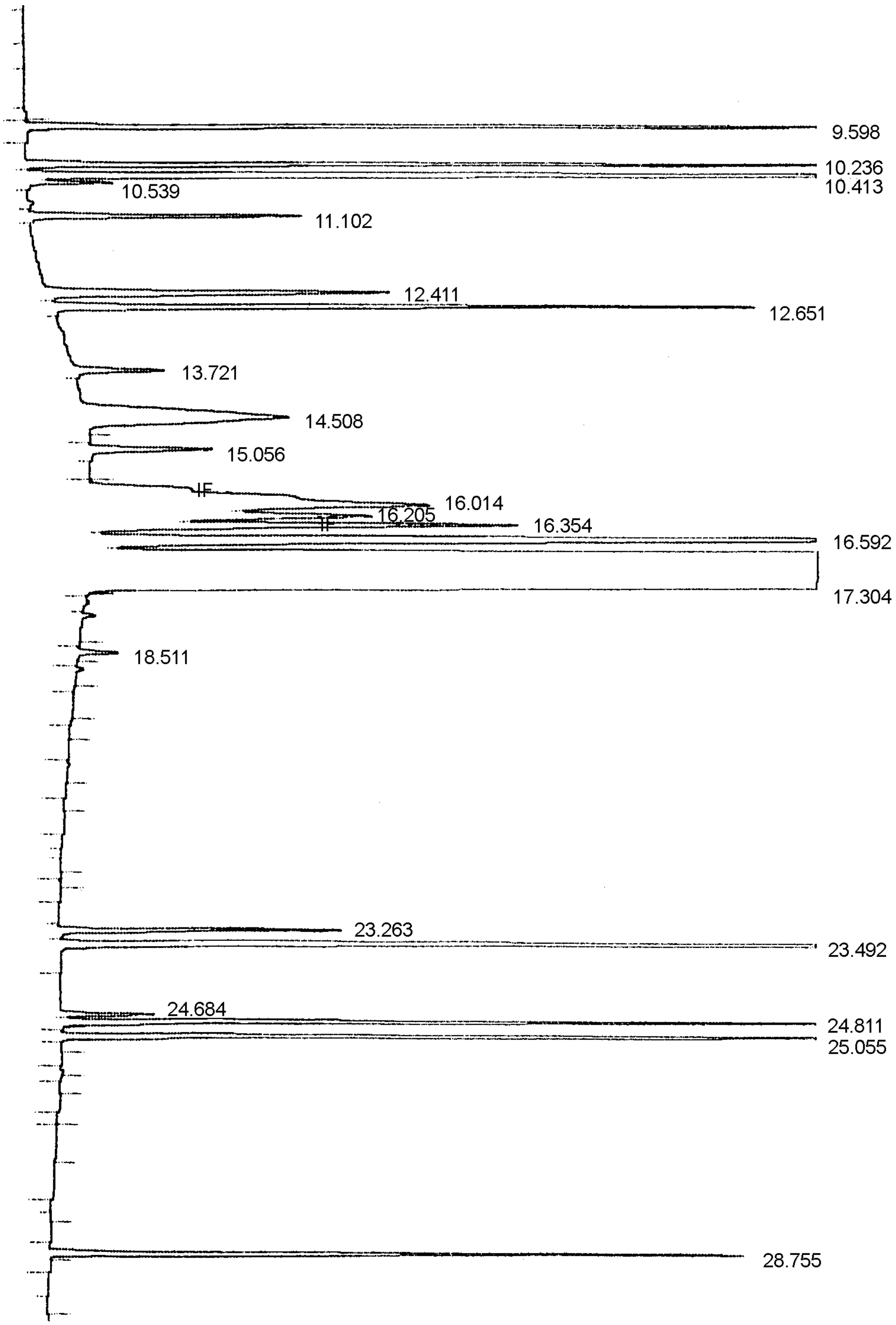


Figure 1. Gas Chromatogram of R-113

Table 1. Component Statistical Parameters

Component	Detection Limit, ppm	Concentration Investigated, ppm	Precision at 95% Confidence Limit, ppm	Relative Mean Error, %
115	5	50	1.2	1.3
1113	2	60	2.3	-0.7
12	10	70	0.8	-1.2
22	5	70	0.8	1.0
114	5	40	0.7	0.8
216ba	2	50	2.5	-1.9
133a	1	50	0.7	-2.3
1112a	2	20	0.3	-3.3
11	15	120	4.1	0.8
C-316bb	2	30	6.8	-1.1
123a	2	50	1.3	-2.5
123	2	50	1.5	-1.1
225da	1	30	0.9	-2.1
318mbb	2	30	0.8	-0.7
122	2	80	2.3	0.4
10	5	100	4.7	2.6
112	3	75	2.5	-1.1
1120	2	30	1.4	0.3
1110	2	30	1.7	0.8

Table 2. Primary Calibration Standard Components

Component	Mol. Wt.	Vol. Added, mL	µg Added (1)	ppm Added (2)	Total ppm Present (3)
115(4)	154	2.0	12653	53	
1113(4)	116	3.0	14294	60	
12(4)	121	3.5	17336	72	
22(4)	86	5.0	17690	74	
114(4)	171	1.5	10502	44	
216ba	221	8 µL	12722	53	
133a(4)	118	2.5	12137	50	
1112a(4)	133	0.8	4349	18	
11	137	20 µL	29000	121	
123a	153	8 µL	11984	50	
123	153	8 µL	11984	50	
225da	203	5 µL	7782	32	
318mbb	271	5 µL	8400	35	
122	169	5 µL	7723	32	
C-316bb	233	5 µL	7650	32	
112	204	--	20000	83	
TCE	131	5 µL	7278	30	
PCE	166	5 µL	8156	34	
10	154	15 µL	23925	100	

(1) If necessary, correct the µg added for the purity of the calibration component previously established.
(2) Values shown are for illustration; exact values are determined at Section 9.1 step f of the General Procedure.
(3) Column to be filled in (Section 9.1, step g of the General Procedure) after determining ppm present in stock R-113 (see Note 1 in Section 9.6 of the General Procedure).
(4) These impurities are gases at ambient room temperature, the others are liquids with low boiling points. For 1112a, warm the vial or cylinder and sample the headspace vapor.

Part 9

R-114 GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Section 10 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-114.

Section 2. Limitations and Interferences

This method is applicable and calibrated for only those impurities found in Table 2 and Table 3. This method will not detect any impurities that may elute within the comparatively large R-114 peak matrix.

Section 3. Gas Chromatographic Analysis Equipment and Conditions

Packed column: 7.32 m x 3.17 mm OD stainless steel, 1% SP-1000 on Carbopack B, 60/80 mesh, Supelco, Bellefonte, PA.

Detector	FID
Carrier Gas	30 mL He/Min.
Initial Column Temp.	40°C
Initial Hold	6 Min.
Program	10K/Min.
Final Column Temp.	175°C
Post Hold	18 Min.
Sample	0.50 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Section 4. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1. The data was obtained by analyzing an R-114 calibration mixture 7 times during one day by one operator.

Section 5. Tables

The following tables are to be used in conjunction with Section 10 of the General Procedure.

RT (Min.)	Comp.
2,67	χ
3,45	23
5,68	13
7,61	152a
8,29	134a
8,94	22
11,46	115
13,11	12
14,47	124a
15,17	124
15,74	133a
16,45	217ba
17,03	217ca
17,65	114
17,99	114a
19,75	11
20,50	1112a
20,72	123a
21,32	123
23,98	113
24,40	113a
29,64	122
32,34	1120

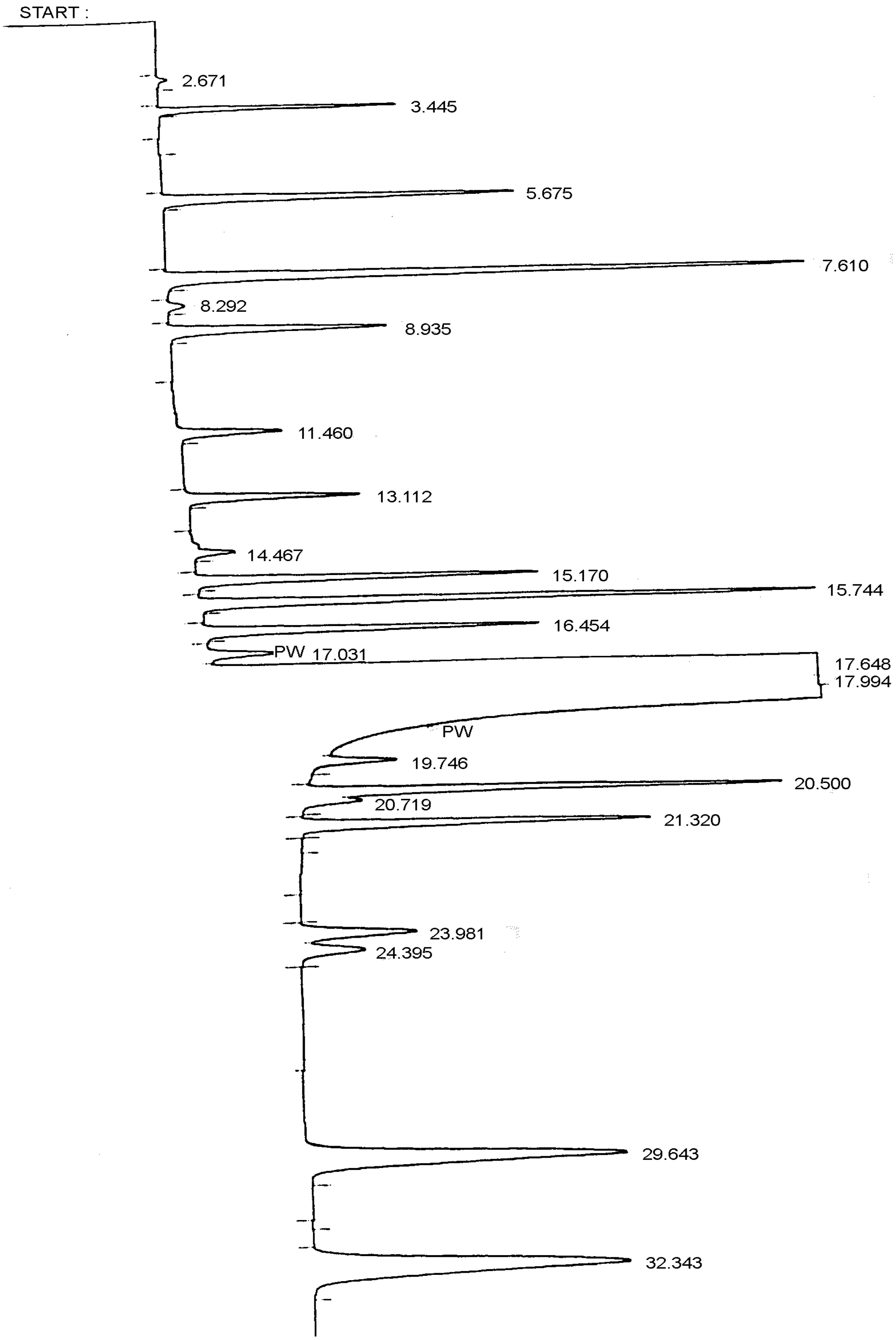


Figure 1. Gas Chromatogram of R-114

Table 1. Component Statistical Parameters

Component	Detection Limit, ppm	Concentration Investigated, ppm	Precision at 95% Confidence Limit, ppm	Relative Mean Error, %
23	2	15	0.28	-3.2
13	3	25	0.44	-3.8
152a	1	25	0.40	0.8
22	2	60	0.67	1.7
115	2	100	1.67	1.1
12	2	60	0.91	-1.1
124a	1	15	0.75	-2.3
124	1	30	0.50	1.6
133a	1	50	0.50	1.1
217ca	2	20	0.67	2.7
217ba	2	20	1.33	-3.4
11	4	45	0.67	1.7
123a	2	25	0.50	-2.7
123	2	65	0.77	-3.4
113	2	50	1.1	-3.7
113a	2	30	1.23	-2.7
122	2	30	0.67	-1.3
TCE	2	30	0.33	-2.3

Table 2. Primary Calibration Standard Components

Component	Mol. Wt.	Vol. Added, µL	µg Added (1)	ppm Added (2)	Total ppm Present (3)
23	70	20	57.2	15	
13	104	20	85.6	22	
152a	66	35	94.5	26	
22	86	65	230.0	60	
115	154	60	379.2	98	
12	121	50	247.2	64	
124a	136	10	55.8	14	
124	136	20	111.7	29	
133a	118	40	193.8	50	
217ca	204	10	83.6	22	
217ba(5)	204	10	83.6	22	
11(4)	137		(6)	75	
123a(4)	153		(6)	19	
123(4)	153		(6)	37	
113a(4)	188		(6)	40	
113(4)	188		(6)	60	
122(4)	169		(6)	39	
TCE(4)	131		(6)	37	

(1) If necessary, correct the µg added for the purity of the calibration component previously established.
(2) Values shown are for illustration; exact values are determined at step p.
(3) Column to be filled in (step q) after determining ppm present in stock R-114 (see Note 1 in Section 10.7 of the General Procedure).
(4) These components are liquids at ambient laboratory temperature and are added to the 500 mL bulb as described in Section 10.1 of the General Procedure (steps k through n).
(5) Note that 217ba often contains 15 to 20% of the 217ca isomer.
(6) From step n.

Table 3. Liquid Impurities For Calibration Standard Preparation

Component	Vol. Added (mL)	Density at 20°C	g
TCE	4.0	1.456	5.824
122	4.0	1.544	6.176
113	6.0	1.565	9.390
113a	4.0	1.579	6.316
123a	2.0	1.492	2.984
123	4.0	1.470	5.880
11	8.0	1.487	11.896

Part 10

R-123 GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Section 9 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-123.

Section 2. Limitations and Interferences

This method is applicable and calibrated for only those impurities found in Table 2. Other impurities that have been detected on occasion are listed (with retention times) in Table 3. This method will not detect any impurities that may elute within the comparatively large R-123 peak matrix. The packed column is necessary because 123a, 113 and 113a all nearly coelute on the capillary column. The other impurities do not interfere with these isomers on the packed column.

Section 3. Gas Chromatographic Analysis Equipment and Conditions

3.1 *Chromatographic Equipment and Conditions, Packed Column*

Packed column: 7.32 m x 3.17 mm OD stainless steel, 1% SP-1000 on Carbopack B, 60/80 mesh, Supelco, Bellefonte, PA.

Detector	FID
Carrier Gas	40 mL He/Min.
Column Temp.	125°C (isothermal)
Sample	2.0 µL (liquid syringe)
Detector Temp.	250°C
Inj. Port Temp.	150°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Externally cool the syringe to 10°C before sampling.

3.2 *Chromatographic Equipment and Conditions, Capillary Column*

Capillary Column: 120m (Connect the below two columns together with the DB-1701 column end attached to the injection port):

1. 60m DB-1701, 0.25mm, 1µ, J&W Scientific Co., Folsom CA.

2. 60m SPB-5, 0.32 mm, 1µ, Supelco, Bellefonte, PA.

Detector	FID
Carrier Gas	approx. 1mL He/Min
Injection Port Temp	150°C
Detector Temp.	250°C
Sample	2µl
Max. Safe Column Temp.	280°C
Initial Col. Temp	15°C **???**
Initial Hold	10 Min.
Program	7K/Min.
Final Column Temp.	60°C
Post Hold	22 Min.
Split Ratio	50:1
Pressure	200 kPa
Makeup Gas	20 mL He/Min.

Section 4. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1. The data was obtained by analyzing an R-123 calibration mixture 7 times during one day by one operator.

Section 5. Tables

The following tables are to be used in conjunction with Section 9 of the General Procedure.

RT (Min.)	Comp.
10,07	1113
10,29	12
10,71	22
11,22	114
11,43	1317-T
11,83	1317-C
12,80	31
13,12	216
13,60	1326
14,10	133a
14,54	328lcc
14,88	114B1
15,15	1112a
15,87	1112
17,26	11
20,40	123b
20,95	113/123a
21,22	113a/123
24,81	30
30,58	1111

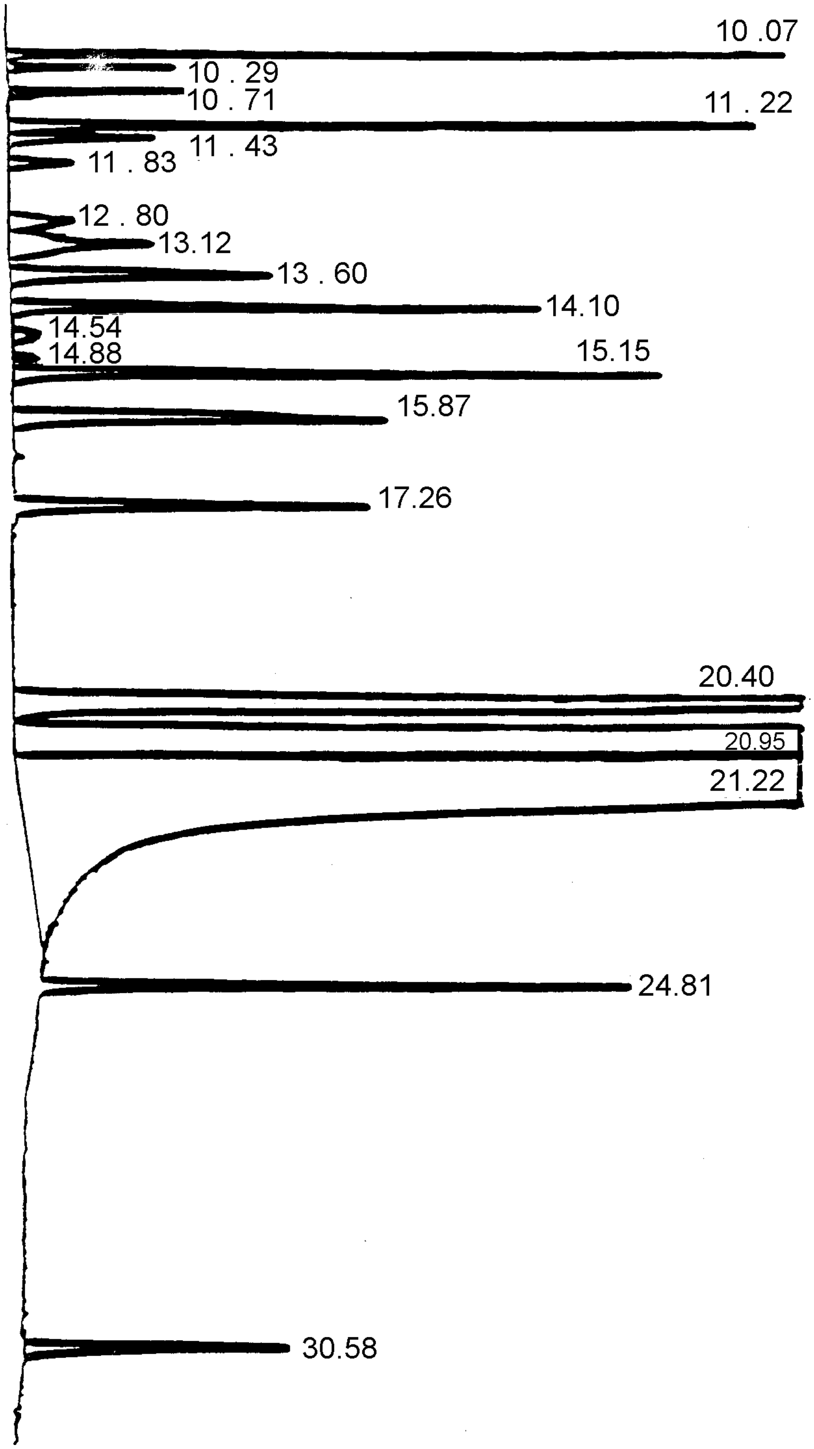
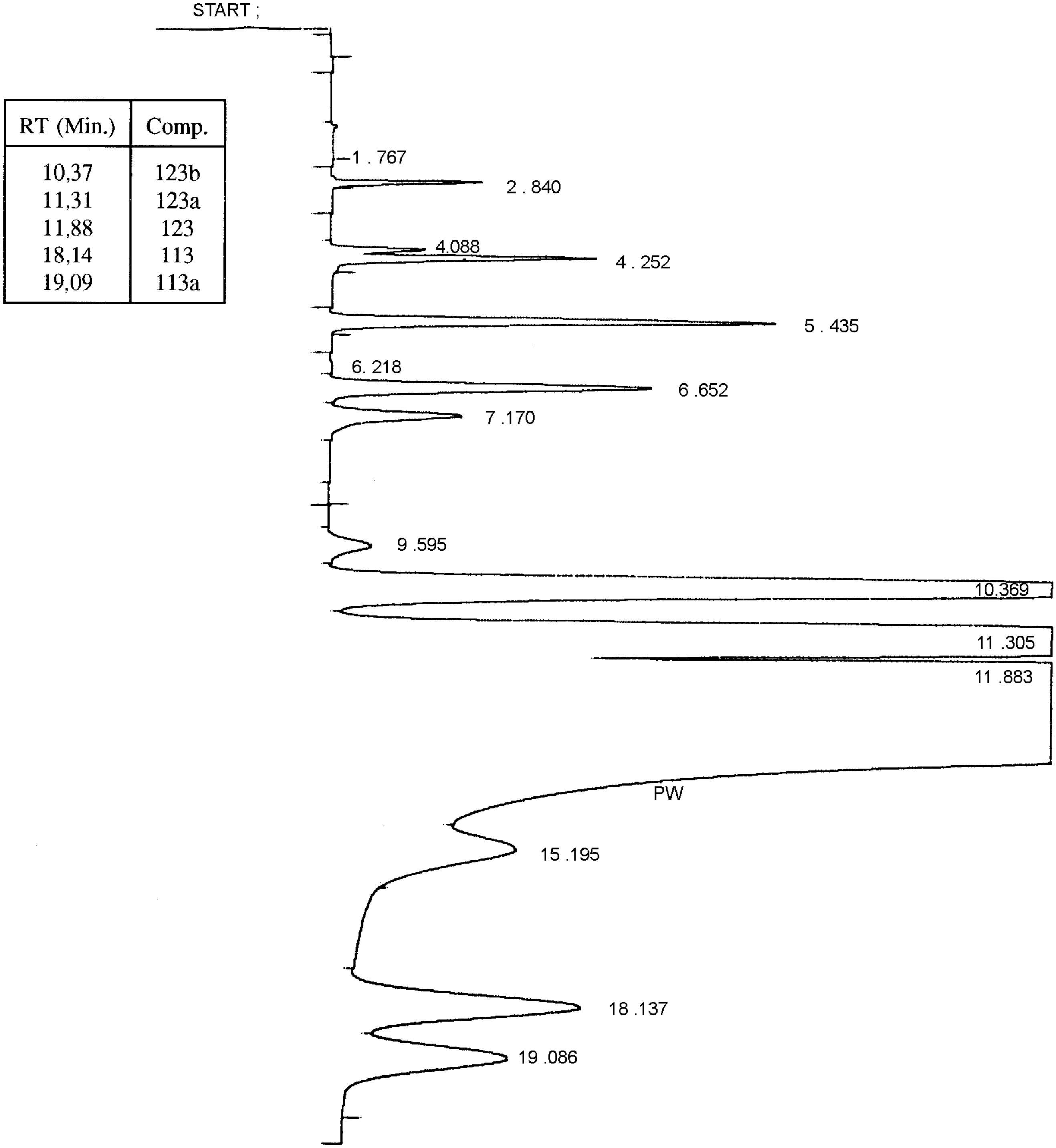


Figure 1. Packed Column Gas Chromatogram of R-123



RT (Min.)	Comp.
10,37	123b
11,31	123a
11,88	123
18,14	113
19,09	113a

Figure 2. Capillary Column Gas Chromatogram of R-123

Table 1. Component Statistical Parameters

Component	ECN (1)	Detection Limit, ppm	Concentration Investigated, ppm	Precision at 95% Confidence Limit, ppm	Relation Mean Error, %
1113	1.69	1	25	0.37	0.95
12	0.35	3	25	0.37	-1.1
22	0.40	2	25	0.24	1.4
114	1.04	2	50	1.20	-2.1
1317mx	3.63	1	30	0.88	4.3(2)
31	0.92	1	10	0.52	2.2
216ba	2.16	1	20	0.67	-1.8
1326mxz	3.65	1	15	0.33	0.7
133a	1.93	1	40	0.67	1.9
114B1	0.95	2	50	0.80	2.4
1112a	1.64	1	25	0.30	-0.7
1112	1.64	1	15	0.27	-0.5
123a	1.84	2	5%	0.13%	0.30
123b	1.80	2	400	12.7	--
11	0.43	3	60	2.20	1.8
30	0.63	2	50	1.10	0.3
113	1.60	3	300	7.30	-0.2
113a	1.68	3	250	7.00	-0.15
1111	1.90	2	15	0.67	0.8

(1) Effective Carbon Numbers (ECN) determined experimentally or estimated (see J. Chromatog. Sci., 30, 280 and 301 (1992).)
(2) Combining both isomers.

Table 2. Primary Calibration Standard Components

Component	Mol. Wt.	Vol. Added, mL	µg Added (1)	ppm Added (2)	Total ppm Present (3)
1113	116	1.00	4765	22	
12	121	1.00	4946	23	
22	86	1.50	5307	24	
114	170	1.50	10454	48	
1317mx(4)	216	6.0 µL	9289	41(6)	
31	68	0.75	2101	10	
216ba(5)	221	0.50	4517	21	
1326mxz	198	0.40	3270	15	
133a	118	1.80	8720	40	
114B1(4)	215	6.0 µL	11109	49	
1112a	133	1.00	5450	25	
1112	133	0.50	2725	13	
123a	153	Refer to Note 4 in Section 9.6	Refer to Note 4 in Section 9.6	1-7%	
123b	153	Refer to Note 4 in Section 9.6	Refer to Note 4 in Section 9.6	200-700	
11(4)	137	10.0 µL	14869	65	
30(4)	85	10.0 µL	13360	59	
113(4)	188	50 µL	78795	361	
113a(4)	188	50 µL	78986	362	
1111(4)	149	6.0 µL	9279	41	

(1) If necessary, correct the µg added for the purity of the calibration component previously established.
(2) Values shown are for illustration; exact values are determined at step f.
(3) Column to be completed (Section 9.1, step g of the General Procedure) after determining ppm present in stock R-123 (see Notes 1 and 4 in Section 9.6 of the General Procedure).
(4) Add by syringe injection of the liquid.
(5) Although other 216 isomers comprise the usual 216 peak multiplet, the 216ba isomer (available) is used for calibration purposes.
(6) The 1317mx will resolve into the cis and trans isomer peaks with a ratio of one to two, respectively.

Table 3. Additional Impurities Observed in R-123, Quantitation by Effective Carbon Number Method

1. Additional impurities observed in samples of R-123 are as follows:

Impurity	Capillary Column Retention Times (Min.)	Effective Carbon Numbers (1)
1132	9.18	2.0
125	9.46	0.79
134a	9.8	1.67
114a	11.22	1.17
124a	11.56	1.27
1122	11.57	1.76
124	11.77	1.33
328lcc ether (2)	14.59	3.9
114aB1	15.0	0.8
141b	19.9	2.0
1121	23.0	1.75
132b	25.35	1.9
1130-T	25.64	2.25
123B1	28.72	1.70
122b	36.28	1.75
122a	37.24	1.75
122	38.0	1.76
112a	43.55	1.48

(1) Refer to References (see J. Chromatog. Sci., 30, 280 and 301 , (1992).)
(2) Structure tentatively identified as: $CHClF\text{-}CF_2\text{-}O\text{-}CF_2\text{-}CF_3$.

2. Quantitation by ECN Method

Select a nearby peak in the chromatogram whose identification and response factor (RF) have been established (the Internal Standard).

Then:

$$RF_I = \frac{ECN_r}{ECN_I} \times \frac{MW_i}{MW_r}$$

Where: RF = either absolute or Relative Response Factor.
MW_i = molecular weight of the component to be determined.
MW_r = molecular weight of the Internal Standard Reference.

Part 11

R-124 GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Section 10 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-124.

Section 2. Limitations and Interferences

This method is applicable and calibrated for only those impurities found in Table 2 and Table 3. Other impurities that have been detected on occasion are listed (with retention times) in Table 4. This method will not detect any impurities that may elute within the comparatively large R-124 peak matrix.

Section 3. Gas Chromatographic Analysis Equipment and Conditions

Packed column: 7.32 m x 3.17 mm OD stainless steel, 1% SP-1000 on Carbopack B, 60/80 mesh, Supelco, Bellefonte, PA.

Detector	FID
Carrier Gas	40 mL He/Min.
Initial Column Temp.	40°C
Initial Hold	10 Min.
Program	10K/Min.
Final Column Temp.	160°C
Post Hold	18 Min.
Sample	0.30 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	150°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Section 4. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1. The data was obtained by analyzing an R-124 calibration mixture 7 times during one day by one operator.

Section 5. Tables

The following tables are to be used in conjunction with Section 10 of the General Procedure.

RT (Min.)	Comp.
2,70	23
3,84	116
4,53	13
5,17	1123
5,35	143a
6,26	152a
6,78	125
7,07	134a
7,80	22
8,50	13B1
11,90	115
14,39	12
15,60	263fb
16,53	124a
17,08	124
18,20	133a
18,89	217ba
19,66	217ca
20,47	114
20,87	114a
22,22	11
23,51	1112a
24,22	123
26,71	113
28,78	C-316bb
29,71	C-316bb

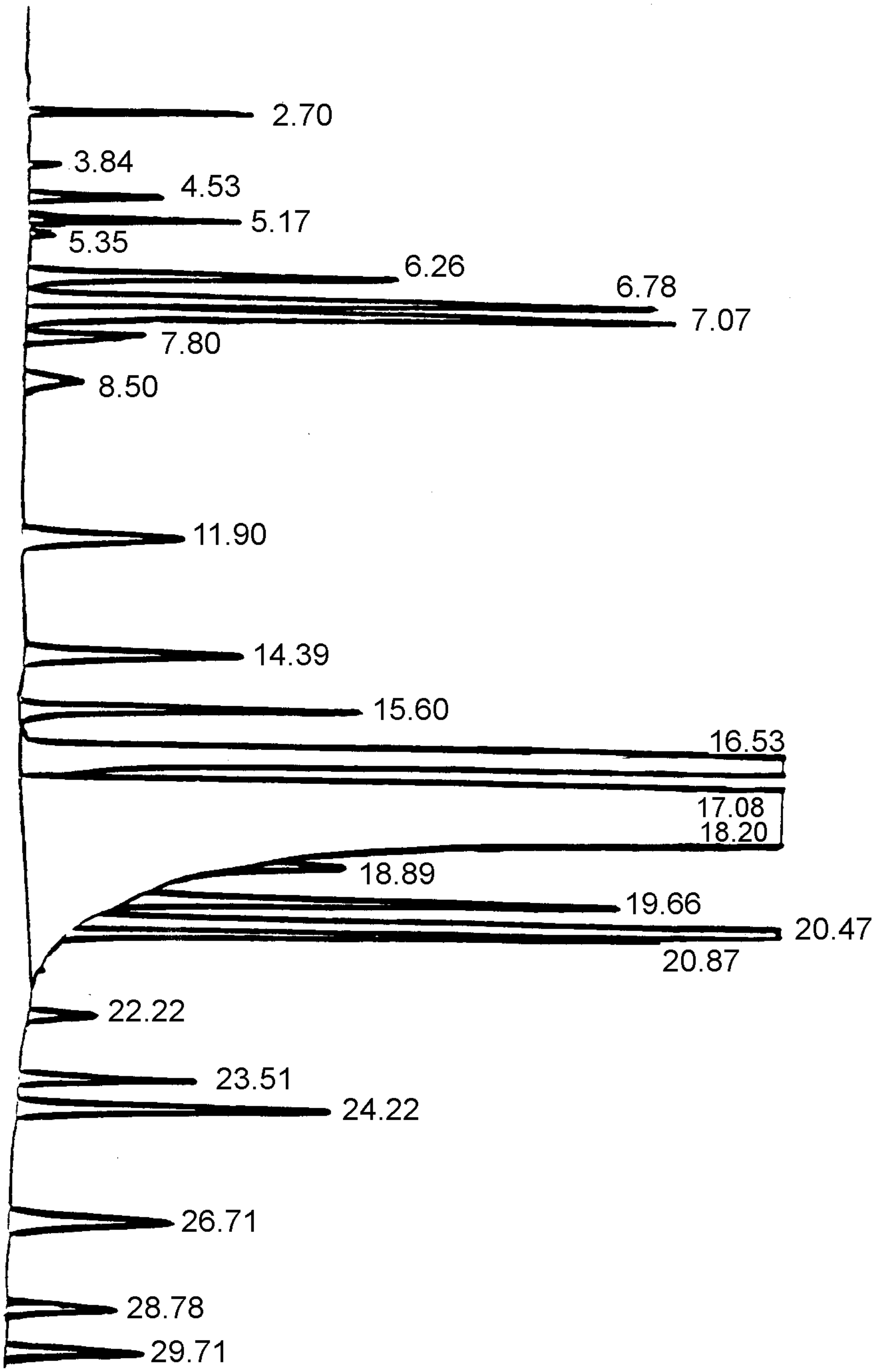


Figure 1. Gas Chromatogram of R-124

Table 1. Component Statistical Parameters

Component	Detection Limit, ppm	Concentration Investigated, ppm	Precision at 95% Confidence Level, ppm	Relative Mean Error, %
23	5	20	0.70	6.67
13	5	30	0.33	-1.33
1123	2	15	0.22	-2.67
143a	1	10	0.22	-4.05
152a	1	25	0.33	2.50
125	2	40	0.88	1.33
134a	2	50	0.70	2.50
22	3	20	0.73	-1.70
13B1	3	30	0.95	-2.67
115	3	35	1.13	3.7
12	4	25	0.70	4.67
263fb	2	25	0.88	3.33
124a	2	0.2%	0.009%	4.67
133a	5	50	3.33	-5.07
217ba	3	20	2.66	3.33
217ca	2	20	1.23	1.67
114	3	20	2.33	3.67
114a	3	50	3.66	4.33
11	6	40	0.87	-1.08
1112a	2	15	0.37	2.33
123	2	30	0.83	-2.67
113	3	25	0.96	-3.33
C-316bb	3	30	2.33	-5.33

Table 2. Primary Calibration Standard Components

Component	Mol. Wt.	Vol. Added, µL	µg Added(1)	ppm Added(2)	Total ppm Present(3)
23	70	22	63.0	20.0	
13	104	20	85.4	28.0	
1123	82	14	47.0	15.0	
143a	84	10	34.4	11.0	
152a	66	28	75.6	24.0	
125	120	25	122.7	39.0	
134a	102	38	158.5	51.0	
22	86	18	63.7	20.0	
13B1	149	15	91.4	29.0	
115	154	17	107.4	34.5	
12	121	15	74.1	24.0	
263fb	98	20	80.2	26.0	
124a(4)	136	--	--	--	
133a	118	25	121.2	39.0	
217ba	204	8	66.9	21.5	
217ca	204	8	66.9	21.5	
114	171	10	69.9	22.5	
114a	171	20	139.8	45.0	
11(5)	137		(6)	56.0	
1112a(5)	133		(6)	11.0	
123(5)	153		(6)	44.6	
113(5)	187		(6)	47.5	
C-316bb(5)	233		(6)	25.0	

(1) If necessary, correct the µg added for the purity of the calibration component previously established.
(2) Values shown are for illustration; exact values are determined at Section 10.1, step p of the General Procedure.
(3) Column to be filled in (Section 10.1, step q of the General Procedure) after determining ppm present in stock R-124 (see Note 1 in Section 10.7 of the General Procedure).
(4) Refer to Note 1 in Section 10.7 of the General Procedure.
(5) These components are liquids at ambient laboratory temperature and are added to the 500 mL bulb as described in Section 10.1 of the General Procedure, steps k through n.
(6) From Section 10.1 of the General Procedure, step n.

Table 3. Primary Calibration Standard Liquid Impurities

Comp.	Vol. Added, mL	Density at 20°C	g
C-316bb	4.0	1.644	6.576
113	8.0	1.565	12.520
123	8.0	1.470	11.760
11	10.0	1.487	14.870
1112a	2.0	1.439(10°C)	2.878

Table 4. Retention Time Data for Identified Impurities Not Normally Observed

Impurity	Retention Time (Min.)
32(1)	2.70
116	3.84
1114	5.00
134	8.07
143	8.30
31	8.53
142b	15.22
365mc	15.30
1113	16.06
1225ye	16.13
Met. Formate	16.92
133	19.05
1318my-T	19.70
1318my-C	21.05
1327	20.50
123b	22.90
114B1	23.50
123a	23.60
216	23.80
113a	27.10
123aB1	28.50
122a	32.70
122	33.50
234	35.35
1111	35.60

(1) 32 and 23 coelute. A one (1) ft. column section of Porapak-T attached to the detector side of the column will resolve these two components—the 23 peak eluting first.

Part 12

R-125 GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Section 10 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-125.

Section 2. Limitations and Interferences

This method is applicable and calibrated for only those impurities found in Table 2 and Table 3. Other impurities that have been detected on occasion are listed (with retention times) in Table 4. This method will not detect any impurities that may elute within the comparatively large R-125 peak matrix.

Section 3. Gas Chromatographic Analysis Equipment and Conditions

Packed column: 7.32 m x 3.17 mm OD stainless steel, 1% SP-1000 on Carbopack B, 60/80 mesh, Supelco, Bellefonte, PA.

Detector	FID
Carrier Gas	45 mL He/Min.
Initial Column Temp.	40°C
Initial Hold	6 Min.
Program	5 K/Min.
Final Column Temp.	130°C
Post Hold	11 Min.
Sample	0.50 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	150°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Section 4. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1. The data was obtained by analyzing an R-125 calibration mixture 7 times during one day by one operator.

Section 5. Tables

The following tables are to be used in conjunction with Section 10 of the General Procedure.

RT (Min.)	Comp.
2,06	Methane
2,75	23/32
3,93	116
4,62	13
5,43	143a
6,21	125
8,44	13BL
11,15	115
13,58	12
15,68	1113
16,15	124a
17,01	115B1
17,51	124
18,29	133a
21,72	114a
28,81	123
33,46	113

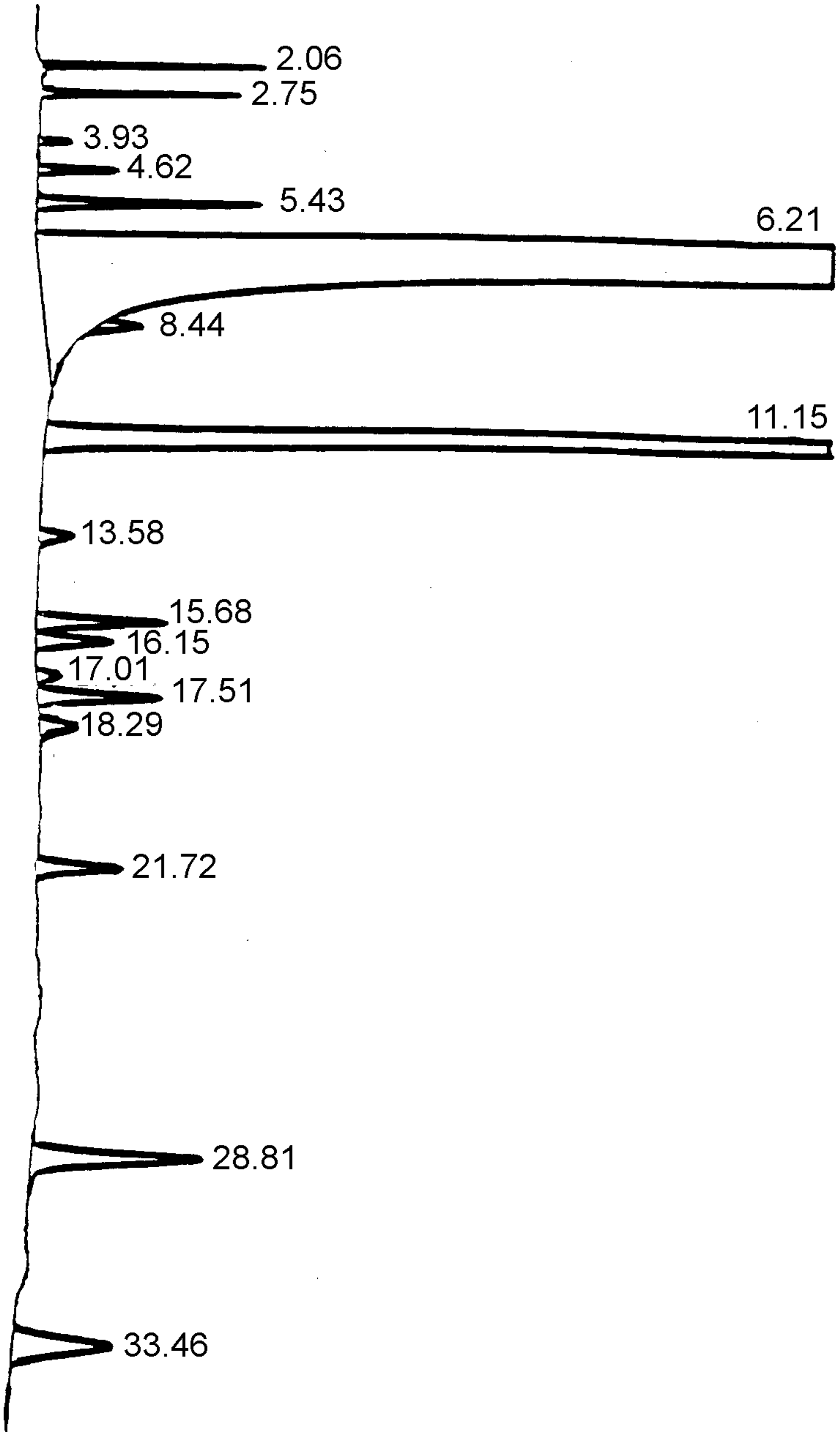


Figure 1. Gas Chromatogram of R-125

Table 1. Component Statistical Parameters

Component	Detection Limit, ppm	Concentration Investigated, ppm	Precision at 95% Confidence Level, ppm	Relative Mean Error, %
Methane	0.25	5	0.06	4.00
23	4	15	0.70	6.67
116	75	200	5.86	2.50
13	5	30	0.24	-3.33
143a	1	10	0.15	-5.00
13B1	10	30	1.10	2.30
115	4	0.20%	0.006%	1.50
12	2	25	0.53	-2.40
1113	1	15	0.20	-4.00
124a	1	10	0.20	4.40
115B1	4	15	0.35	5.33
124	1	20	0.30	2.55
133a	1	15	0.33	-1.05
114a	2	25	0.65	1.33
123	2	30	0.70	-2.66
113	2	30	1.10	-3.35

Table 2. Primary Calibration Standard Components

Component	Mol. Wt.	Vol. Added, µL	µg Added (1)	ppm Added (2)	Total ppm Present (3)
Methane	16	21	14.0	5	
23	70	15	42.5	15	
116	138	0.10 mL	564	207	
13	104	20	85.4	31	
143a	84	8	27.5	10	
13B1	149	14	85.3	31	
115(4)	154	(4)	--	--	
12	121	14	69.2	25	
1113	116	8	38.1	14	
124a	136	5	27.9	10	
115B1	199	5	40.7	15	
124	136	10	55.8	21	
133a	118	8	38.8	14	
114a	171	10	69.9	26	
123(5)	153		(6)	51	
113(5)	187		(6)	32	

(1) If necessary, correct the µg added for the purity of the calibration component previously established.
(2) Values shown are for illustration; exact values are determined at Section 10.1, step p.
(3) Column to be filled in Section 10.1 step q of the General Procedure after determining ppm present in stock R-125 (see Note 1 in Section 10.7).
(4) Refer to Note 1 in Section 10.7.
(5) These components are liquids at ambient laboratory temperature and are added to the 500 mL bulb as described in 10.1, steps k through n.
(6) From Section 10.1 step n.

Table 3. Primary Calibration Standard Liquid Impurities

Comp.	Vol. Added, mL	Density at 20°C	g
R-113	12.0	1.565	18.78
R-123	20.0	1.470	29.40

Table 4. Retention Time Data for Identified Impurities Not Normally Observed

Impurity	Retention Time (Min.)
1141	2.00
14	2.35
32(1)	2.75
C_2H_4	3.33
C_2H_6	4.08
1114	4.94
22	7.77
134a	8.32
218	10.6
C-318	14.4
329	15.0
31-10	19.1
114	21.5
1318my-T	20.77
1318my-C	23.10
227	24.0
1327	24.85

(1) 32 and 23 coelute. A one (1) ft. column section of Porapak-T attached to the detector side of the column will resolve these two components—the 23 peak eluting first.

Part 13

R-134a GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Section 10 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-134a.

Section 2. Limitations and Interferences

This method is applicable and calibrated for only those impurities found in Table 2 and Table 3. Other impurities that have been detected on occasion are listed (with retention times) in Table 4. This method will not detect any impurities that may elute within the comparatively large R-134a peak matrix.

Section 3. Gas Chromatographic Analysis Equipment and Conditions

3.1 *Chromatographic Equipment and Conditions, Packed Column*

Packed column: Formed by joining in series the following 4 columns in the following order given:

1. 1.83 m 5% Bentone 34/5%-SP-1200-on Supelcoport, 100/200 mesh.

2. 6m 5% Krytox on Carbopack B, 60/80 mesh.

3. 4.88m x 3.17 mm OD stainless steel, 1% SP-1000 on Carbopack B, 60/80 mesh.

4. .35m Porapak T, 60/80 mesh.

NOTE: The Bentone column is attached to the injection port all columns are made from 3.17mm OD stainless steel tubing. The individual columns are conditioned separately before joining together. Columns are available from , Supelco, Bellefonte, PA.

Detector	FID
Carrier Gas	20 mL He/Min.
Initial Column Temp.	40°C
Initial Hold	6 Min.
Program	10 K/Min.
Final Column Temp.	165°C
Post Hold	26 Min.
Sample	0.5 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	150°C
Max Safe Column Temp.	175°C (for conditioning purposes)

3.2 *Chromatographic Equipment and Conditions, Capillary Column*

Capillary Column: 120m (or 2-60m) DB-1301, 0.25mm, 1µ, J&W Scientific Co.,Folsom, CA.

Detector	FID
Carrier Gas	approx. 1mL He/Min
Injection Port Temp	150°C
Detector Temp.	250°C
Sample	1.0 mL
Max. Safe Column Temp.	280°C
Initial Col. Temp	-20°C
Initial Hold	7 Min.
Program	8 K/Min.
Final Column Temp.	50°C
Post Hold	5 Min.
Split Ratio	30:1
Pressure	240 kPa
Makeup Gas	30 mL He/Min.
Sub-ambient Cooling	Liquid Nitrogen

See Note 6 of Section 10.7 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography.

Section 4. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1. The data was obtained by analyzing an R-134a calibration mixture 7 times during one day by one operator.

Section 5. Tables

The following tables are to be used in conjunction with Section 10 of the General Procedure.

RT (Min.)	Comp.
8,51	23
9,02	32
12,16	1123
12,67	143a
13,48	125
13,72	134a
17,48	115
18,23	1243zf
18,94	12
19,61	1122
20,42	124
21,13	133a
23,85	1336mzz
24,61	114
24,91	114a
27,70	11
28,79	1112a
29,46	1121-c
30,13	123
31,12	1121-T
36,71	113

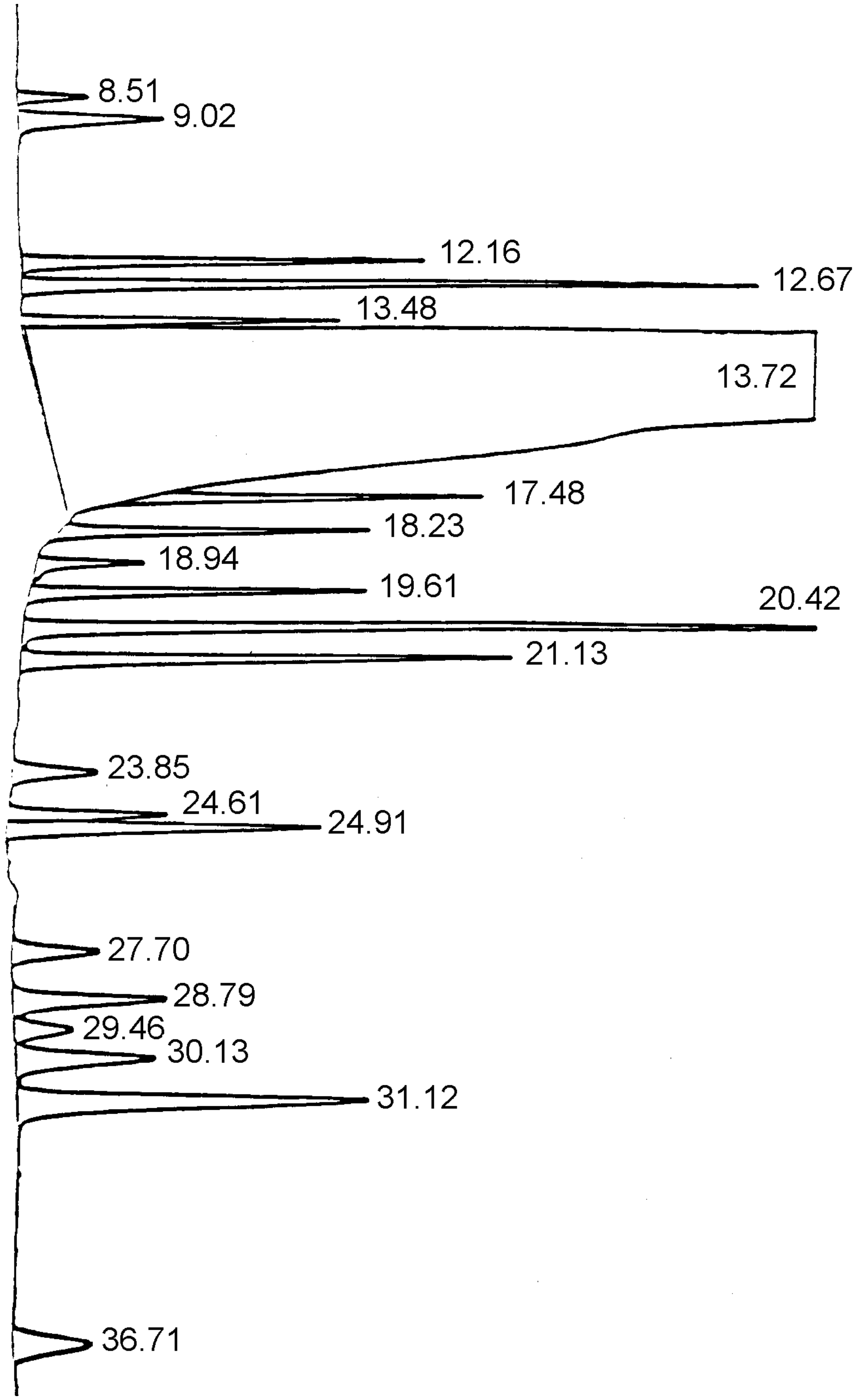


Figure 1. Packed Column Gas Chromatogram of R-134a

RT (Min.)	Comp.
12,54	143a
12,71	125
13,75	1234yf
13,87	134a
14,98	152a
15,47	134
18,92	114a
19,20	40
19,34	1122
19,95	124
21,50	31

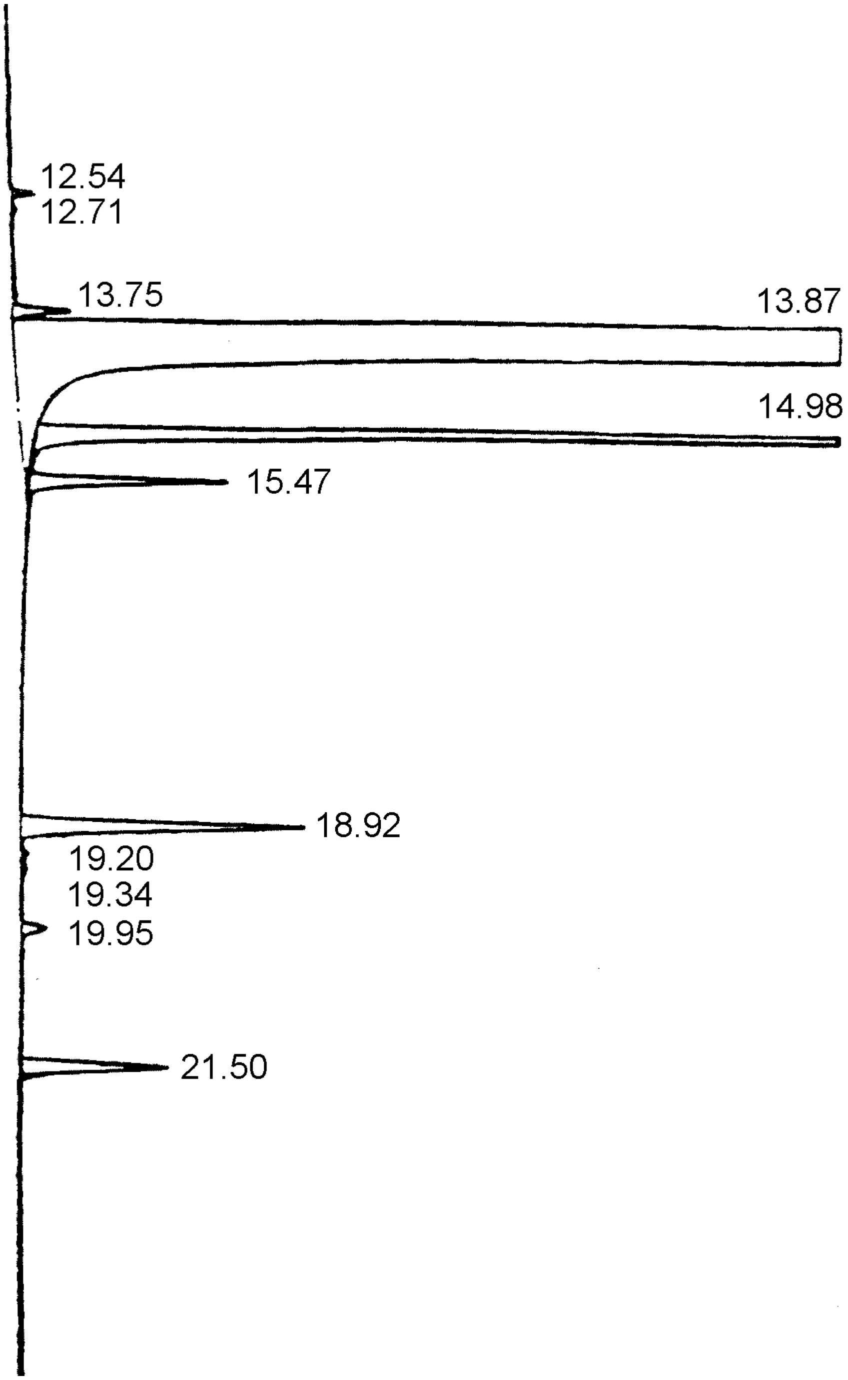


Figure 2. Capillary Gas Chromatogram of R-134a

Table 1. Component Statistical Parameters

Component	Effective Carbon Number (1)	Detection Limit, ppm	Concentration Investigated, ppm	Precision at 95% Confidence Level, ppm	Relative Mean Error, %
23	0.16	4	15	0.70	1.8
32	0.62	2	15	0.30	1.2
1123	1.93	1	20	0.20	-0.8
143a	2.12	1	20	0.20	1.5
125	0.79	2	30	0.25	3.2
115	0.76	5	60	0.65	-1.3
1243zf	2.84	1	10	0.20	-3.6
12	0.35	2	40	0.30	1.8
1122	1.76	1	15	0.20	2.2
124	1.33	1	40	0.45	2.0
31	0.92	1	15	0.80	1.7
133a	1.93	1	25	0.50	1.7
1336mzz	2.90	1	--	0.5(2)	--
114	1.04	2	30	1.10	-3.3
114a	1.10	2	50	1.20	4.3
11	0.43	4	50	2.60	2.6
1112a	1.64	1	15	0.30	-0.2
1121-C	1.75	1	10	0.30	-6.7
123	1.76	2	20	0.90	-3.3
1121-T	1.75	1	30	1.00	4.3
113	1.60	2	20	1.3	1.7
134	1.61	2	30	0.20	1.4
152a	1.08	1	30	0.20	0.8
1234yf	2.65	1	--	0.5(2)	--

(1) Effective Carbon Numbers (ECN) were determined experimentally (see J. Chromatog. Sci., 30, 280, (1992).)
(2) Precision estimated at 10 ppm based upon sample reproducibility.

Table 2. Primary Calibration Standard Components

Component	Mol. Wt.	Vol. Added, µL	µg Added (1)	ppm Added (2)	Total ppm Present (3)
23	70	12	34.73	15	
32	52	16	34.03	15	
1123	82	14	46.98	20	
143a	84	14	48.10	20	
125	120	14	68.72	30	
115	154	22	139.05	60	
134	102	28	116.81	50	
152a	66	25	67.49	30	
12	121	20	98.89	43	
1122	98	8	32.23	15	
124	136	16	89.32	39	
31	68	12	33.61	14.5	
133a	118	12	58.17	25	
114	170	10	69.46	30	
114a	170	20	138.92	60	
11(4)	137		(6)	30	
1112a(4)	133		(6)	18	
1121-C(4)	115		(5,6)	5	
123(4)	153		(6)	19	
1121-T(4)	115		(5,6)	23.5	
113(4)	188		(6)	24	

(1) If necessary, correct the µg added for the purity of the calibration component previously established.
(2) Values shown are for illustration; exact values are determined at 10.1, step p of Part 1. Column to be filled in (Section 10.1, step q) after determining ppm present in stock R-134a.
(3) These components are liquids at ambient temperature and are added to the 500 mL bulb as described in Section 10.1, steps k through n of the General Procedure.
(5) 1121 typically contains about 17.5% cis isomer. µg 1121 added x 0.175 is assigned to the cis isomer, the balance to the trans isomer.
(6) From Section 10.1 of the General Procedure, step n.

Table 3. Primary Calibration Standard Liquid Impurities

Comp.	Vol. Added, mL	Density at 20°C	g
113	6.0	1.565	9.390
1121C&T	8.0	1.403	11.224
123	5.0	1.470	7.350
11	8.0	1.487	11.896
1112a	5.0	1.439(10°C)	7.195

Table 4. Additional Impurities Observed in R-134a, Quantitation by Effective Carbon Number Method

1. Additional impurities occasionally observed in sample of R-134a are tabulated below.

Impurity	Column Retention Time (Min.) Packed	Column Retention Time (Min.) Capillary	Effective Carbon Number ECN (1)
1243zf	18.23	14.98	2.84
1336mzz	31.12		2.90
1234yf	18.20	13.75	2.65
22		16.40	0.40
123a	29.50		1.84
124a	19.75		1.27
245cb	18.50		2.60
1225ye	19.21		2.42
1113	19.20		1.69
263fb	19.21		2.95
1140	18.50	21.50	2.08
132b	31.12		1.90
13	12.16		0.23
1318my-T	23.18		2.95
1318my-C	24.60		2.95

(1) Refer to J. Chromatog. Sci., 30, 280, (1992).

2. Quantitation by ECN Method

Select a nearby peak in the chromatogram whose identification and response factor (RF) have been established (the Internal Standard).

Then:

$$RF_I = \frac{ECN_r}{ECN_I} \times \frac{MW_i}{MW_r}$$

Where: RF = either Absolute or Relative Response Factor.
MW_i = molecular weight of the component to be determined.
MW_r = molecular weight of the Internal Standard Reference.

Part 14

R-143a GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Section 10 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-143a.

Section 2. Limitations and Interferences

This method is applicable and calibrated for only those impurities found in Table 2. This method will not detect any impurities that may elute within the comparatively large R-143a peak matrix.

Section 3. Gas Chromatographic Analysis Equipment and Conditions

3.1 *Chromatographic Equipment and Conditions, Combination Packed Column.*

Packed column: Formed by joining in series the following 4 columns in the following order given:

1. 1.83 m 5% Bentone 34/5%-SP-1200-on Supelcoport, 100/200 mesh.

2. 6m 5% Krytox on Carbopack B, 60/80 mesh.

3. 4.88m x 3.17 mm OD stainless steel, 1% SP-1000 on Carbopack B, 60/80 mesh.

4. 0.35m Porapak T, 60/80 mesh.

NOTE: The Bentone column is attached to the injection port all columns are made from 3.17mm OD stainless steel tubing. The individual columns are conditioned separately before joining together. Columns are available from, Supelco, Bellefonte, PA.

Detector	FID
Carrier Gas	20 mL He/Min.
Initial Column Temp.	40°C
Initial Hold	6 Min.
Program	10 K/Min.
Final Column Temp.	165°C
Post Hold	26 Min.
Sample	0.5 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	175°C (for conditioning purposes)

3.2 *Chromatographic Equipment and Conditions, 1% SP-1000 Packed Column*

Packed Column: 7.32m x 3.17 m OD stainless steel, 1% SP-1000 on Carbopack B, 60/80 mesh, Supelco, Bellefonte, PA.

Detector	FID
Injection Port Temp	200°C
Detector Temp.	250°C
Sample	0.50 mL (gas syringe)
Max. Safe Column Temp.	225°C (for conditioning purposes)
Initial Col. Temp	35°C
Initial Hold	7 Min.
Program	10 K/Min.
Final Column Temp.	150°C

Section 4. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1. The data was obtained by analyzing an R-143a calibration mixture 7 times during one day by one operator.

Section 5. Tables

The following tables are to be used in conjunction with Section 10 of the General Procedure.

RT (Min.)	Comp.
3,00	41
3,37	23/32
4,17	C_2H_4
4,97	C_2H_6
7,04	143a
8,06	152a
8,32	40
8,67	125
8,96	134a
9,66	22
15,00	142b
15,50	1113
16,69	124
18,20	isobutane
19,30	114/114a
19,78	n-butane

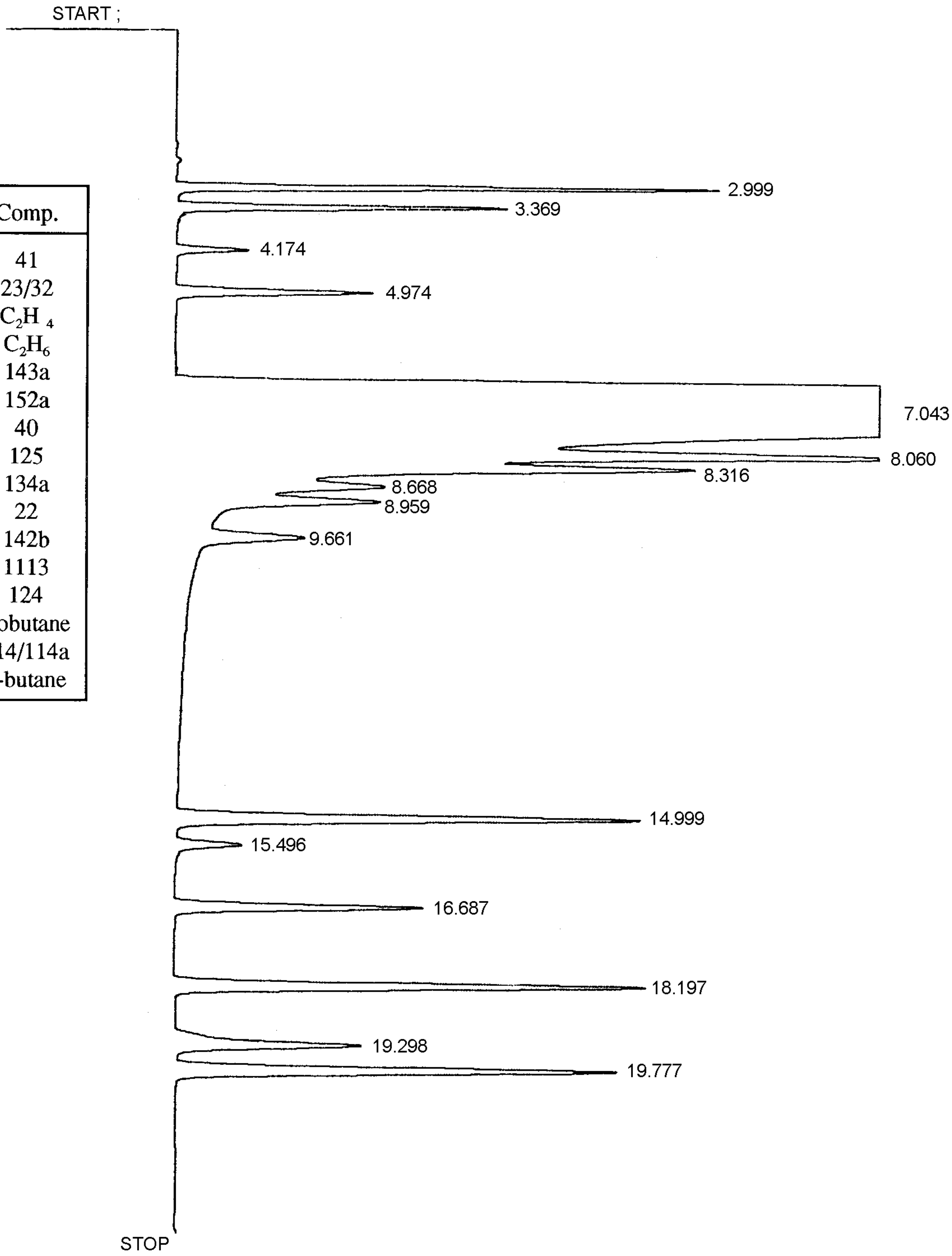


Figure 1. 1% SP-1000 Packed Column Gas Chromatogram of R-143a

RT (Min.)	Comp.
8,51	23
9,02	32
10,97	Ethane
12,72	143a
14,27	134a
14,51	22
17,09	Propane
17,72	115
18,62	1216
19,12	12
19,52	1113
24,56	n-butane

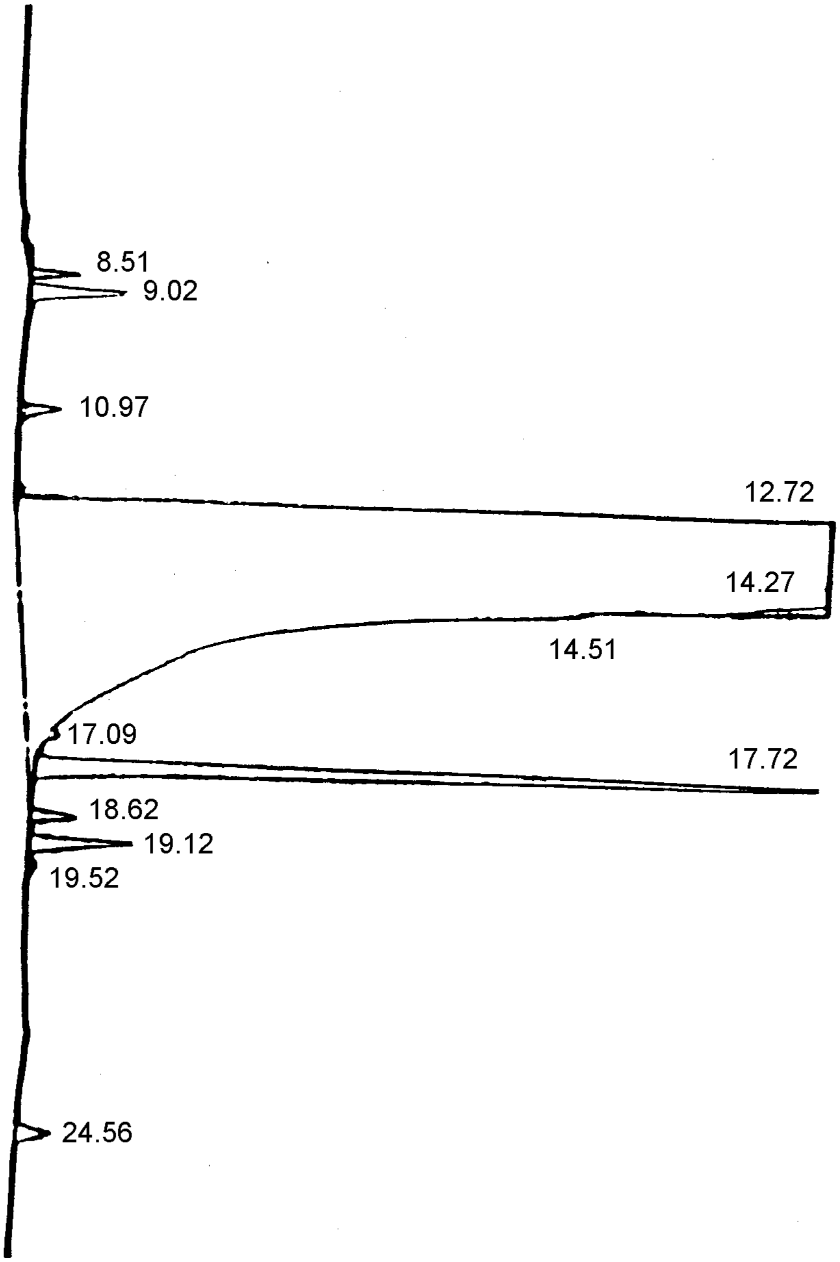


Figure 2. Combination Packed Column Gas Chromatogram of R-143a

Table 1. Component Statistical Parameters

Component	Detection Limit, ppm	Concentration Investigated, ppm	Precision at 95% Confidence Limit, ppm	Relative Mean Error, %
41	1	30	0.53	-1.0
23	4	25	1.19	0.8
32	1	20	0.73	2.1
C_2H_4	1	5	0.53	-3.7
C_2H_6	1	5	0.37	-4.2
152a	2	50	1.81	2.6
40	3	40	1.53	1.3
125	8	40	2.13	2.7
134a	5	25	1.87	-1.2
22	6	40	1.19	1.8
142b	1	65	1.43	1.2
1113	2	15	0.87	-0.9
124	3	25	0.73	2.1
isobutane	1	5	0.33	8.2
114a	5	35	0.87	-0.8
n-butane	1	5	0.33	5.1
C_3H_8	1	5	0.47	-3.7
115	3	75	2.13	0.9
1216	2	25	1.01	-1.7
12	5	40	0.88	3.1

Table 2. Primary Calibration Standard Components

Component	Mol. Wt.	Vol. Added, μL	μg Added (1)	ppm Added (2)	Total ppm Present (3)
41	34	40	55.7	29	
23	70	16	45.8	24	
32	52	18	38.3	20	
C_2H_4	28	8	9.9	5	
C_2H_6	30	8	9.8	5	
152a	66	35	94.5	50	
40	50	35	72.3	38	
125	120	15	73.6	38	
134a	102	10	41.7	22	
22	86	20	70.8	37	
142b	100	30	123.3	65	
1113	116	6	28.6	15	
124	136	8	44.7	23	
isobutane	58	6	14.2	8	
114a	170	10	69.7	37	
n-butane	58	6	14.2	8	
C_3H_8	44	5	9.0	5	
115	154	22	139.0	73	
1216	150	8	49.1	26	
12	121	15	74.2	39	

(1) If necessary, correct the μg added for the purity of the calibration component previously established.

(2) Values shown are for illustration; exact values are determined in Section 10.1, step p of the General Procedure.

(3) Column to be filled in (Section 10.1, step q of the General Procedure) after determining ppm present in stock R-143a (see Note 1 in Section 10.7 of the General Procedure).

PART 15

DETERMINATION OF COMPONENT CONCENTRATIONS OF REFRIGERANT 400 AND 500 SERIES BLENDS AND AZEOTROPES BY GAS CHROMATOGRAPHY

Section 1. Purpose

The purpose of this test method is to determine the component compositions of new and reclaimed Refrigerants 400 (R-400) and 500 (R-500) blends and azeotropes by Gas Chromatography (GC). This method utilizes one (1) GC packed column to separate and quantify the individual components for all of the refrigerant blends and azeotropes.

Section 2. Scope

This test method is for use with all R-400 and R-500 blends and azeotropes as outlined in Section 11.

Section 3. Definitions

Definitions for this part are identical to those of ARI Standards 700-95 and 740-95.

Section 4. Principle

The compositions of new and reclaimed blends and azeotropes are determined by either isothermal or programmed temperature gas chromatography using a packed column and a thermal conductivity detector (TCD). Component peak areas are integrated electronically and quantified by the area normalization-response factor method by reference to a suitable calibration standard prepared to closely match the expected composition of the particular blend.

Section 5. Applicability

This method is applicable to the determination of the composition of new and reclaimed R-400 and R-500 blends and azeotropes.

Section 6. Limitations and Interferences

This method is calibrated for each of the respective R-400 and R-500 blends. If components other than those comprising the given mixture are present in significant amounts, results can be erroneous and/or misleading. Any impurity that coelutes with one of the blend components will interfere if present in significant concentration. This method is limited to the composition of the blends and does not address the organic purity of the blend sample.

Section 7. Sensitivity, Precision, and Accuracy

Values for these statistical parameters are given in the appropriate GC Method Data Sheet.

Section 8. Special Apparatus and Reagents

NOTE: Equivalents may be substituted.

1. Programmable temperature gas chromatograph, single or dual packed column, TCD detector, low sensitivity position: Model 6890, Hewlett Packard, Wilmington, DE.
2. Electronic integrator: Model# 3396 or suitable data acquisition system and GC software, Hewlett Packard, Wilmington, DE.
3. Packed column: 7.32 m x 3.17 mm OD stainless steel, 1% SP-1000 on Carbopack B, 60/80 mesh. This column may be assembled by attaching two or three shorter columns together in series. Condition O/N at 225°C. Supelco, Bellefonte, PA.
4. Gas collecting tube: 250 mL, LG-8601, Lab Glass Inc., Vineland, NJ. (Enlarge side outlet opening to accommodate a crimp-on 2-cm septum. Apply fiberglass tape outside for protection from breakage).
5. Steel cylinder: 1L, with a single #9 valve (#1014-C, Superior Valve), 3/8" pipe neck, E. F. Britten Co., Cranford, NJ.
6. Syringe, Gastight, 1.0 mL, Hamilton 1001TLL; Supelco, P/N 2-0997M.
7. HP Steel Cylinder, EFB-56, 400 psig, single #9 valve (#1014-C Superior Valve), 3/8” pipe neck, approximately 925 mL internal volume, E.F. Britten Co., Cranford, NJ.
8. VHP Stainless Steel Cylinder, 1L, 1800 psig, 304L-HDF4-100. Valve SS-IRS4-A, Whitey, Wilmington Valve and Fitting, Co., New Castle, DE.

9. The fluorochemicals may be purchased from Lancaster, Windham, NH and Synquest, Inc., Alachua, FL. The hydrocarbons may be purchased from Scott Specialty Gases, Inc., Plumbsteadville, PA.

NOTE: The purity of each calibration component must be predetermined by gas chromatography and, if necessary, by GC/Mass Spectroscopy (GC-MS).

Section 9. Procedure

9.1 *Calibration Standard Preparation*

NOTE: The following procedure is generalized and is followed for the preparation of each blend calibration standard. The weights of each blend component to be added and the order of addition are given in the appended GC Method Data Sheets for each respective refrigerant blend. Normally, the least volatile components are added first. Refer to Note 1 for additional information.

a. Pressure test the clean, dry calibration standard cylinder to about 2000 kPa (using N_2) to insure there are no leaks.

b. Evacuate the cylinder to below 0-10 kPa (i.e. full vacuum). Close the valve and record the cylinder weight to the nearest 0.1 g (i.e. tare weight)

c. Using the Teflon® transfer line, loosely connect the cylinder containing the first component to be added to the calibration standard cylinder. Briefly purge the transfer line, then connect tightly to the standard cylinder valve.

d. Transfer the calculated weight (approximately) of the component to the standard cylinder.

NOTE: If the amount added is less than desired, the cylinder is purged until the desired weight is obtained. Purging the cylinder is permitted only during addition of the first component.

e. Reweigh and record the cylinder weight. This weight less the tare weight (step b) gives the weight of the first added component.

f. Externally cool the calibration cylinder in an ice water bath for at least 20 minutes (Refer to Note 4).

g. Connect the Teflon® transfer line from the second component cylinder and transfer the desired weight of the second component as was done above.

NOTE: Generally, it is simpler to prepare a second cylinder containing the exact weight of the second component to be added, then transferring the entire contents (or nearly so) into the calibration standard cylinder.

h. Close the cylinder valves, remove the transfer line, remove the calibration standard cylinder from the ice water bath, and allow the cylinder and contents to warm to ambient laboratory temperature.

i. After drying the cylinder externally, weigh the calibration standard cylinder and then subtract the gross weight recorded in step e. The net difference is the weight of the second added component.

j. Repeat above steps f through i for a third and (if necessary) for a fourth component.

k. Add the weights of all the added components and then determine the weight percentage (to the nearest 0.01%) of each component in the calibration mixture. If necessary, correct the individual component percentages for the purities determined in Section 8.

l. Attach a label to the calibration standard cylinder containing the name of the refrigerant standard, weight percentage of each component present, date of preparation, total gross weight (i.e. cylinder plus contents), and gross weight for renewal (See Note 2). Store the standard in a cool, secure location.

9.2 *Determination of Component Response Factors*

a. Set-up the cylinder sampling apparatus as shown in Figure 1. The plastic line connecting metering valve “E” to the gas bulb valve “B” must be kept as short as possible as to minimize component fractionation during the transfer.

b. With valves “E”, “B”, and “C” open, slowly open valve “D” and evacuate the apparatus up to cylinder valve “A”. Close valve “D” and insure the system is holding vacuum.

c. Close valve “E” and then open cylinder valve “A”.

d. Slowly open valve “E” and introduce sample into the gas bulb until the vacuum gauge reads 102 kPa pressure. Close valve “A”.

e. Repeat steps b through d.

f. Set the GC integrator (or work station) for an area normalization response factor calibration run.

g. Set the GC to the operating conditions given in the GC Method Data Sheet for the refrigerant blend to be calibrated. The TCD is always set in the least sensitive position for all blend component analyses.

h. Using the 1.0 mL syringe, withdraw vapor sample from the gas bulb and inject into the GC. Withdraw slowly, allowing sufficient time (5 seconds) for the calibration standard to fill the syringe.

i. Repeat the GC analysis twice more.

j. Store the average of the three determined Relative Response Factors for each blend component in the Method Table as follows:

$$\mathrm{ARF_i} = \frac{\mathrm{Wt\%\ \ Comp.\,i\ in\ Cal.\,Std.}}{\mathrm{A_i}}$$

$$\mathrm{RRF_i} = \frac{\mathrm{ARF_i}}{\mathrm{ARF_n}}$$

Where:

ARF_i = absolute response factor for component i.
ARFn= absolute response factor for the component with the highest weight percentage in the mixture.
RRF_i = relative response factor for component i.
A_i = peak area of component i.

NOTE: RRFn will always equal 1.0. The largest component (peak area) is normally selected as the reference peak. RRF values are computed to the nearest 0.0001 unit.

9.3 *Sampling*

High Pressure (HP) sample cylinders should be filled at least 80% but no more than 90% liquid full. This will both minimize component fractionation within the sample container and will avoid overfilling for safety considerations. Very high-pressure (VHP) sample cylinders (vapor phase) may be filled to less than saturation pressure, if desired.

9.4 *Sample Analysis*

Analyze the sample using the chromatographic conditions described in the GC Method Data Sheet. Load the sample as illustrated in Figure 1 in the same manner as described for the GC method calibration standard. Questionable sample results are most often resolved by re-analysis of the calibration standard. The temperature of the sample must equal (essentially) that of the calibration standard at the time of GC calibration. In the case of VHP refrigerants, the sample must be at or above the critical temperature before analysis.

9.5 *Calculations*

a. The weight percentage of each component is calculated as follows:

$$\mathrm{W_i} = \frac{\mathrm{100\ x\ RRF_i\ \ x\ A_i}}{\sum (\mathrm{A_i\ \ x\ RRF_i})}$$

Where:

W_i = weight percentage of component i.
RRF_i = relative response factor for component i.
A_i = peak area of component i.
Σ…= sum of all component peak areas times their respective relative response factors.

Report weight percentage results to the nearest 0.01%. (Note 3)

Section 10. Notes

1. The weights of each HP Standard component to be added have been calculated such that, after final loading, the HP standard cylinder will be about 90% filled with liquid phase. This is not the case for the very high-pressure (VHP) standards whose critical temperatures are either at or below ambient laboratory temperatures. For VHP standards (R-508, for example), the refrigerant vapor phase is used for calibration and sample analysis. Because of pressure considerations, two calibration standard cylinders are used, one for HP and the other for VHP refrigerants. In cases where one refrigerant blend has more than one composition (for example, R-401A and R-401B), where possible, just one calibration standard is prepared with composition approximately the average of the A and B compositions.

2. Experimental data has shown that vapor/liquid phase equilibrium for several R-400 series blends is essentially constant within a container following removal (and after re-equilibration) of about one third of the original liquid phase from the cylinder (see Reference 1). Hence, a freshly prepared calibration standard with 90% liquid phase will retain its original component ratio until the liquid phase has been depleted to below 60% of the cylinder volume—at which point the standard should be renewed. This renewal point is conveniently determined by subtracting the cylinder tare weight from the final cylinder gross weight, taking 66% of this difference, and then adding the tare weight to this 66% value. This value (recorded on the calibration standard label) is the cylinder gross weight at which the HP standard should be renewed. This equilibrium consideration does not apply to the VHP standards that are all vapor at ambient laboratory temperature.

3. The sum of the individual component weight percentages will not necessarily equal 100.00%. This procedure determines the blend component amounts present and does not address other volatile organic impurities that are more or less present in the mixture.

4. In preparing the VHP refrigerant calibration standards, it is usually necessary to externally cool the 1L cylinder in

dry ice in order to add the remaining refrigerant components.

Section 11. GC Method Data Sheets

Table 1. GC Method Data Sheets

Refrigerant	GC Method Data Sheet Part Number
R-401	Part 16
R-402	Part 17
R-403B	Part 18
R-404A	Part 19
R-406A	Part 20
R-407	Part 21
R-408A	Part 22
R-409	Part 23
R-410	Part 24
R-411	Part 25
R-412	Part 26
R-500	Part 27
R-502	Part 28
R-503	Part 29
R-507	Part 30
R-508	Part 31
R-509	Part 32

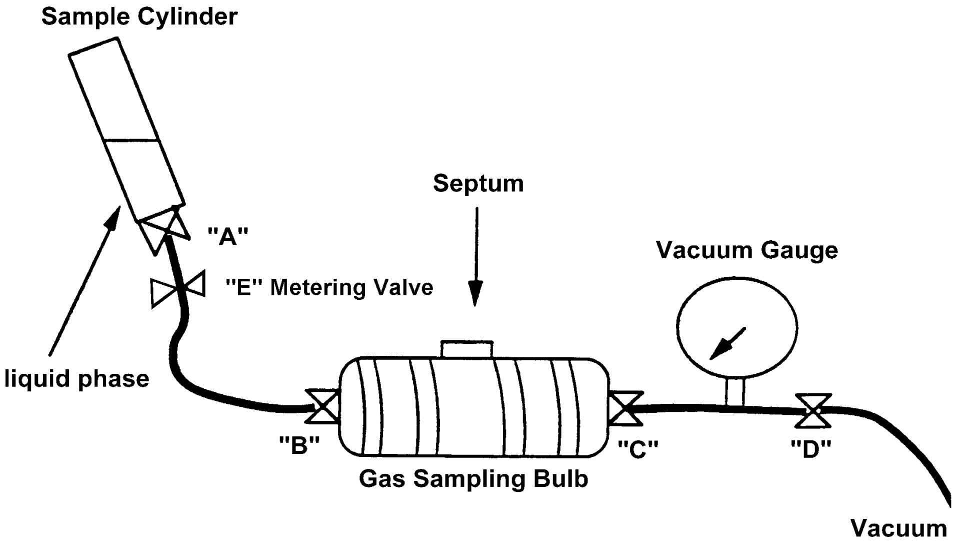
Section 12. References

1. Vapor-liquid equilibrium studies regarding liquid phase depletion have been examined for R-407C, R-408A, R-409A, and R-416A, unpublished work, National Refrigerants, Inc., Rosenhayn, NJ.

2. Air-Conditioning and Refrigeration Institute, *Appendix C to ARI Standard 700-95: Analytical Procedures for ARI Standard 700-95*, 4301 North Fairfax Drive, Arlington, Virginia 22203.

3. *Methods Development for Organic Contaminant Determination in Fluorocarbon Refrigerant Azeotropes and Blends*, Final Report, Nov. 30, 1997, ARTI MCLR Project Number 665-54600, Integral Sciences Inc., Columbus, OH.

Figure 1. Apparatus Used for Sampling Calibration Standards and Samples



Part 16

R-401 Composition GC Method Data Sheet

Section 1. Gas Chromatographic Conditions

Detector	TCD, Low Sensitivity
Carrier Gas	30 mL He/Min.
Initial Column Temp.	75°C
Initial Hold	5 Min.
Program	20 K/Min.
Final Column Temp.	175°C
Post Hold	5 Min.
Sample	1.0 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Table 1. Blend Component Weight %

R-401 Blend	Wt%			
Component	R-401A	R-401B	Tolerance	GC Retention Time
R-152a	13.0	11.0	+.50,-1.50	6.20
R-22	53.0	61.0	±2.0	6.60
R-124	34.0	28.0	±1.0	10.55

Section 2. Precision, and Accuracy

Statistical parameters for each impurity are listed below. The data was obtained by analyzing an R-401 calibration mixture 7 times during one day by one operator.

Table 2. Component Statistical Parameters

R-401 Component	95% CL	Relative Mean Error, wt%
R-22	±0.03%	0.06
R-152a	±0.03%	0.22
R-124	±0.06%	0.17

Section 3. Calibration Standard Preparation

The following table is to be used in conjunction with Part 15, *The Determination of the Component Concentrations of Refrigerant 400 and 500 Series Blends and Azeotropes by Gas Chromatography.*

Table 3. Blend Calibration Standard Preparation

R-401 Component	Amount to Weigh (g)	Approximate Weight %
R-124	307	31
R-152a	119	12
R-22	564	57

RT (Min)	Comp.
2.33	Air (1)
6.19	152a
6.61	22
10.20	124a
10.55	124

(1) Ignored in Calculations.

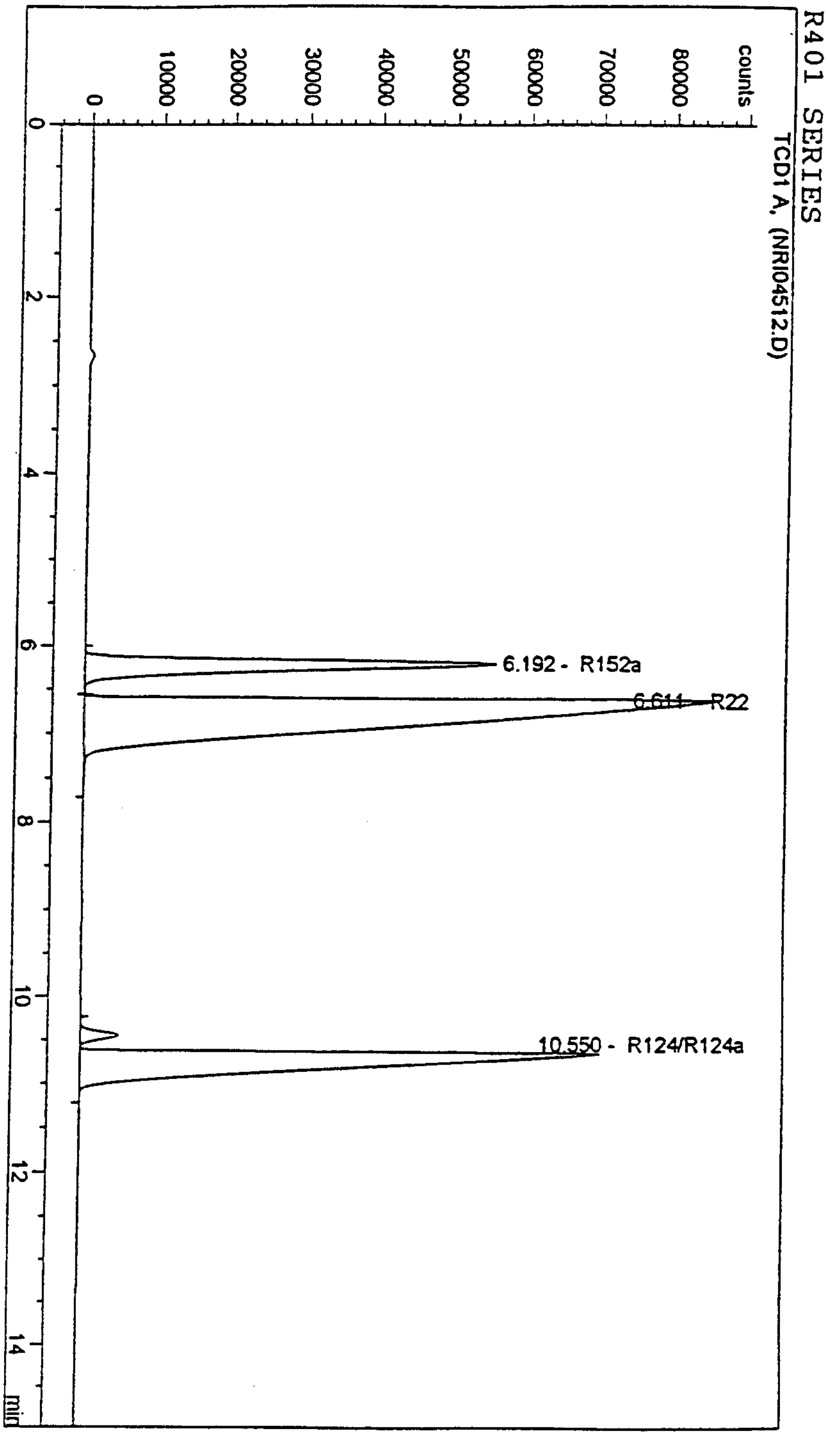


Figure 1. Gas Chromatogram of R-401

Part 17

R-402 Composition GC Method Data Sheet

Section 1. Gas Chromatographic Conditions

Detector	TCD, Low Sensitivity
Carrier Gas	30 mL He/Min.
Initial Column Temp.	75°C
Initial Hold	7 Min.
Program	25 K/Min.
Final Column Temp.	150°C
Post Hold	2 Min.
Sample	1.0 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Table 1. Blend Component Wt %

R-402 Blend	Wt%			
Component	R-402A	R-402B	Tolerance	GC Retention Time (Min)
R-125	60.0	38.0	±2.0	6.00
R-22	38.0	60.0	±2.0	6.95
HC-290	2.0	2.0	±1.0	9.40

Section 2. Precision, and Accuracy

Statistical parameters for each impurity are listed below. The data was obtained by analyzing an R-402 calibration mixture 7 times during one day by one operator.

Table 2. Component Statistical Parameters

R-402 Component	95% CL	Relative Mean Error, wt%
R-125	±0.010%	0.08
R-22	±0.009%	0.07
HC-290	±0.001%	0.07

Section 3. Calibration Standard Preparation

The following table is to be used in conjunction with Part 15, *The Determination of the Component Concentrations of Refrigerant 400 and 500 Series Blends and Azeotropes by Gas Chromatography.*

Table 3. Blend Calibration Standard Preparation

R-402 Component	Amount to Weigh (g)		Approximate Weight %	
	R-402A	R-402B	R-402A	R-402B
HC-290	19	19	2	2
R-22	364	578	38	60
R-125	575	366	60	38

RT (Min)	Comp.
2.33	Air (1)
5.97	125
6.94	22
9.42	290

(1) Ignored in Calculations.

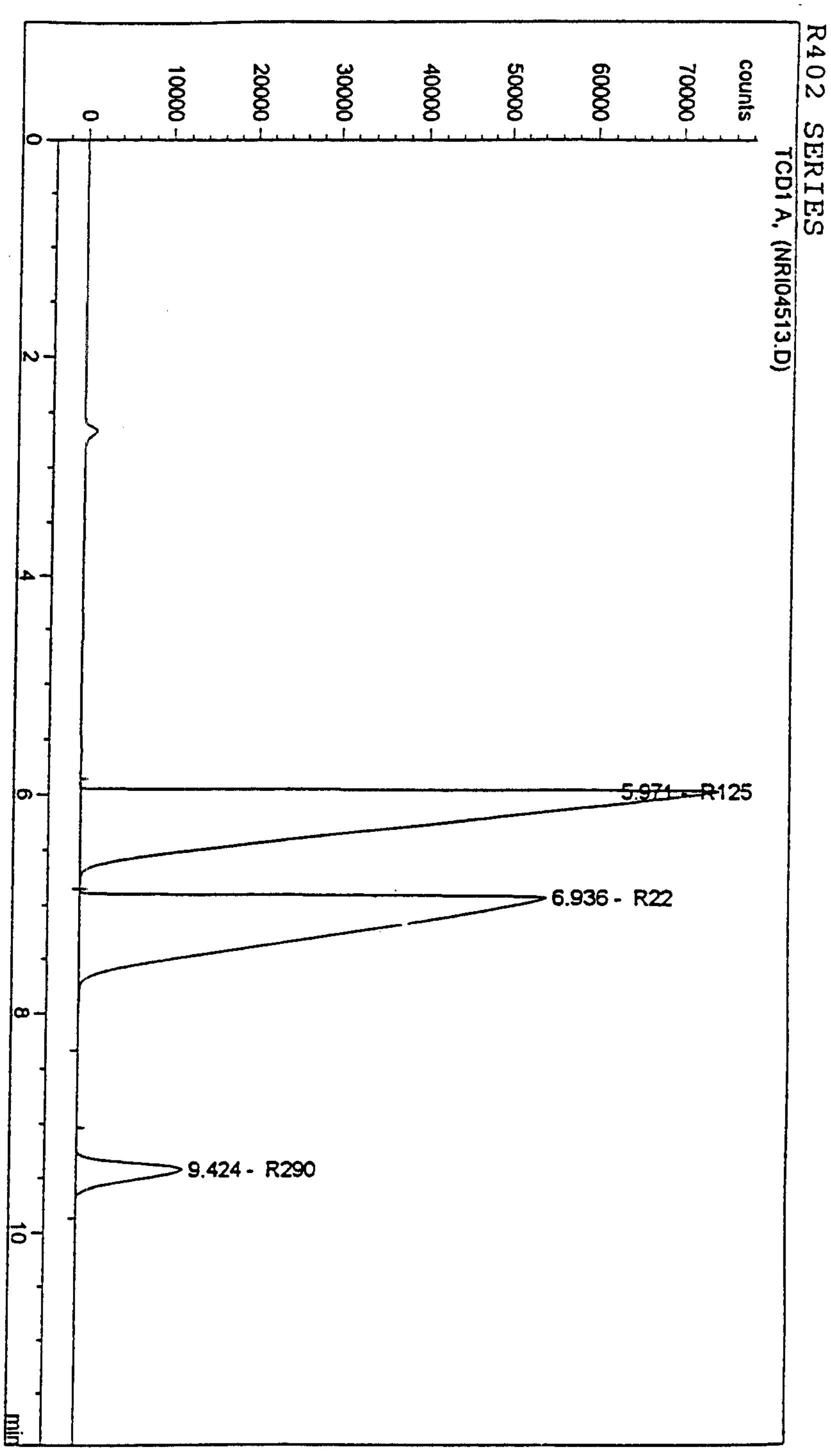


Figure 1. Gas Chromatogram of R-402

Part 18

R-403B Composition GC Method Data Sheet

Section 1. Gas Chromatographic Conditions

Detector	TCD, Low Sensitivity
Carrier Gas	30 mL He/Min.
Initial Column Temp.	100°C (isothermal)
Initial Hold	-- Min.
Program	-- K/Min.
Final Column Temp.	100°C
Post Hold	10 Min.
Sample	0.25 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Table 1. Blend Component Wt. %

R-403 Blend	Wt%		
Component	R-403B (%)	Tolerance (%)	GC Retention Time (Min.)
R-22	56.0	±2.0	5.20
FC-218	39.0	±2.0	6.20
HC-290	5.0	±0.2	7.20

Section 2. Precision, and Accuracy

Statistical parameters for each impurity are listed below. The data was obtained by analyzing an R-403B calibration mixture 7 times during one day by one operator.

Table 2. Component Statistical Parameters

R-403B Component	95% CL	Relative Mean Error, wt%
R-22	±0.018%	0.09
FC-218	±0.019%	0.10
HC-290	±0.024%	1.62

Section 3. Calibration Standard Preparation

The following table is to be used in conjunction with Part 15, *The Determination of the Component Concentrations of Refrigerant 400 and 500 Series Blends and Azeotropes by Gas Chromatography.*

Table 3. Blend Calibration Standard Preparation

R-403B Component	Amount to Weigh (g)	Approximate Weight %
FC-218	374	39
HC-290	48	5
R-22	537	56

RT (Min)	Comp.
2.60	Air (1)
5.22	22
6.23	218
7.19	290
(1) Ignored in Calculations.	

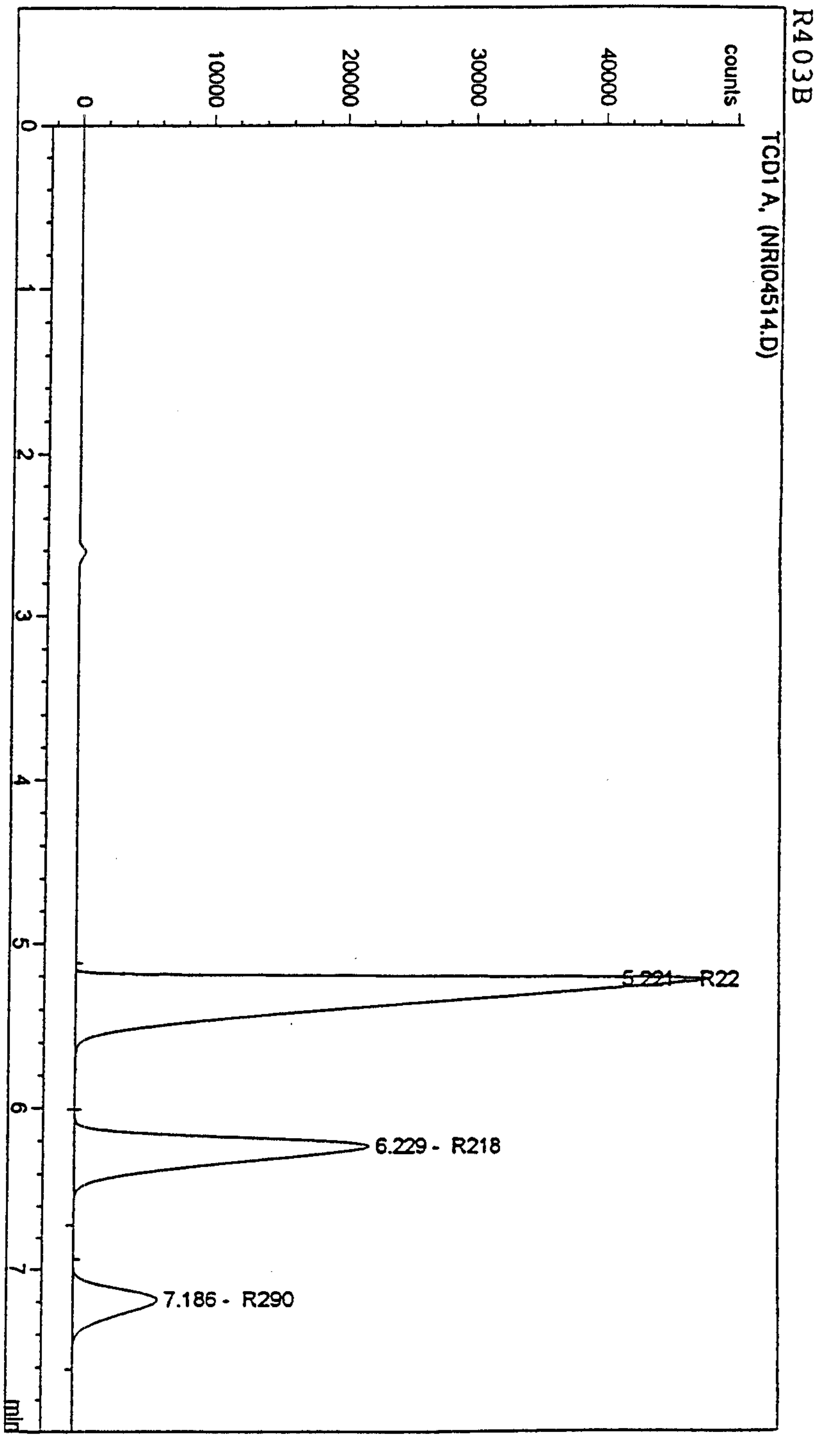


Figure 1. Gas Chromatogram of R-403B

Part 19

R-404A Composition GC Method Data Sheet

Section 1. Gas Chromatographic Conditions

Detector	TCD, Low Sensitivity
Carrier Gas	30 mL He/Min.
Initial Column Temp.	35°C
Initial Hold	8 Min.
Program	15 K/Min.
Final Column Temp.	75°C
Post Hold	5 Min.
Sample	0.5 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Table 1. Blend Component Wt. %

R-404 Blend	Wt%		
Component	R-404A (%)	Tolerance (%)	GC Retention Time (Min.)
R-125	44.0	±1.0	10.90
R-143a	52.0	±1.0	9.55
R-134a	4.0	±1.0	11.60

Section 2. Precision, and Accuracy

Statistical parameters for each impurity are listed below. The data was obtained by analyzing an R-404A calibration mixture 7 times during one day by one operator.

Table 2. Component Statistical Parameters

R-404A Component	95% CL	Relative Mean Error, wt%
R-125	±0.017%	0.023
R-143a	±0.024%	0.097
R-134a	±0.038%	0.740

Section 3. Calibration Standard Preparation

The following table is to be used in conjunction with Part 15, *The Determination of the Component Concentrations of Refrigerant 400 and 500 Series Blends and Azeotropes by Gas Chromatography.*

Table 3. Blend Calibration Standard Preparation

R-404A Component	Amount to Weigh (g)	Approximate Weight %
R-134a	35	4
R-143a	454	52
R-125	384	44

RT (Min)	Comp.
2.80	Air (1)
9.57	143a
10.91	125
11.60	134a
(1) Ignored in Calculations.	

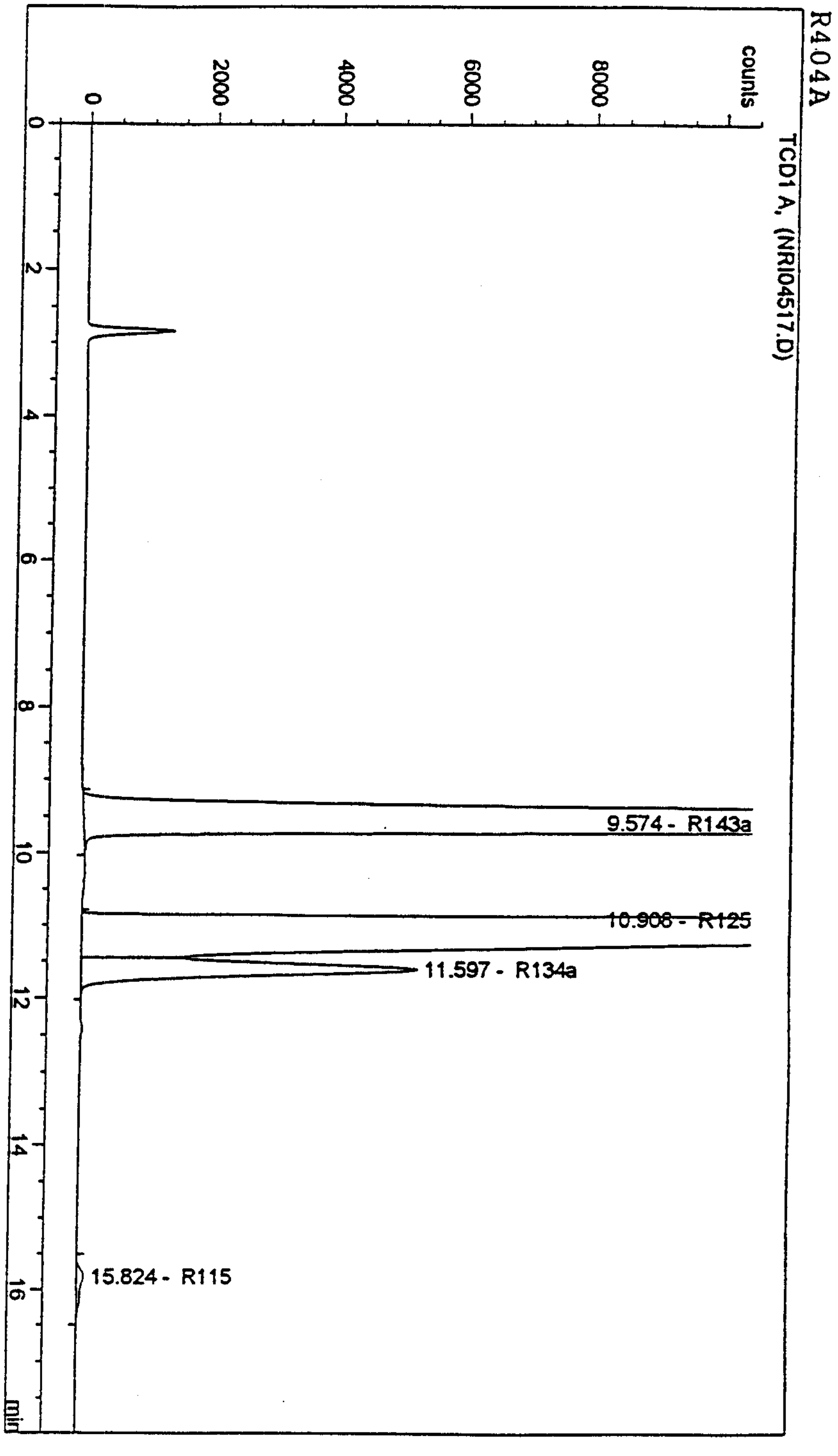


Figure 1. Gas Chromatogram of R-404A

Part 20

R-406A Composition GC Method Data Sheet

Section 1. Gas Chromatographic Conditions

Detector	TCD, Low Sensitivity
Carrier Gas	30 mL He/Min.
Initial Column Temp.	150°C (Isothermal)
Initial Hold	-- Min.
Program	-- K/Min.
Final Column Temp.	150°C
Post Hold	9 Min.
Sample	1.0 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Table 1. Blend Component Wt. %

R-406 Blend	Wt%		
Component	R-406A (%)	Tolerance (%)	GC Retention Time (Min.)
R-142b	41.0	±1.0	3.90
HC-600a	4.0	±1.0	5.30
R-22	55.0	±2.0	2.85

Section 2. Precision, and Accuracy

Statistical parameters for each impurity are listed below. The data was obtained by analyzing an R-406A calibration mixture 7 times during one day by one operator.

Table 2. Component Statistical Parameters

R-406A Component	95% CL	Relative Mean Error, wt%
R-142b	±0.060%	-0.12
HC-600a	±0.009%	0.25
R-22	±0.023%	0.07

Section 3. Calibration Standard Preparation

The following table is to be used in conjunction with Part 15, *The Determination of the Component Concentrations of Refrigerant 400 and 500 Series Blends and Azeotropes by Gas Chromatography.*

Table 3. Blend Calibration Standard Preparation

R-406A Component	Amount to Weigh (g)	Approximate Weight %
HC-600a	37	4
R-142b	380	41
R-22	509	55

RT (Min)	Comp.
2.20	Air (1)
2.84	22
3.88	142b
5.28	600a
(1) Ignored in Calculations.	

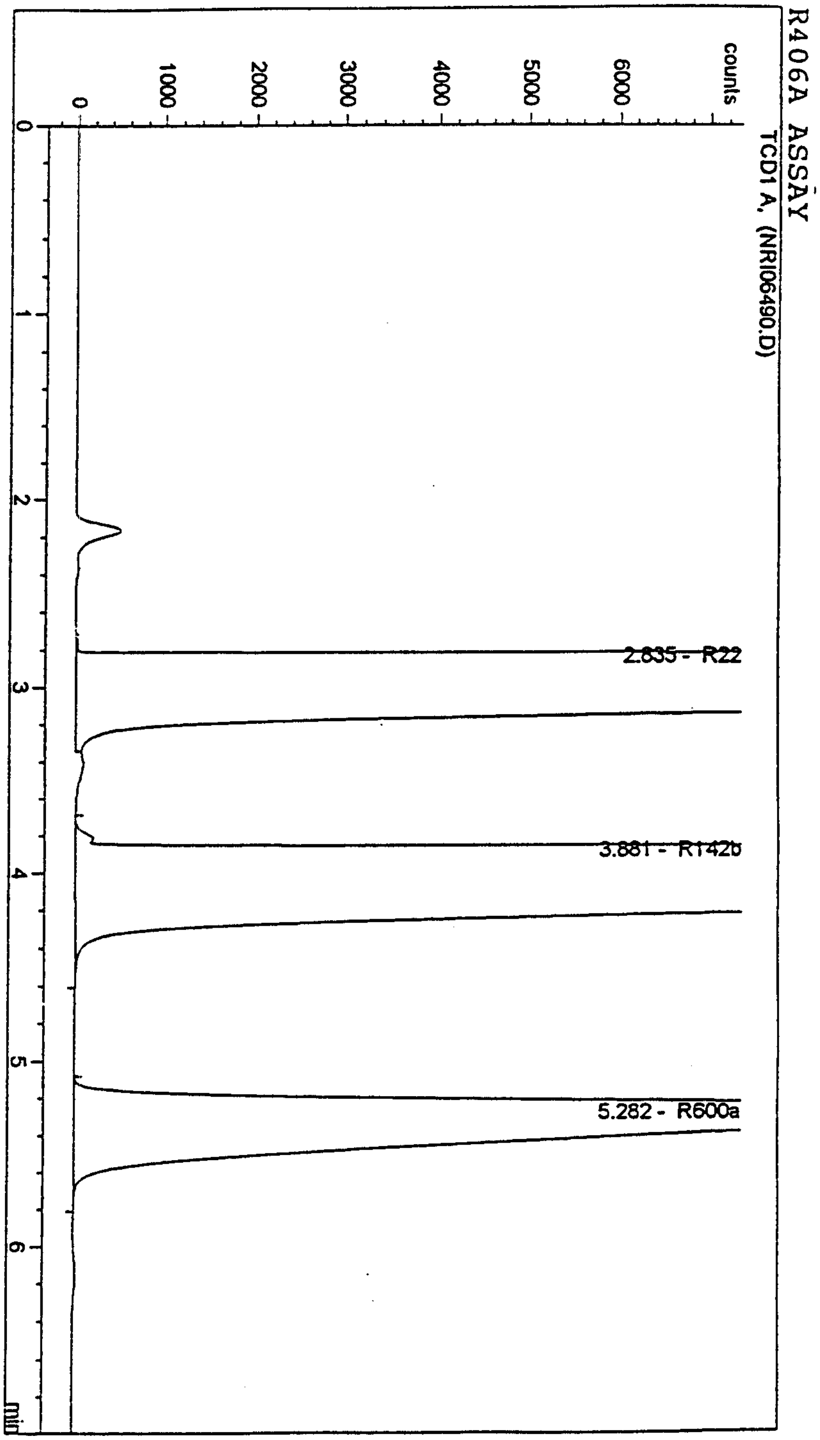


Figure 1. Gas Chromatogram of R-406A

Part 21

R-407 Composition GC Method Data Sheet

Section 1. Gas Chromatographic Conditions

Detector	TCD, Low Sensitivity
Carrier Gas	30 mL He/Min.
Initial Column Temp.	50°C (Isothermal)
Initial Hold	-- Min.
Program	-- K/Min.
Final Column Temp.	50°C
Post Hold	15 Min.
Sample	0.25 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Table 1A. Blend Component Wt. % for R-407A

R-407A Blend	Wt%		
Component	R-407A (%)	Tolerance (%)	GC Retention Time (Min.)
R-32	20.0	±1.0	4.05
R-125	40.0	±2.0	9.30
R-134a	40.0	±2.0	9.90

Table 1B. Blend Component Wt. % for R-407B

R-407B Blend	Wt%		
Component	R-407B (%)	Tolerance (%)	GC Retention Time (Min.)
R-32	10.0	±1.0	4.05
R-125	70.0	±2.0	9.30
R-134a	20.0	±2.0	9.90

Table 1C. Blend Component Wt. % for R-407C

R-407C Blend	Wt%		
Component	R-407C (%)	Tolerance (%)	GC Retention Time (Min.)
R-32	23.0	±1.0	4.05
R-125	25.0	±2.0	9.30
R-134a	52.0	±2.0	9.90

Section 2. Precision, and Accuracy

Statistical parameters for each impurity are listed below. The data was obtained by analyzing an R-407C calibration mixture 7 times during one day by one operator.

Table 2. Component Statistical Parameters

R-407C Component	95% CL	Relative Mean Error, wt%
R-32	±0.020%	0.13
R-125	±0.052%	0.06
R-134a	±0.059%	0.03

Section 3. Calibration Standard Preparation

The following table is to be used in conjunction with Part 15, *The Determination of the Component Concentrations of Refrigerant 400 and 500 Series Blends and Azeotropes by Gas Chromatography.*

Table 3. Blend Calibration Standard Preparation

R-407A Component	Amount to Weigh (g)	Approximate Weight %
R-134a	380	40
R-32	190	20
R-125	380	40
R-407B Component	Amount to Weigh (g)	Approximate Weight %
R-134a	194	20
R-32	97	10
R-125	680	70
R-407C Component	Amount to Weigh (g)	Approximate Weight %
R-134a	502	52
R-32	222	23
R-125	241	25

RT (Min)	Comp.
2.80	Air (1)
4.07	32
9.32	125
9.90	134a

(1) Ignored in Calculations.

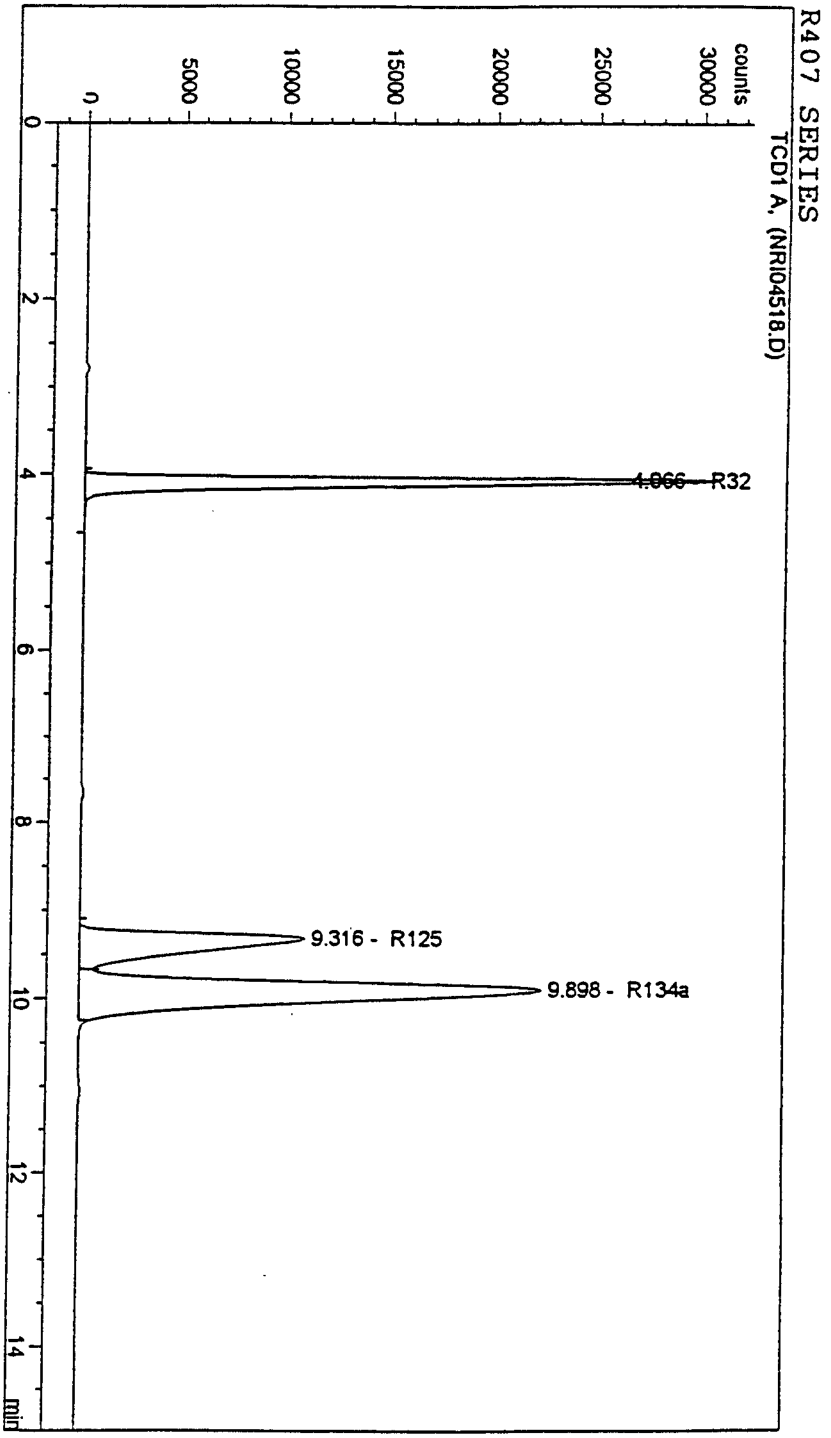


Figure 1. Gas Chromatogram of R-407

Part 22

R-408A Composition GC Method Data Sheet

Section 1. Gas Chromatographic Conditions

Detector	TCD, Low Sensitivity
Carrier Gas	30 mL He/Min.
Initial Column Temp.	40°C (isothermal)
Initial Hold	6 Min.
Program	10 K/Min.
Final Column Temp.	130°C
Post Hold	0 Min.
Sample	1.0 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Table 1. Blend Component Wt %

R-408A Blend	Wt%		
Component	R-408A (%)	Tolerance (%)	GC Retention Time (Min.)
R-143a	46.0	±1.0	8.45
R-125	7.0	±2.0	9.85
R-22	47.0	±2.0	10.55

Section 2. Precision, and Accuracy

Statistical parameters for each impurity are listed below. The data was obtained by analyzing an R-408A calibration mixture 7 times during one day by one operator.

Table 2. Component Statistical Parameters

R-408A Component	95% CL	Relative Mean Error, wt%
R-125	±0.015%	0.001
R-143a	±0.034%	0.044
R-22	±0.023%	0.044

Section 3. Calibration Standard Preparation

The following table is to be used in conjunction with Part 15, *The Determination of the Component Concentrations of Refrigerant 400 and 500 Series Blends and Azeotropes by Gas Chromatography.*

Table 3. Blend Calibration Standard Preparation

R-408A Component	Amount to Weigh (g)	Approximate Weight %
R-143a	407	46
R-22	416	47
R-125	62	7

RT (Min)	Comp.
2.80	Air (1)
8.45	143a
9.85	125
10.55	22

(1) Ignored in Calculations.

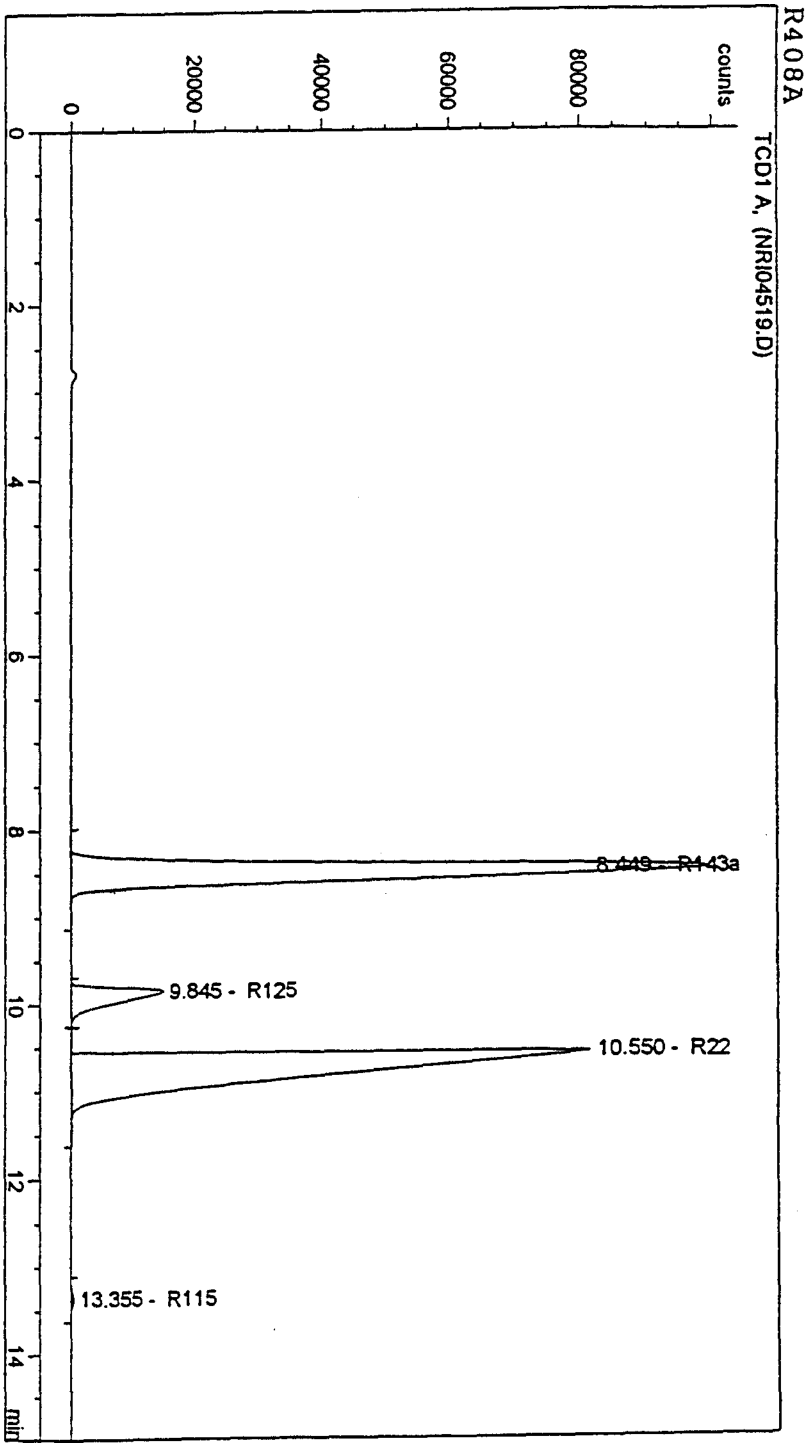


Figure 1. Gas Chromatogram of R-408A

Part 23

R-409 Composition GC Method Data Sheet

Section 1. Gas Chromatographic Conditions

Detector	TCD, Low Sensitivity
Carrier Gas	30 mL He/Min.
Initial Column Temp.	100°C (Isothermal)
Initial Hold	-- Min.
Program	-- K/Min.
Final Column Temp.	100°C
Post Hold	15 Min.
Sample	0.20 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Table 1. Blend Component Wt. %

R-409 Blend	Wt%			
Component	R-409A (%)	R-409B (%)	Tolerance (%)	GC Retention Time (Min)
R-22	60.0	65.0	±2.0	5.20
R-142b	15.0	10.0	±1.0	9.50
R-124	25.0	25.0	±2.0	11.00

Section 2. Precision, and Accuracy

Statistical parameters for each impurity are listed below. The data was obtained by analyzing an R-409 calibration mixture 7 times during one day by one operator.

Table 2. Component Statistical Parameters

R-409 Component	95% CL	Relative Mean Error, wt%
R-22	±0.027%	-0.03
R-142b	±0.039%	-0.13
R-124	±0.027%	0.17

Section 3. Calibration Standard Preparation

The following table is to be used in conjunction with Part 15, *The Determination of the Component Concentrations of Refrigerant 400 and 500 Series Blends and Azeotropes by Gas Chromatography.*

Table 3. Blend Calibration Standard Preparation

R-409 Component	Amount to Weigh (g)	Approximate Weight %
R-124	255	25
R-142b	153	15
R-22	611	60

RT (Min)	Comp.
2.65	Air (1)
5.19	22
9.49	142b
10.55	124a
11.34	124

(1) Ignored in Calculations.

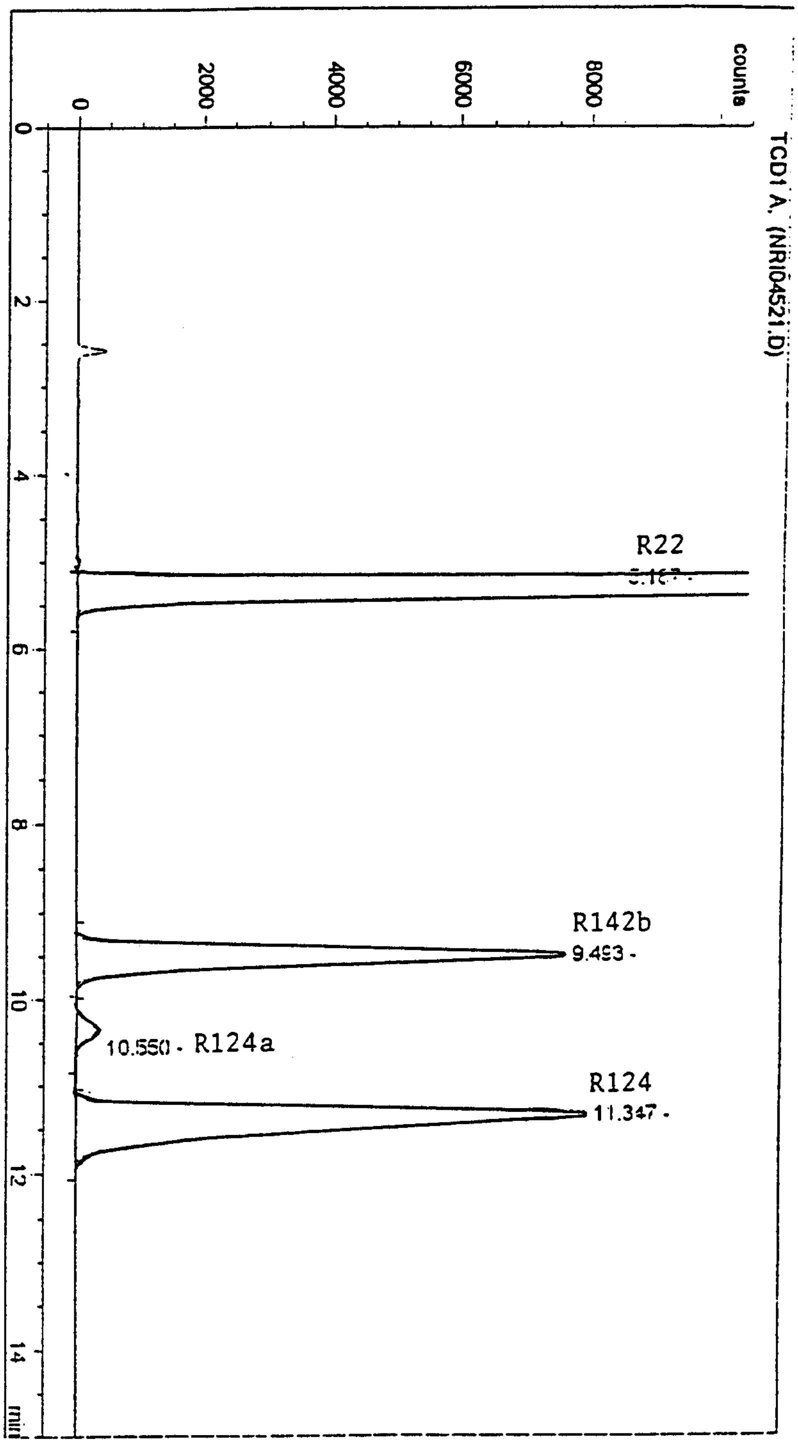


Figure 1. Gas Chromatogram of R-409

Part 24

R-410 Composition GC Method Data Sheet

Section 1. Gas Chromatographic Conditions

Detector	TCD, Low Sensitivity
Carrier Gas	30 mL He/Min.
Initial Column Temp.	100°C (Isothermal)
Initial Hold	-- Min.
Program	-- K/Min.
Final Column Temp.	100°C
Post Hold	7 Min.
Sample	1.0 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Table 1. Blend Component Wt. %

R-410 Blend	Wt%			
Component	R-410A (%)	R-410B (%)	Tolerance (%)	GC Retention Time (Min)
R-32	50.0	45.0	-1.50, +.50, ±1.0	3.00
R-125	50.0	55.0	-.50, +1.50, ±1.0	4.60

Section 2. Precision, and Accuracy

Statistical parameters for each impurity are listed below. The data was obtained by analyzing an R-410B calibration mixture 7 times during one day by one operator.

Table 2. Component Statistical Parameters

R-410 Component	95% CL	Relative Mean Error, wt%
R-32	±0.030%	0.047
R-125	±0.025%	0.13

Section 3. Calibration Standard Preparation

The following table is to be used in conjunction with Part 15, *The Determination of the Component Concentrations of Refrigerant 400 and 500 Series Blends and Azeotropes by Gas Chromatography.*

Table 3. Blend Calibration Standard Preparation

R-410 Component	Amount to Weigh (g)	Approximate Weight %
R-32	430	48
R-125	465	52

RT (Min)	Comp.
2.56	Air (1)
3.00	32
4.60	125

(1) Ignored in Calculations.

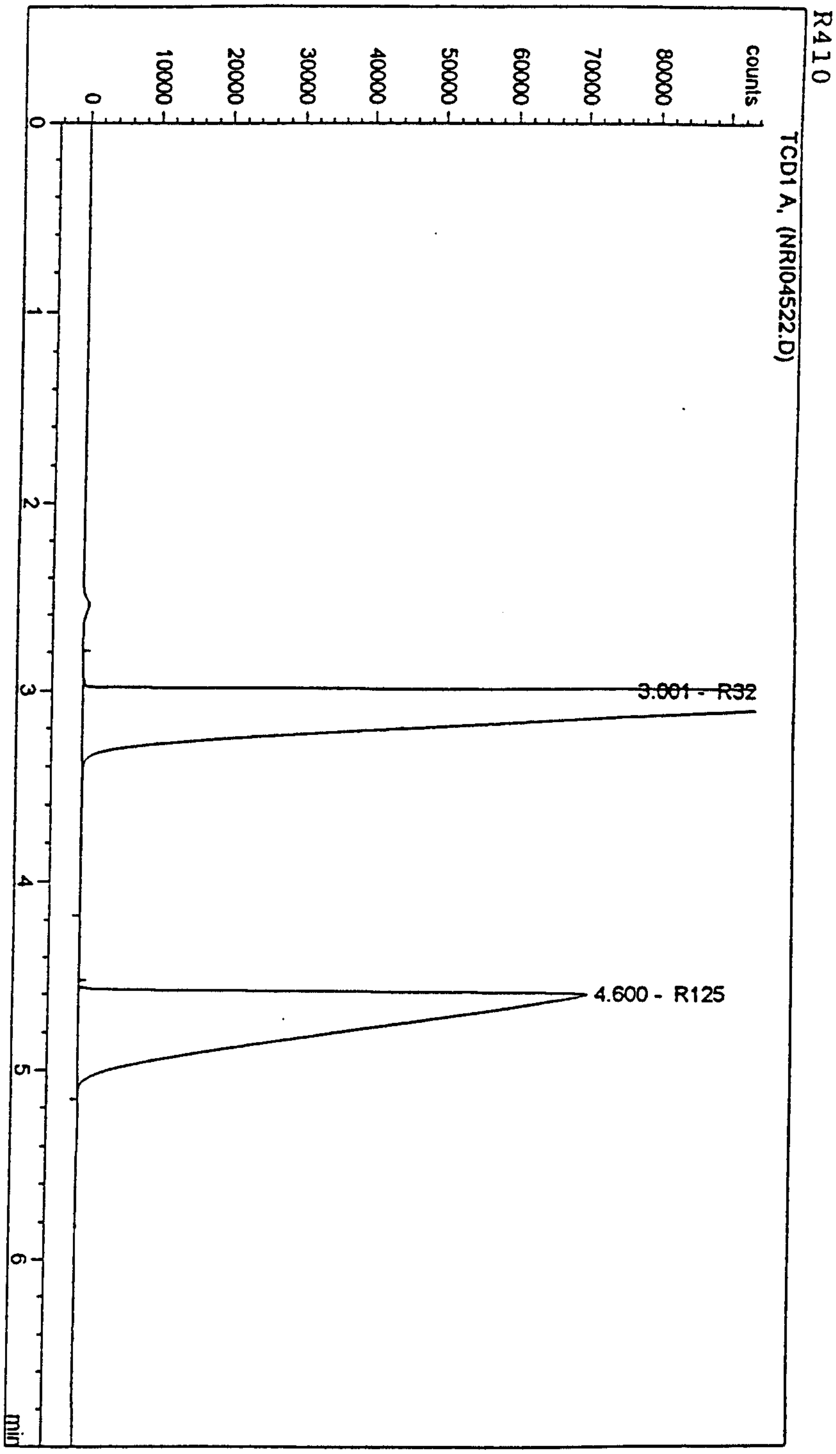


Figure 1. Gas Chromatogram of R-410

Part 25

R-411 Composition GC Method Data Sheet

Section 1. Gas Chromatographic Conditions

Detector	TCD, Low Sensitivity
Carrier Gas	30 mL He/Min.
Initial Column Temp.	75°C (Isothermal)
Initial Hold	-- Min.
Program	-- K/Min.
Final Column Temp.	75°C
Post Hold	12 Min.
Sample	1.0 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Table 1A. Blend Component Wt. % for R-411A

R-411A Blend	Wt%		
Component	R-411A (%)	Tolerance (%)	GC Retention Time (Min.)
R-152	11.0	+0, -1.0	6.15
HC-1270	1.5	+0, -1.0	6.60
R-22	87.5	+2.0, -0	9.55

Table 1B. Blend Component Wt. % for R-411B

R-411B Blend	Wt%		
Component	R-411A (%)	Tolerance (%)	GC Retention Time (Min.)
R-152	3.0	+0, -1.0	6.15
HC-1270	3.0	+0, -1.0	6.60
R-22	94.0	+2.0, -0	9.55

Table 1C. Blend Component Wt. % for R-411C

R-411C Blend	Wt%		
Component	R-411A (%)	Tolerance (%)	GC Retention Time (Min.)
R-152	1.5	+0, -1.0	6.15
HC-1270	3.0	+0, -1.0	6.60
R-22	95.5	+2.0, -0	9.55

Section 2. Precision, and Accuracy

Statistical parameters for each impurity are listed below. The data was obtained by analyzing an R-406A calibration mixture 7 times during one day by one operator.

Table 2. Component Statistical Parameters

R-411 Component	95% CL	Relative Mean Error, wt%
R-152a	±0.004%	0
HC-1270	±0.002%	0.09
R-22	±0.002%	0

Section 3. Calibration Standard Preparation

The following table is to be used in conjunction with Part 15, *The Determination of the Component Concentrations of Refrigerant 400 and 500 Series Blends and Azeotropes by Gas Chromatography.*

Table 3. Blend Calibration Standard Preparation

R-411A Component	Amount to Weigh (g)	Approximate Weight %
HC-1270	28	3.0
R-152a	46	5.0
R-22	855	92.0

RT (Min)	Comp.
2.67	Air (1)
6.17	152a
6.61	22
9.55	1270

(1) Ignored in Calculations.

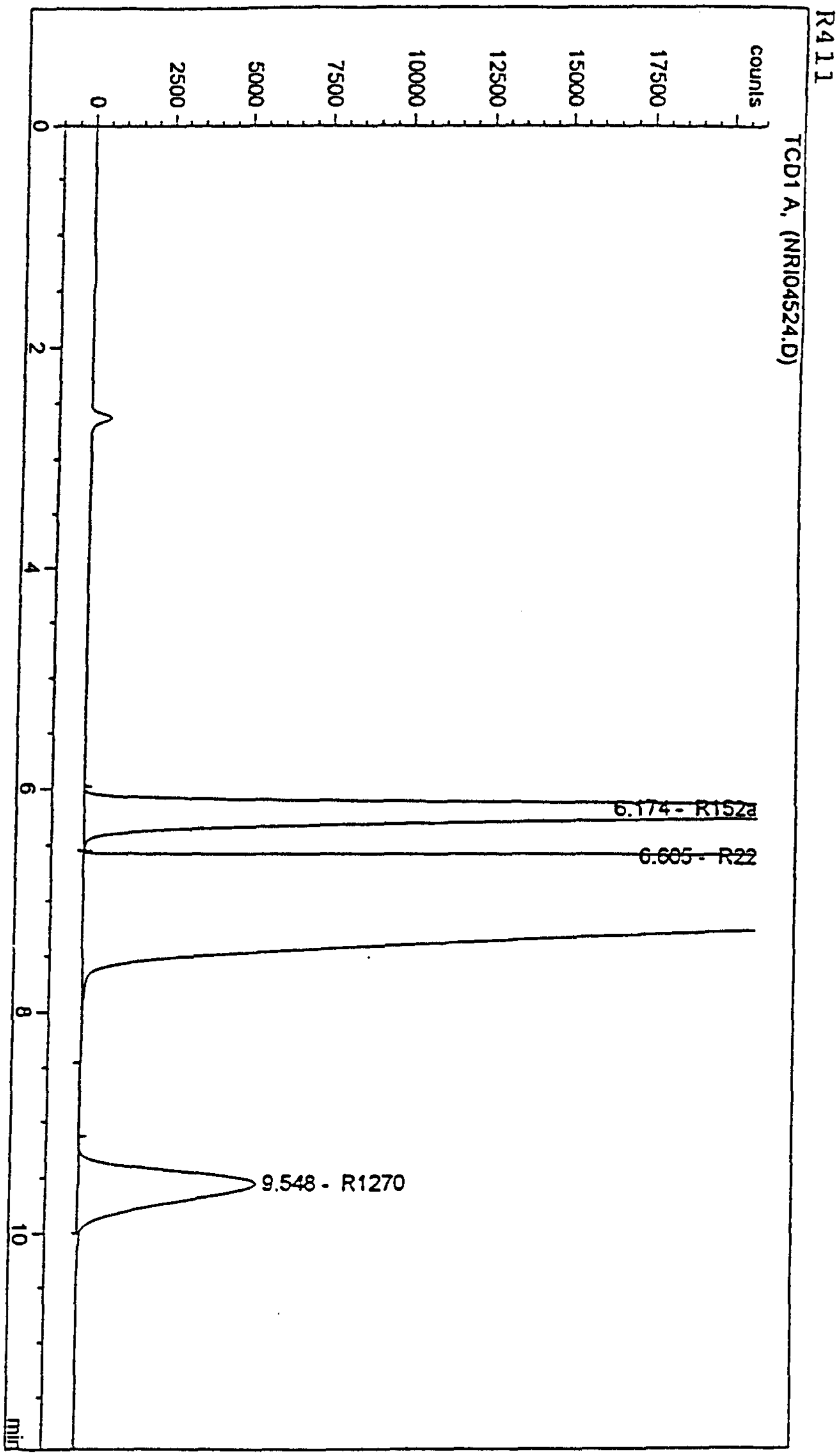


Figure 1. Gas Chromatogram of R-411

Part 26

R-412A Composition GC Method Data Sheet

Section 1. Gas Chromatographic Conditions

Detector	TCD, Low Sensitivity
Carrier Gas	30 mL He/Min.
Initial Column Temp.	100°C (isothermal)
Initial Hold	-- Min.
Program	-- K/Min.
Final Column Temp.	100°C
Post Hold	10 Min.
Sample	0.5 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Table 1. Blend Component Wt. %

R-412A Blend	Wt%		
Component	R-412A (%)	Tolerance (%)	GC Retention Time (Min.)
R-22	70.0	±2.0	3.9
FC-218	5.0	±2.0	4.5
R-142b	25.0	±1.0	6.9

Section 2. Precision, and Accuracy

Statistical parameters for each impurity are listed below. The data was obtained by analyzing an R-412A calibration mixture 7 times during one day by one operator.

Table 2. Component Statistical Parameters

R-412A Component	95% CL	Relative Mean Error, wt%
R-22	±0.034%	0
FC-218	±0.014%	0.23
R-142b	±0.037%	0.4

Section 3. Calibration Standard Preparation

The following table is to be used in conjunction with Part 15, *The Determination of the Component Concentrations of Refrigerant 400 and 500 Series Blends and Azeotropes by Gas Chromatography*.

Table 3. Blend Calibration Standard Preparation

R-412A Component	Amount to Weigh (g)	Approximate Weight %
R-142b	244	25
FC-218	49	5
R-22	682	70

RT (Min)	Comp.
2.35	Air (1)
3.93	22
4.53	218
6.89	142b
(1) Ignored in Calculations.	

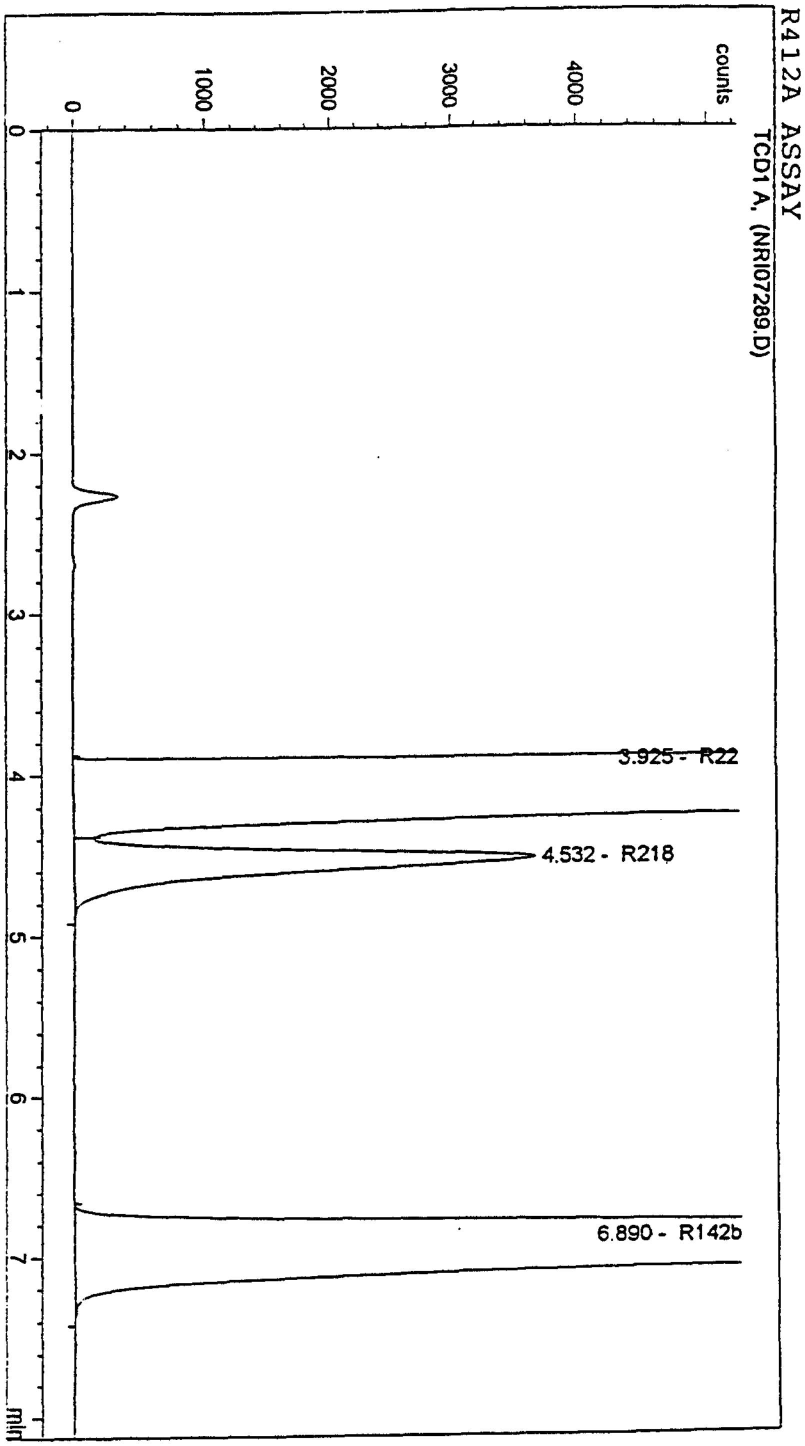


Figure 1. Gas Chromatogram of R-412

Part 27

R-500 Composition GC Method Data Sheet

Section 1. Gas Chromatographic Conditions

Detector	TCD, Low Sensitivity
Carrier Gas	30 mL He/Min.
Initial Column Temp.	175°C (isothermal)
Initial Hold	-- Min.
Program	-- K/Min.
Final Column Temp.	175°C
Post Hold	5 Min.
Sample	1.0 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Table 1. Blend Component Wt. %

R-500	Wt%		
Component	R-500 (%)	Tolerance (%)	GC Retention Time (Min.)
R-152a	26.2	±1.0	2.60
R-12	73.8	±1.0	3.15

Section 2. Precision, and Accuracy

Statistical parameters for each impurity are listed below. The data was obtained by analyzing an R-500 calibration mixture 7 times during one day by one operator.

Table 2. Component Statistical Parameters

R-500 Component	95% CL	Relative Mean Error, wt%
R-12	±0.011%	0.05
R-152a	±0.011%	0.16

Section 3. Calibration Standard Preparation

The following table is to be used in conjunction with Part 15, *The Determination of the Component Concentrations of Refrigerant 400 and 500 Series Blends and Azeotropes by Gas Chromatography*.

Table 3. Blend Calibration Standard Preparation

R-500 Component	Amount to Weigh (g)	Approximate Weight %
R-12	718	73.8
R-152a	255	26.2

RT (Min)	Comp.
2.18	Air (1)
2.62	152a
3.17	22
(1) Ignored in Calculations.	

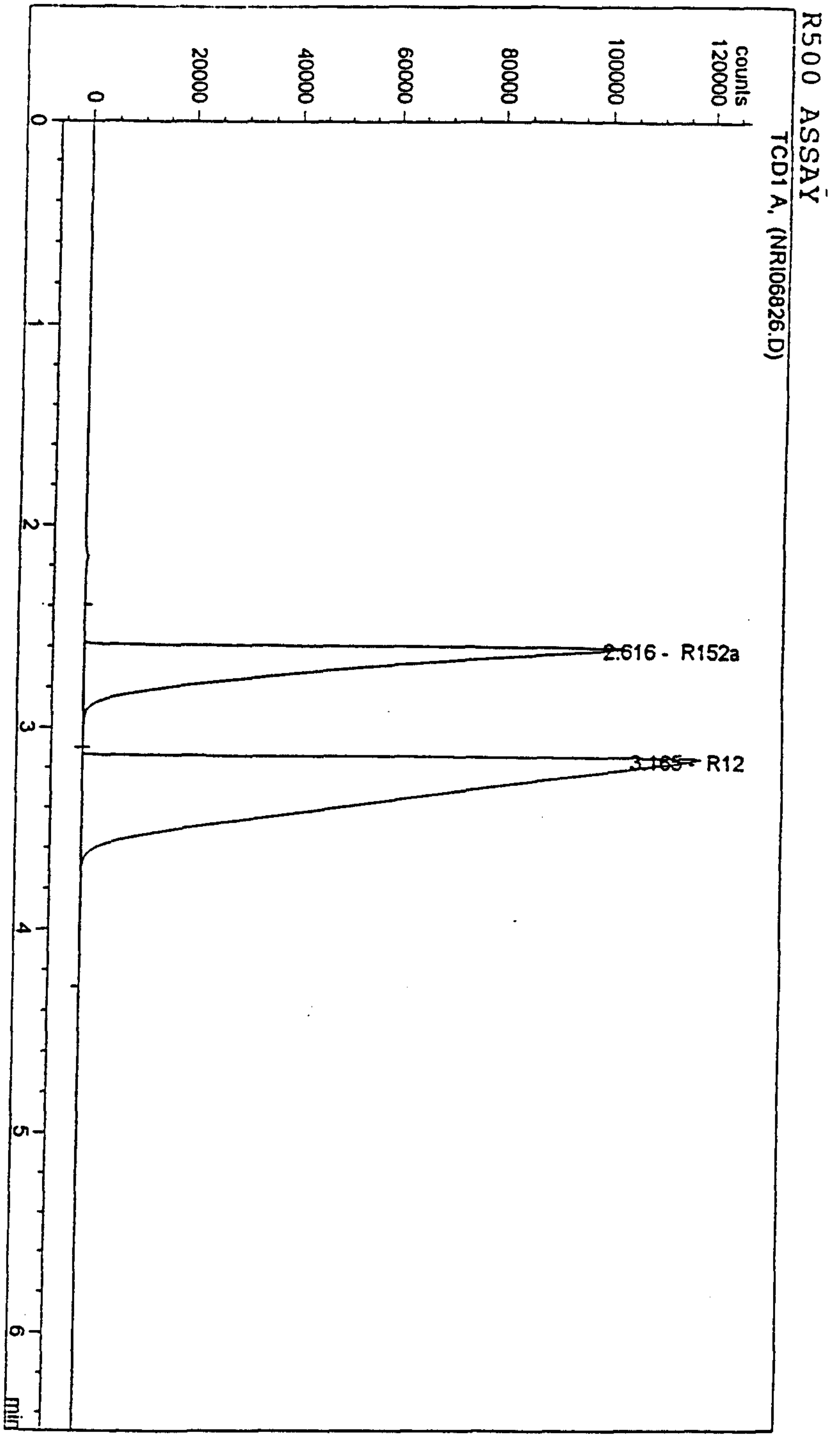


Figure 1. Gas Chromatogram of R-500

Part 28

R-502 Composition GC Method Data Sheet

Section 1. Gas Chromatographic Conditions

Detector	TCD, Low Sensitivity
Carrier Gas	30 mL He/Min.
Initial Column Temp.	150°C (isothermal)
Initial Hold	-- Min.
Program	-- K/Min.
Final Column Temp.	150°C
Post Hold	6 Min.
Sample	1.0 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Table 1. Blend Component Wt. %

R-502	Wt%		
Component	R-502 (%)	Tolerance (%)	GC Retention Time (Min.)
R-22	48.8	±4.0	2.85
R-115	51.2	±4.0	3.30

Section 2. Precision, and Accuracy

Statistical parameters for each impurity are listed below. The data was obtained by analyzing an R-502 calibration mixture 7 times during one day by one operator.

Table 2. Component Statistical Parameters

R-502 Component	95% CL	Relative Mean Error, Wt%
R-22	±0.062%	0.06
R-115	±0.062%	0.06

Section 3. Calibration Standard Preparation

The following table is to be used in conjunction with Part 15, *The Determination of the Component Concentrations of Refrigerant 400 and 500 Series Blends and Azeotropes by Gas Chromatography*.

Table 3. Blend Calibration Standard Preparation

R-502 Component	Amount to Weigh (g)	Approximate Weight %
R-22	495	48.8
R-115	519	51.2

RT (Min)	Comp.
2.20	Air (1)
2.85	22
3.31	115
(1) Ignored in Calculations.	

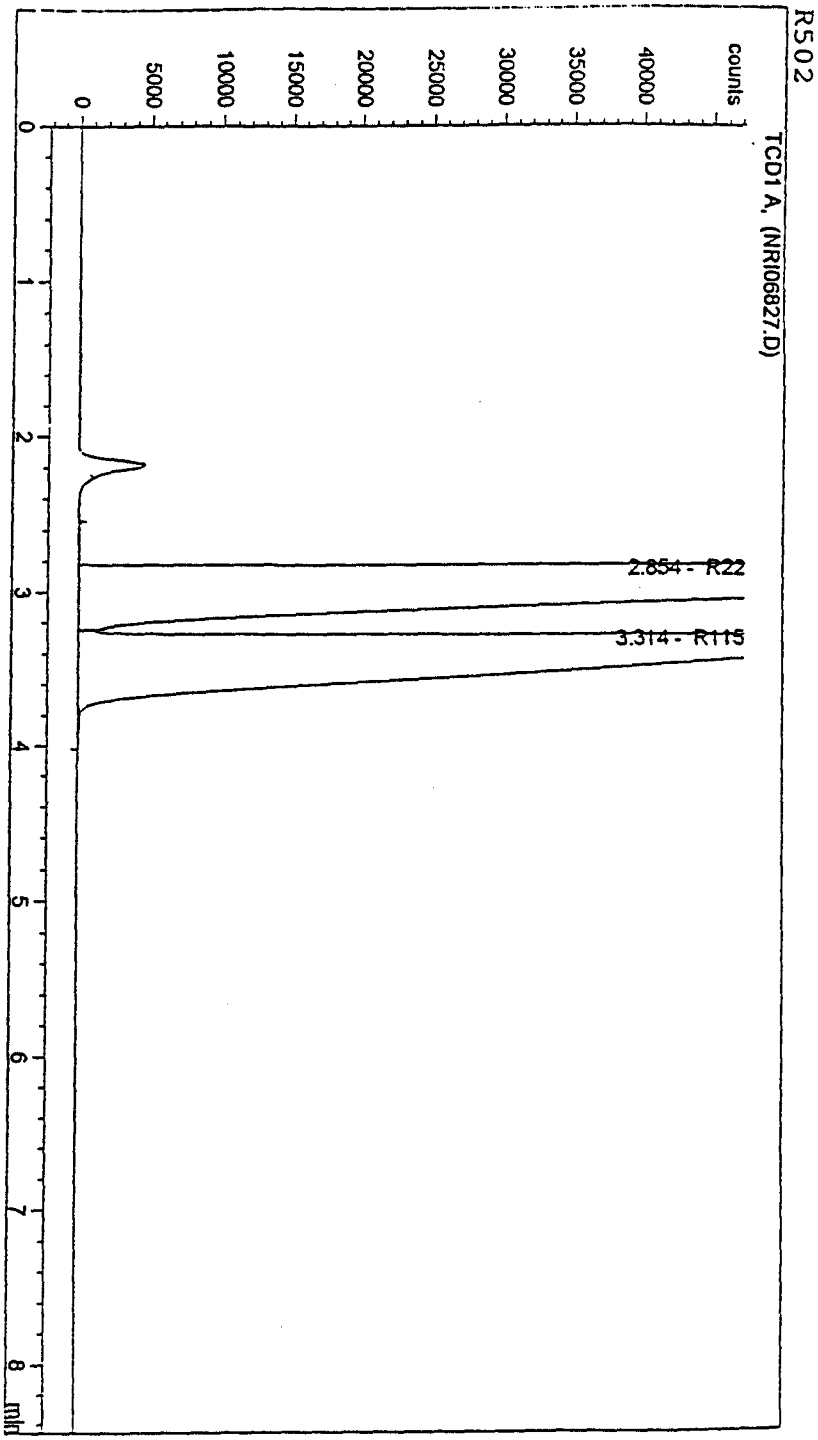


Figure 1. Gas Chromatogram of R-502

Part 29

R-503 Composition GC Method Data Sheet

Section 1. Gas Chromatographic Conditions

Detector	TCD, Low Sensitivity
Carrier Gas	30 mL He/Min.
Initial Column Temp.	60°C (isothermal)
Initial Hold	-- Min.
Program	-- K/Min.
Final Column Temp.	60°C
Post Hold	7 Min.
Sample	1.0 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Table 1. Blend Component Wt. %

R-503	Wt%		
Component	R-503 (%)	Tolerance (%)	GC Retention Time (Min.)
R-23	40.0	±1.0	2.85
R-13	60.0	±1.0	3.95

Section 2. Precision, and Accuracy

Statistical parameters for each impurity are listed below. The data was obtained by analyzing an R-503 calibration mixture 7 times during one day by one operator.

Table 2. Component Statistical Parameters

R-503 Component	95% CL	Relative Mean Error, Wt%
R-23	±0.02%	0.03
R-13	±0.02%	0.03

Section 3. Calibration Standard Preparation

The following table is to be used in conjunction with Part 15, *The Determination of the Component Concentrations of Refrigerant 400 and 500 Series Blends and Azeotropes by Gas Chromatography*.

Table 3. Blend Calibration Standard Preparation

R-503 Component	Amount to Weigh (g)	Approximate Weight %
R-23	200	40
R-13	300	60

RT (Min)	Comp.
2.35	Air (1)
2.87	23
3.96	13

(1) Ignored in Calculations.

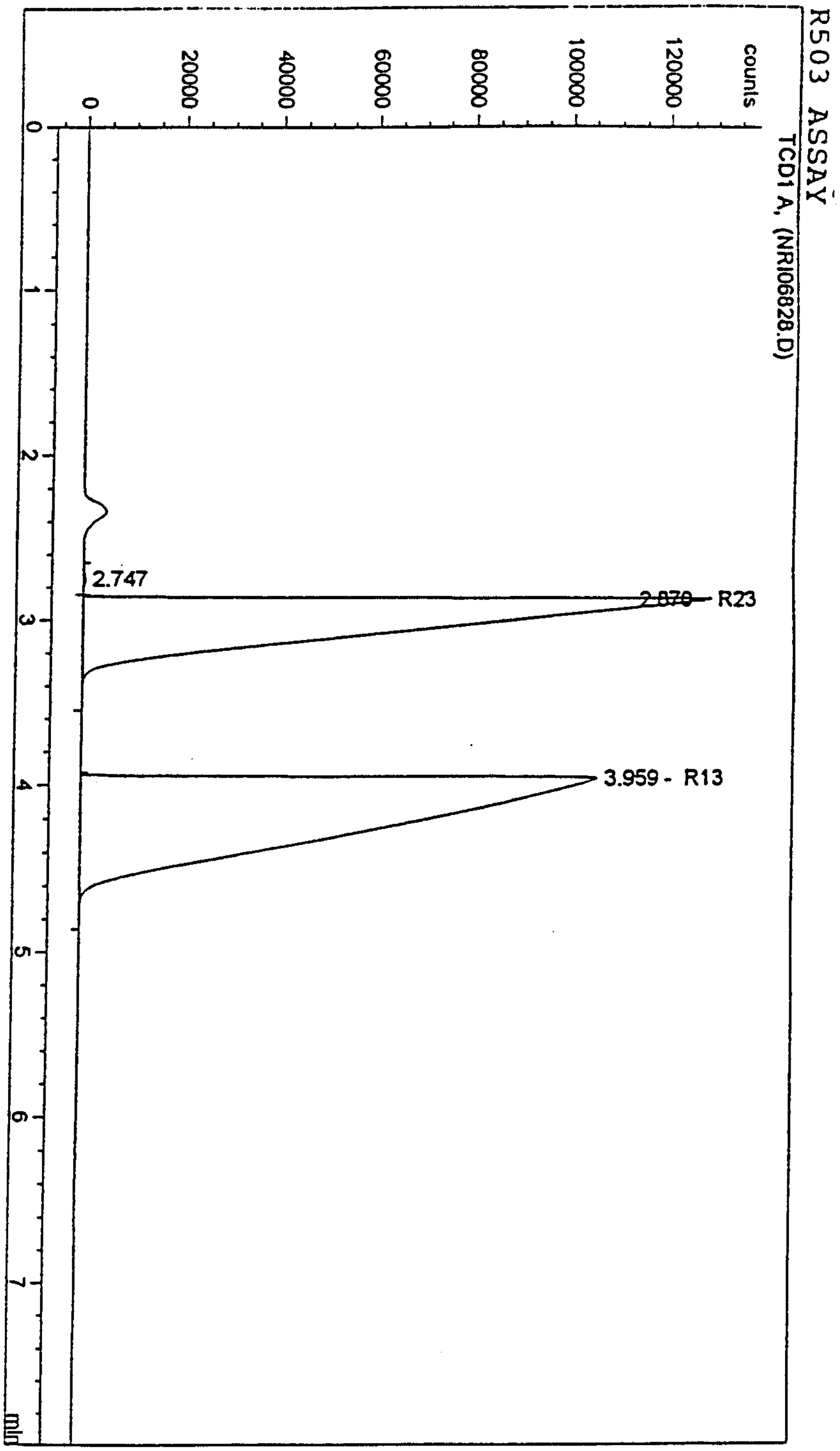


Figure 1. Gas Chromatogram of R-503

Part 30

R-507 Composition GC Method Data Sheet

Section 1. Gas Chromatographic Conditions

Detector	TCD, Low Sensitivity
Carrier Gas	30 mL He/Min.
Initial Column Temp.	40°C (isothermal)
Initial Hold	-- Min.
Program	-- K/Min.
Final Column Temp.	40°C
Post Hold	10 Min.
Sample	0.5 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Table 1. Blend Component Wt. %

R-507 Component	Wt% R-507 (%)	Tolerance (%)	GC Retention Time (Min.)
R-143a	50.0	±1.0	7.00
R-125	50.0	±1.0	8.15

Section 2. Precision, and Accuracy

Statistical parameters for each impurity are listed below. The data was obtained by analyzing an R-507 calibration mixture 7 times during one day by one operator.

Table 2. Component Statistical Parameters

R-507 Component	95% CL	Relative Mean Error, Wt%
R-143a	±0.049%	0.11
R-125	±0.047%	-0.15

Section 3. Calibration Standard Preparation

The following table is to be used in conjunction with Part 15, *The Determination of the Component Concentrations of Refrigerant 400 and 500 Series Blends and Azeotropes by Gas Chromatography*.

Table 3. Blend Calibration Standard Preparation

R-507 Component	Amount to Weigh (g)	Approximate Weight %
R-143a	488	50
R-125	488	50

RT (Min)	Comp.
2.41	Air (1)
5.43	143a
6.36	125

(1) Ignored in Calculations.

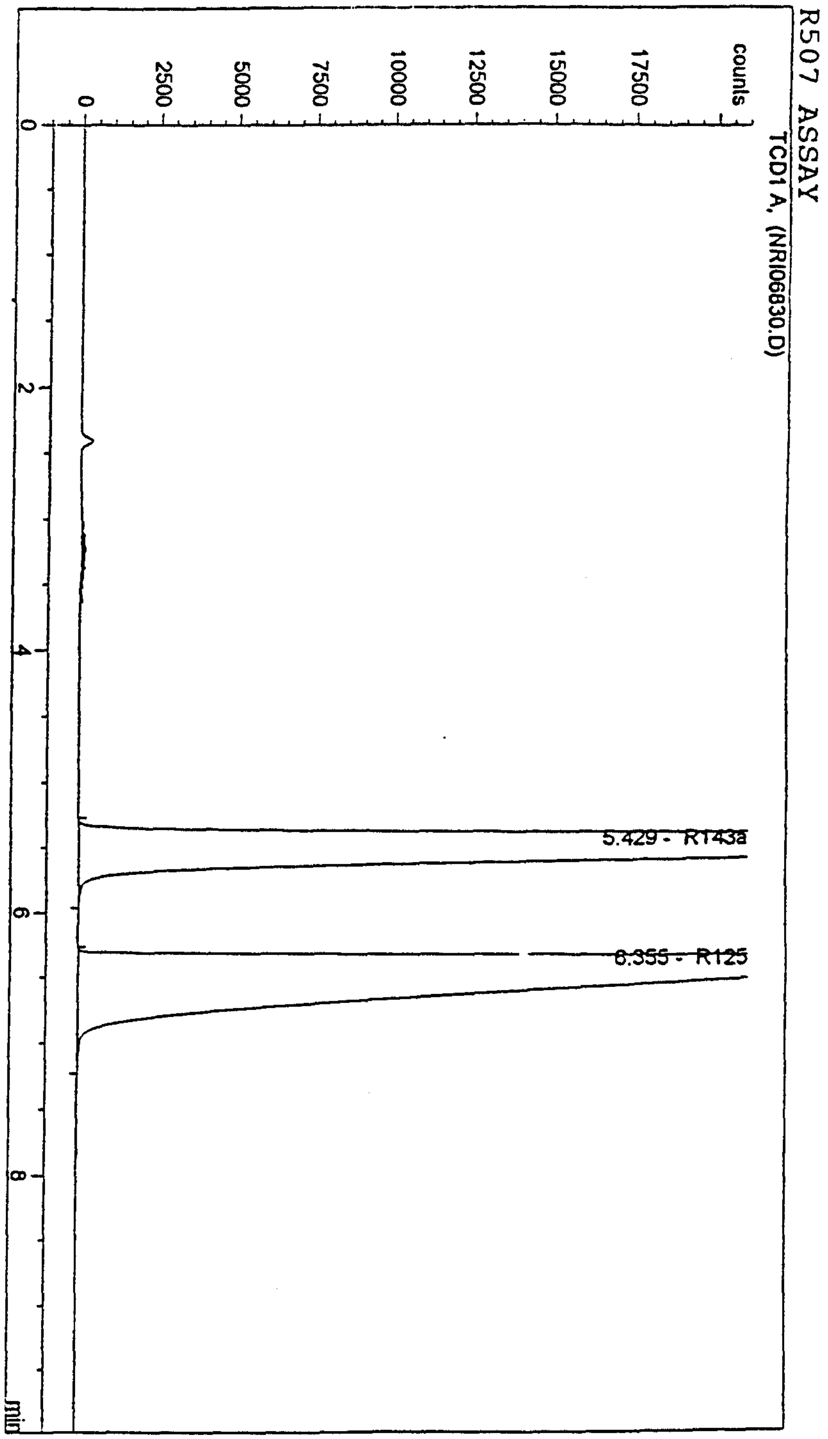


Figure 1. Gas Chromatogram of R-507

Part 31

R-508 Composition GC Method Data Sheet

Section 1. Gas Chromatographic Conditions

Detector	TCD, Low Sensitivity
Carrier Gas	30 mL He/Min.
Initial Column Temp.	75°C (isothermal)
Initial Hold	-- Min.
Program	-- K/Min.
Final Column Temp.	75°C
Post Hold	6 Min.
Sample	0.5 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Table 1A. Blend Component Wt. % R-508A

R-508	Wt%		
Component	R-508A (%)	Tolerance (%)	GC Retention Time (Min.)
R-23	39.0	±2.0	2.80
R-116	61.0	±2.0	3.35

Table 1B. Blend Component Wt. % R-508B

R-508	Wt%		
Component	R-508B (%)	Tolerance (%)	GC Retention Time (Min.)
R-23	46.0	±2.0	2.80
R-116	54.0	±2.0	3.35

Section 2. Precision, and Accuracy

Statistical parameters for each impurity are listed below. The data was obtained by analyzing an R-508 calibration mixture 7 times during one day by one operator.

Table 2. Component Statistical Parameters

R-508B Component	95% CL	Relative Mean Error, Wt%
R-23	±0.020%	0.043
R-116	±0.020%	0.028

Section 3. Calibration Standard Preparation

The following table is to be used in conjunction with Part 15, *The Determination of the Component Concentrations of Refrigerant 400 and 500 Series Blends and Azeotropes by Gas Chromatography.*

Table 3. Blend Calibration Standard Preparation

R-508 Component	Amount to Weigh (g)	Approximate Weight %
R-23	342	57
R-116	258	43

RT (Min)	Comp.
2.36	Air (1)
2.80	23
3.36	116
(1) Ignored in Calculations.	

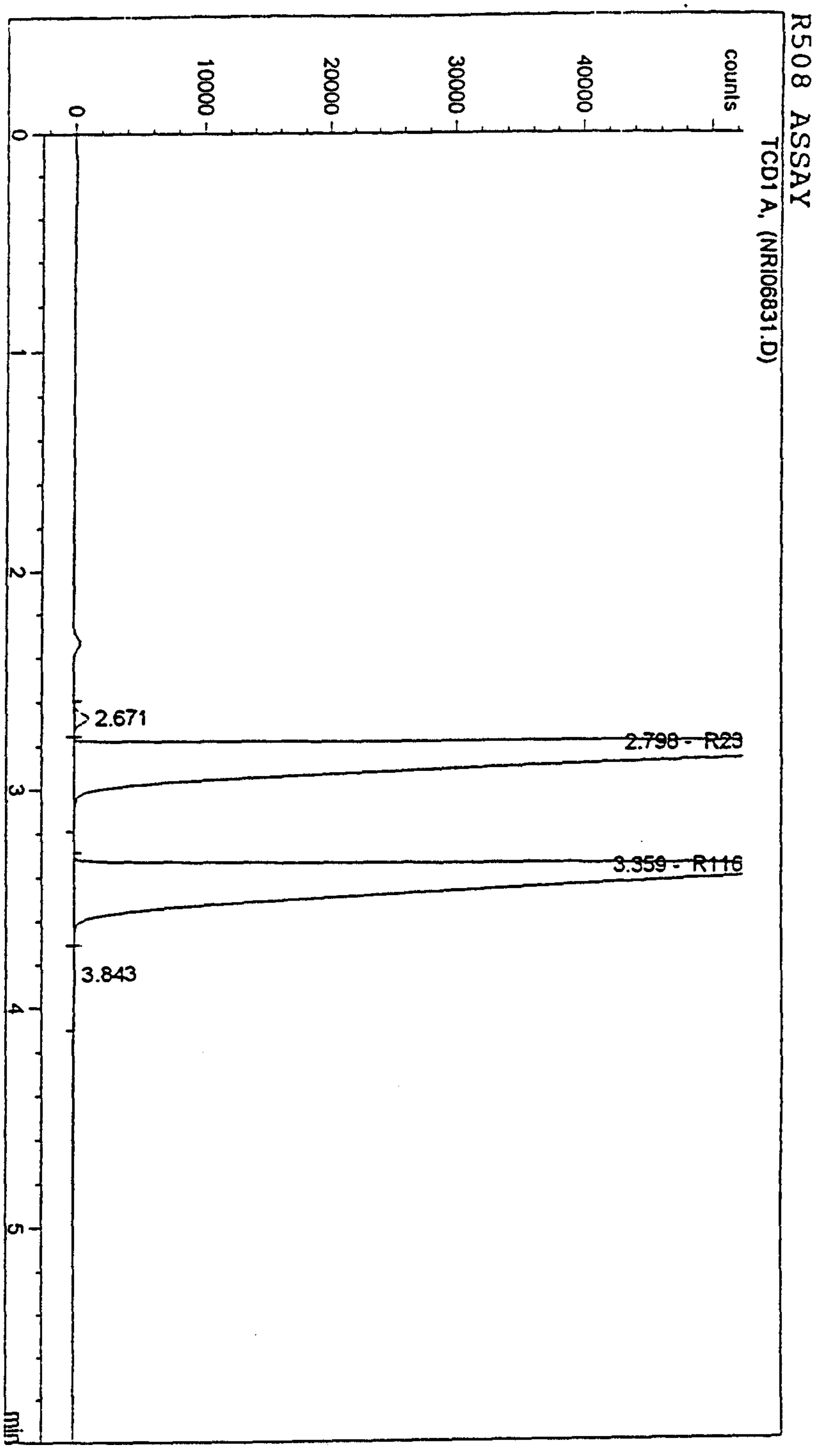


Figure 1. Gas Chromatogram of R-508

Part 32

R-509 Composition GC Method Data Sheet

Section 1. Gas Chromatographic Conditions

Detector	TCD, Low Sensitivity
Carrier Gas	30 mL He/Min.
Initial Column Temp.	75°C (isothermal)
Initial Hold	-- Min.
Program	-- K/Min.
Final Column Temp.	75°C
Post Hold	10 Min.
Sample	0.5 mL (gas syringe)
Detector Temp.	250°C
Inj. Port Temp.	200°C
Max Safe Column Temp.	225°C (for conditioning purposes)

Table 1. Blend Component Wt. %

R-509	Wt%		
Component	R-509 (%)	Tolerance (%)	GC Retention Time (Min.)
R-22	44.0	±2.0	5.20
FC-218	56.0	-0, +4.0	5.90

Section 2. Precision, and Accuracy

Statistical parameters for each impurity are listed below. The data was obtained by analyzing an R-509 calibration mixture 7 times during one day by one operator.

Table 2. Component Statistical Parameters

R-509 Component	95% CL	Relative Mean Error, Wt%
R-22	±0.044%	0.10
FC-218	±0.041%	0.08

Section 3. Calibration Standard Preparation

The following table is to be used in conjunction with Part 15, *The Determination of the Component Concentrations of Refrigerant 400 and 500 Series Blends and Azeotropes by Gas Chromatography*.

Table 3. Blend Calibration Standard Preparation

R-509 Component	Amount to Weigh (g)	Approximate Weight %
FC-218	597	56
R-22	469	44

RT (Min)	Comp.
2.30	Air (1)
5.16	22
5.90	218

(1) Ignored in Calculations.

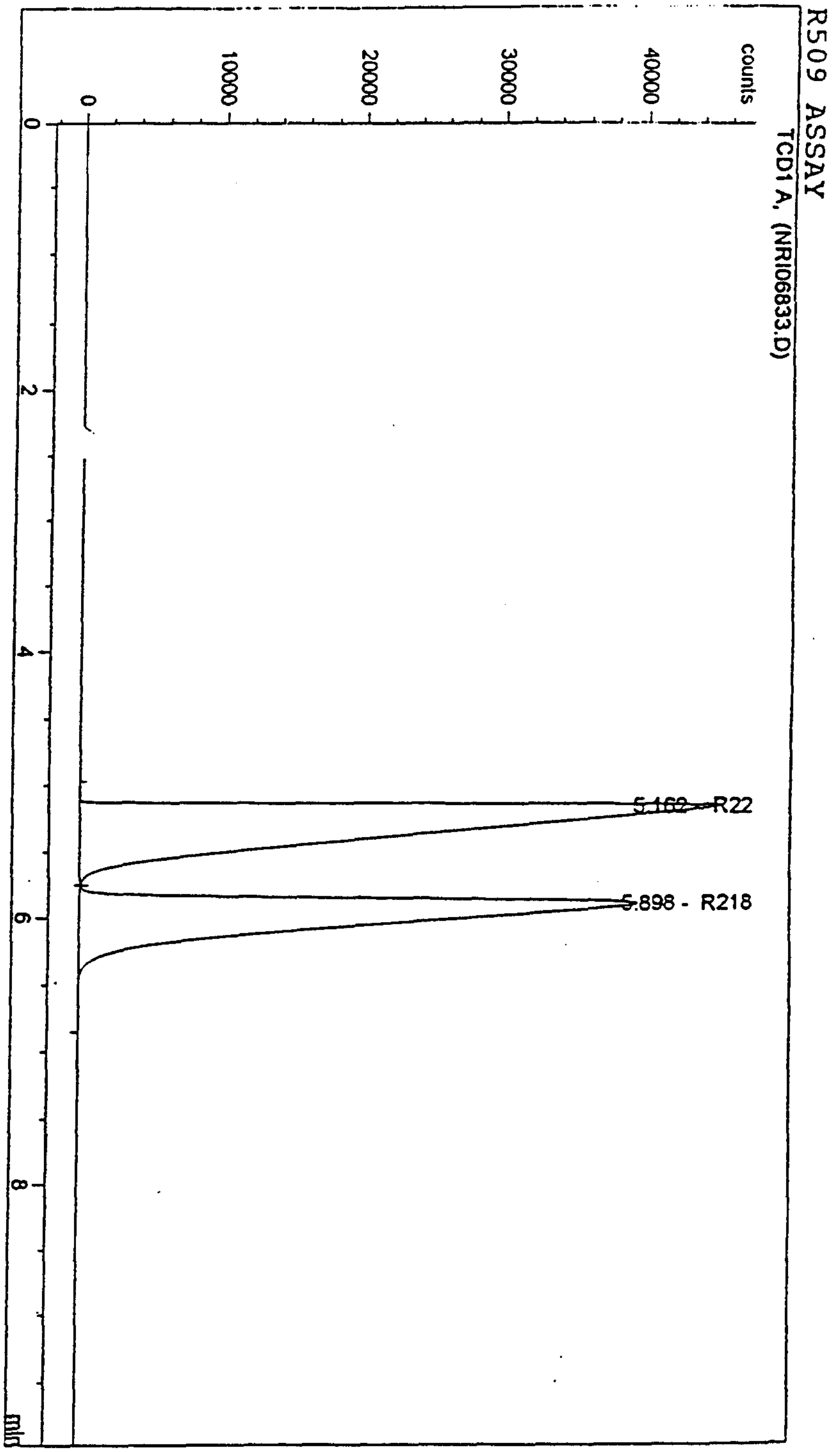


Figure 1. Gas Chromatogram of R-509

PART 33

GENERAL PROCEDURE FOR THE DETERMINATION OF PURITY OF NEW AND RECLAIMED REFRIGERANT BLENDS AND AZEOTROPES BY GAS CHROMATOGRAPHY

Section 1. Purpose

The purpose of this test method is to determine the purity of new and reclaimed Refrigerants 400 (R-400) and 500 (R-500) series blends and azeotropes by Gas Chromatography (GC).

Section 2. Scope

This test method is for use in conjunction with the GC Method Data Sheets for the refrigerants listed in Section 11 of this method.

Section 3. Definitions

Definitions for this part are identical to those of ARI Standards 700-95 and 740-95.

Section 4. Principle

The organic purity of new and reclaimed refrigerant blends and azeotropes is determined by programmed temperature gas chromatography using a packed column and a flame ionization detector (FID). Component peak areas are integrated electronically and quantified by the area normalization-response factor method.

Section 5. Applicability

This method is applicable to the determination of the impurities typically present in new and reclaimed refrigerants.

Section 6. Limitations and Interferences

For new refrigerant blends and azeotropes, it is imperative that the constituent components of the blend be separately analyzed prior to blending; this will permit greater scrutiny than analysis after blending. This method will not detect any impurity that may elute with one of the blend components.

If blend composition determination is desired, refer to Part 15, *Determination of the Component Concentrations of Refrigerant 400 and 500 Series Blends and Azeotropes by Gas Chromatography*.

Section 7. Sensitivity, Precision, and Accuracy

7.1 *Sensitivity*

Values for these statistical parameters are given in Table 1A and 1B of each respective refrigerant's GC Method Data Sheet.

Section 8. Special Apparatus and Reagents

NOTE: Equivalents may be substituted.

1. Gas chromatograph: Model 5890, equipped with FID, Hewlett Packard, Wilmington, DE.
2. Electronic integrator: Model# 3396, Hewlett Packard, Wilmington, DE.
3. Packed column: formed by joining together two 7.32 m x 3.17 mm OD stainless steel, 1% SP-1000 on Carbopack B, 60/80 mesh, Supelco, Bellefonte, PA.
4. Glass collecting tubes: 500 mL, 250 mL and 125 mL, LG-8601, Lab Glass Inc., Vineland, NJ. (Enlarge side outlet opening to accommodate a crimp-on 2-cm septum. Apply fiberglass tape outside for protection from breakage)
5. Steel cylinder: 1L, with a single #9 valve (#1014-C, Superior Valve), 3/8" pipe neck, E. F. Britten Co., Cranford, NJ.
6. Deflected point needles: Cat# 7174, #22, Popper and Sons, Inc., New Hyde Park, NY.
7. Swivel union: US44, United Refrig. Inc., Philadelphia, PA.
8. Serum bottle: 125 mL, (Note: Bottle holds 160 mL when liquid full.) Cat# 223748, Wheaton Glass, Vineland, NJ.
9. Impurities for calibration standard preparation: The fluorochemicals may be purchased from Lancaster, Windham, NH and Synquest, Inc., Alachua, FL. The hydrocarbons may be purchased from Scott Specialty Gases, Inc., Plumbsteadville, PA. All other impurities may be purchased from Aldrich, Milwaukee, WI. See Table 2 of the GC Method Data sheet for the specific impurities required for each refrigerant.

NOTE: The purity of each calibration component must be predetermined by gas chromatography and, if necessary, by GC/Mass Spectroscopy (GC-MS).

10. Stainless steel cylinder: 1L, 304L-WDF4-1000, and 300mL cylinder, 304L-WDF4-300, 1/4" pipe, Whitey Co., Highland Heights, OH.

Section 9. Procedure

NOTE: The following procedure is generalized for the determination of purity for blends and azeotropes. Each respective GC Method Data Sheet contains the chromatogram, statistical parameters and impurities to be added for calibration standard preparation.

9.1 *Chromatographic Operating Conditions*

Detector	FID
Carrier Gas	Helium; 28 ml min^{-1}
Initial Column Temp.	35º C
Initial Hold	14 min
Program	10 K min^{-1}
Final Column Temp.	160º C
Post Hold	34 min
Sample	500 µl loop
Detector Temp.	200º C
Injection Port Temp.	150º C

9.2 *Calibration Standard Preparation: Major Components*

NOTE: The procedure here is generalized and is followed for the preparation of each blend calibration standard. The weights of each blend component to be added and the order of addition are given in the appended GC Method Data Sheets for each respective refrigerant blend. Normally, the least volatile components are added first. To determine the composition of the prepared blends and azeotropes, refer to Part 15, Determination of the Component Concentrations of Refrigerants 400 and 500 Series Blends and Azeotropes by Gas Chromatography.

a. Evacuate a clean, dry 1L stainless steel Calibration Standard cylinder to 25 Pa. Break the vacuum and allow the cylinder to fill with air. Re-evacuate the cylinder to 25 Pa.

b. Weigh the Calibration Standard cylinder to the nearest 0.01 gram.

c. Using a Teflon® flex transfer line, attach the Calibration Standard cylinder to the first component supply cylinder (liquid phase valve) via a vacuum manifold and absolute pressure gauge (refer to Table 1C of the appropriate GC Method Data Sheet).

d. Gradually open the metering valve and add the first component as a vapor (vaporized liquid phase) until the gauge reaches the pressure indicated in Table 1C of the GC Method Data Sheet.

NOTE: If too much refrigerant component is added, the cylinder must be vented. Venting is permitted only during addition of the first component and not thereafter.

e. Close the Calibration Standard cylinder valve, remove it from the vacuum manifold, and reweigh the cylinder to the nearest .01g.

f. Subtract the cylinder tare weight (step b) from the weight in step e and record the as the weight of the first component.

g. Repeat steps c through f for the second component.

NOTE: It is often simpler to prepare a second cylinder containing the exact weight of the second and also subsequent components to be added—then transferring the entire contents (or nearly so) into the Calibration Standard cylinder using dry ice or liquid nitrogen to externally cool the Calibration Standard cylinder. Allow the cylinder to warm to ambient temperature before weighing.

h. Repeat steps c through f for a third and (if necessary) a fourth component.

i. Add the weights of all the added components and then determine the weight percentage (to the nearest 0.01%) of each component in the component mixture. If necessary, correct the individual component percentages for the purities determined in the Note of Section 8.9. (Also, see Note 1 of Section 10)

9.3 *Calibration Standard Preparation: Addition of Contaminants*

a. Attach a Swagelok nut and septum to the Calibration Standard cylinder and then chill the base of the cylinder in liquid nitrogen for 15 minutes. Do not purge the cylinder when attaching the septum, as this would alter the relationship between the blend components and the contaminants. Instead, the small amount of air behind the septum is permitted to enter the standard when the valve is opened.

b. Verify that the septum is still attached securely, then open the cylinder valve while keeping the cylinder immersed in the liquid nitrogen.

c. Using appropriately sized vapor syringes, individually and in turn add the gaseous contaminants listed in Table 2 of each respective GC Method Data Sheet in the amounts indicated. This is done by flashing liquid phase into the gas bulb shown in Figure 1 and then withdrawing the specified amount of vapor into the gas tight syringe and injecting through the septum attached to the cylinder. The indicated vapor densities are based on a laboratory temperature of 20.0º C and a barometric pressure of 100.0 kPa. This data must be adjusted to reflect actual laboratory conditions.

d. Using appropriately sized liquid syringes, individually and in turn add the liquid contaminants listed in Table 2 in the amounts indicated (Special care must be taken to account for the syringe needle volume). For best accuracy, these contaminants and syringes should be pre-chilled in a freezer (approximately -20º C) and promptly transferred. The indicated liquid densities are based on a liquid temperature of 0º C and a barometric pressure of 100.0 kPa. This data must be adjusted to reflect the actual conditions surrounding the transfer.

e. Close the Standard Cylinder valve and allow the Calibration Standard cylinder to return to ambient temperature. Set the cylinder aside for a minimum of 12 hours to allow the cylinder's contents to equilibrate. Roll the cylinder while still cold for at least 4 hours to ensure thorough mixing.

f. Using a gas tight syringe, withdraw the vapor sample from the Calibration Standard cylinder and chromatograph the exact volume listed in Section 9.1. If a gas sampling loop is attached to the GC, purge the loop with the calibration standard, allow the loop to return to atmospheric pressure and then inject the sample into the GC. Chromatograph the standard as indicated in Section 9.1, and adjust the standard's recorded component and contaminant levels, in milligrams, to account for any significant impurities present in the starting materials using the method of Standards Addition (see Note 1 in Section 10).

g. Determine the weight percentage of each major component and each contaminant in the calibration standard using the formula:

$$\text{Wt\% x} = \frac{(100)(\text{Mass of x in Cal. Std.})}{\text{Total Mass of Cal. Std.}}$$

Weight percentages are calculated to the nearest 0.01% for each major component and to the nearest 0.0001% for each contaminant.

9.4 *Determination of Major Component Response Factors*

a. Analyze the standard in triplicate using the chromatographic conditions given in Section 9.1. Because the standard is vapor phase, it can be transferred directly to the gas chromatograph (via the gas sampling loop or gas sampling bulb/syringe) without further preparation or sampling.

b. Average the peak areas obtained for each major component of either the blend or azeotrope.

c. Calculate relative response factors (RRF's) for each major component as follows:

$$\text{ARF}_i = \frac{\text{Wt\% Comp. i in Cal. Std.}}{A_i}$$

Where:

RRF_i = relative response factor for component i.
A_i = peak area of component i.

Then, using the component with the highest area count in the mixture as the reference peak the RRF Factors are determined as follows:

$$RRF_i = \frac{ARF_i}{ARF_x} \quad \text{and}$$

$$RRF_x = \frac{ARF_x}{ARF_x} = 1$$

ARF_i = absolute response factor for component i.
ARFx = absolute response factor for the component with the highest area count in the mixture.
RRF_i = relative response factor for component i.

RRF_x = relative response factor for the component with the highest area count in the mixture.

RRF values are computed to the nearest 0.0001 unit.

9.5 *Determination of Contaminant Response Factors*

Using the highest area count major component as the reference peak, determine and record each contaminant's relative response factor (RRF) as follows:

$$ARF_i = \frac{\text{Wt\% i in Cal. Std.}}{A_i}$$

Where:

ARF_i = Absolute Response Factor of contaminant i.
A_i = peak area of component i (average of 3 determinations).

Then using the highest area count component in the blend as the reference peak:

$$RRF_i = \frac{ARF_i}{ARFx}$$

Again, RRF_i values are computed to the nearest 0.0001 unit.

9.6 *Sampling*

High Pressure (HP) sample cylinders should be filled at least 80% but no more than 90% liquid full. This will both minimize component fractionation within the sample container and will avoid overfilling for safety considerations. Very high pressure (VHP) sample cylinders (vapor phase) may be filled to less than saturation pressure, if desired.

9.7 *Sample Analysis*

Analyze the sample using the chromatographic conditions described in Section 9.1. Load the sample as illustrated in Figure 1 except that the gas sampling bulb is replaced by a 300 mL stainless steel cylinder. Flash the sample liquid phase so as to bring the pressure to just below saturation. Questionable sample results are most often resolved by re-analysis of the calibration standard. The temperature of the sample must equal (essentially) that of the calibration standard at the time of GC calibration. In the case of VHP refrigerants, the sample must be at or above the critical temperature before analysis.

9.8 *Calculations*

a. The weight percentage of each component is calculated as follows:

$$W_i = \frac{100 \text{ x } RRF_i \text{ x } A_i}{\sum (A_i \text{ x } RRF_i)}$$

Where:

W_i = weight percentage of component i.
RRF_i = relative response factor for component i.

A_i = peak area of component i.
Σ…= sum of all component peak areas times their respective relative response factors.

Section 10. Notes

1. The purest refrigerant blend will contain some of the contaminants listed in Table 1 of the GC Method Data Sheet in low concentrations. Individual impurity peak areas are increased in the calibration standard by the peak areas that correspond to the mass of the impurity added. The amounts of each contaminant in the stock components are thereby determined by the method of Standards Addition. The mass of each contaminant present is combined with the mass added to give the total mass of each contaminant in the calibration standard.

Section 11. GC Method Data Sheets

Table 1. GC Method Data Sheets

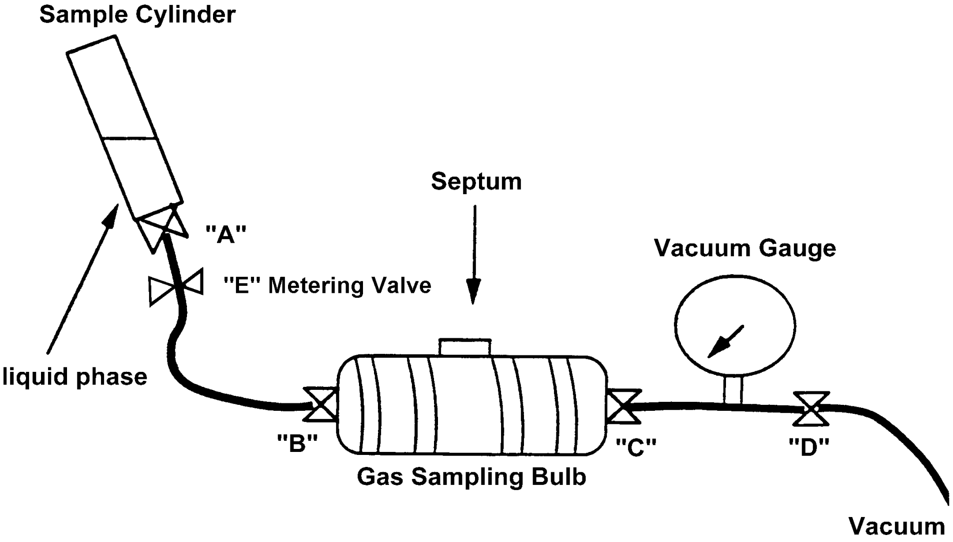
Refrigerant	GC Method Data Sheet Part Number
R-401	Part 34
R-402	Part 35
R-404	Part 36
R-405	Part 37
R-406	Part 38
R-407	Part 39
R-408	Part 40
R-409	Part 41
R-410	Part 42
R-411	Part 43
R-412	Part 44
R-500	Part 45
R-502	Part 46
R-503	Part 47
R-507	Part 48
R-508	Part 49
R-509	Part 50

Section 12. References

1. Air-Conditioning and Refrigeration Institute, *Appendix C to ARI Standard 700-95: Analytical Procedures for ARI Standard 700-95*, 4301 North Fairfax Drive, Arlington, Virginia 22203.

2. Integral Sciences, Inc., *Methods Development for Organic Contaminant Determination in Fluorocarbon Refrigerant Azeotropes and Blends*, ARTI MCLR Project Number 665-54600.

Figure 1. Apparatus Used for Sampling Calibration Standards and Samples



Part 34

R-401 Purity GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Part 33, *General Procedure for the Determination of Purity of New and Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography* (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-401 blends.

Section 2. Limitations and Interferences

This method is applicable and calibrated for only those impurities commonly present in R-401 blends (See Table 2). This method will not detect any impurity that may elute within the comparatively large HCFC-22, HCFC-124, and HFC-152a peak matrices. For example, HFC-125 elutes on the far shoulder of the large HFC-152a peak and is therefore difficult to detect at low concentrations. Linearity of the HFC-125 response is marginal and limits the precision in the determination of this impurity. The contaminants HFC-23 and HFC-32 also elute together, but these may be separated if needed by adding a 0.30 m column section of Porapak-T to the end of the primary column.

Section 3. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1A and B. The data was obtained by analyzing an R-401 calibration mixture 7 times during one day by one operator.

Section 4. Tables

The following tables are to be used in conjunction with Section 9 of the General Procedure for the Determination of Purity of New and Reclaimed Refrigerants by Gas Chromatography.

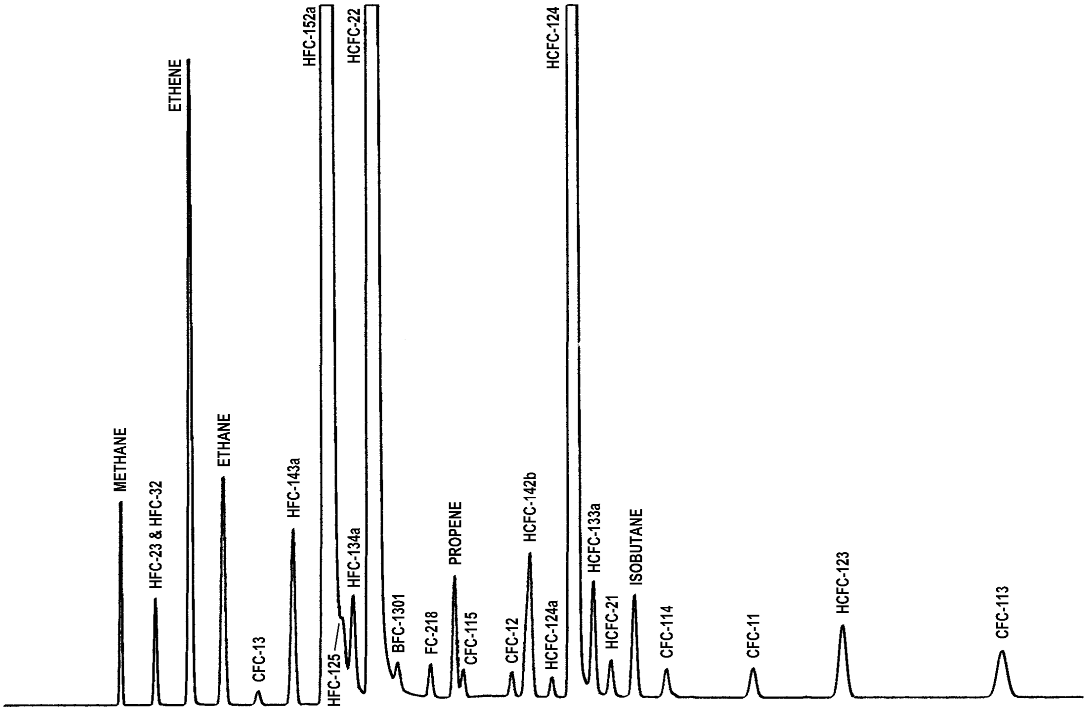


Figure 1. Gas Chromatogram of R-401

Table 1A. Contaminant Statistical Parameters

Component	Detection Limit, ppm	Range Investigated, ppm	Precision at 95% Confidence Limit, ppm
methane	1	100	2.8
23	10	450	10.
32	5	450	7.8
ethene	1	450	7.1
ethane	1	200	2.6
13	12	450	9.5
143a	2	450	12
134a	3	450	5.1
1301	9	450	4.1
218	6	450	7.0
propene	1	100	1.4
115	7	450	32
12	8	450	13
142b	2	450	29
133a	2	450	18
21	5	450	31
isobutane	1	100	2.3
114	5	450	8.0
11	6	450	41
123	2	450	44
113	2	450	60.

Table 1B. Component Statistical Parameters

Component	Detection Limit, ppm	Range Investigated, Wt. %	Precision at 95% Confidence Limit
152a	2	13 %	0.10 %
22	5	53 %	0.51 %
124	5	34 %	0.26 %

Table 1C. Blend Component Balance Preparation

Component	Final Pressure After Component Addition (kPa)	Weight of Refrigerant Added (g)
HCFC-124	118	6.80
HFC-152a	206	2.60
HCFC-22	466	10.60

Table 2. Primary Calibration Standard Impurities

Component	Vapor Density, mg/ml	Volume Added, ml	mg Added (1)	ppm Added (2)	Total ppm Present (3)
methane	0.656	4.0	2.62	130	
23	2.895	3.5	10.13	500	
32	2.162	4.7	10.16	502	
ethene	1.147	4.0	4.58	226	
ethane	1.230	4.0	4.92	243	
13	4.331	2.4	10.39	513	
143a	3.511	2.9	10.18	503	
125	5.004	2.1	10.50	519	
134a	4.279	2.4	10.26	507	
1301	6.205	1.7	10.54	521	
218	7.923	1.3	10.29	508	
propene	1.756	5.8	10.18	503	
115	6.486	1.6	10.37	512	
12	5.077	2.0	10.15	501	
142b	4.248	2.4	10.19	503	
133a	4.846	2.1	10.17	502	
21	4.367	2.4	10.48	517	
isobutane	2.452	4.2	10.29	508	
114	7.319	1.4	10.24	506	
Component	**Liquid Density, mg/µl**	**Volume Added, µl**	**mg Added (1)**	**ppm Added (2)**	**Total ppm Present (3)**
11	1.537	6.6	10.14	501	
123	1.526	6.7	10.22	505	
113	1.619	6.3	10.19	503	

(1) If necessary, correct the mg added for the purity of the calibration component previously established.

(2) Values shown are for illustration; exact values are determined at Part 33, Section 9.3, steps c and d.

(3) Column to be filled in (Part 33, Section 9.3, step g) after determining ppm present in the stock components (see Note 1 in Section 10).

Part 35

R-402 Purity GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Part 33, *General Procedure for the Determination of Purity of New and Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography* (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-402 blends.

Section 2. Limitations and Interferences

This method is calibrated only for those impurities commonly present in R-402 blends. This method will not detect any impurity that may elute within the comparatively large HCFC-22, HFC-125, and propane peak matrices. For example, HFC-134a elutes on the far shoulder of the large HFC-125 peak and is therefore difficult to detect at low concentrations. Linearity of the HFC-134a response is marginal and limits the precision in the determination of this impurity. Detection of CFC-115 is not resolved as it would coelute with the propane. The contaminants HFC-23 and HFC-32 also elute together, but these may be separated if needed by adding a 0.30 m column section of Porapak-T to the end of the primary column.

Section 3. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1A and B. The data was obtained by analyzing an R-402 calibration mixture 7 times during one day by one operator.

Section 4. Tables

The following tables are to be used in conjunction with Part 33 of the *General Procedure for the Determination of Purity of Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography*.

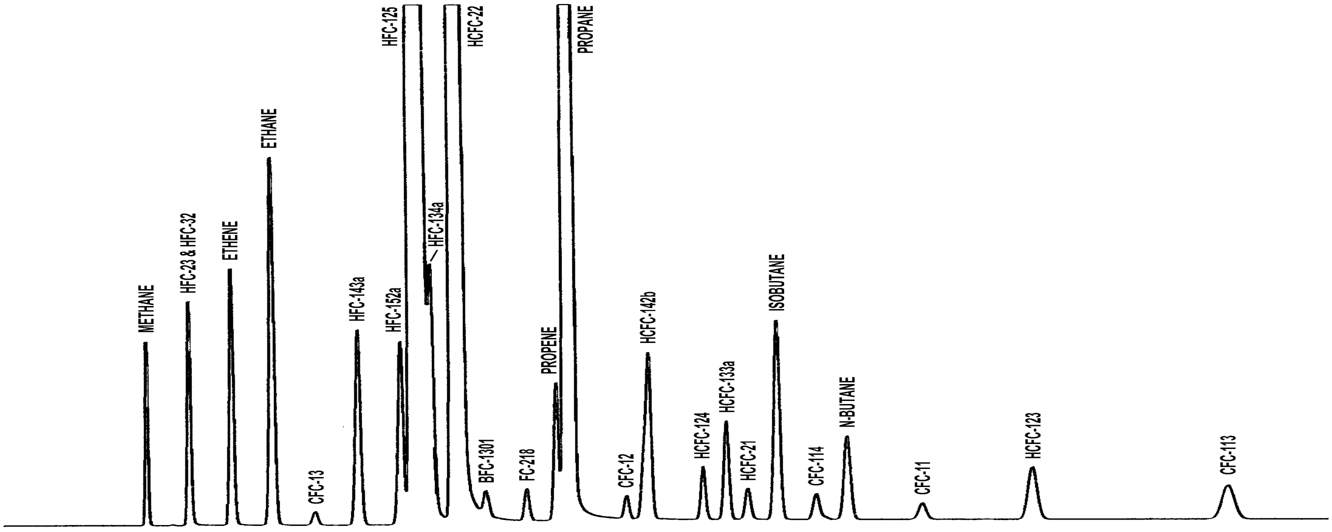


Figure 1. Gas Chromatogram of R-402

Table 1A. Contaminant Statistical Parameters			
Component	**Detection Limit, ppm**	**Range Investigated, ppm**	**Precision at 95% Confidence Limit, ppm**
methane	1	100	0.8
23	9	500	8.5
32	4	500	38
ethene	1	200	1.5
ethane	1	280	2.5
13	11	500	14
143a	1	500	6.7
134a	100	500	82
1301	8	500	4.5
218	5	500	3.6
propene	1	100	0.7
12	8	500	2.9
142b	2	500	49
124	3	500	9.0
133a	2	500	4.4
21	5	500	11
isobutane	1	150	1.4
114	5	500	2.8
n-butane	1	100	1.4
11	7	500	15
123	2	500	13
113	2	500	18

Table 1B. Component Statistical Parameters			
Component	**Detection Limit, ppm**	**Range Investigated, %**	**Precision at 95% Confidence Limit**
125	5	60 %	0.17 %
22	5	38 %	0.20 %
propane	1	2 %	0.01 %

Table 1C. Blend Component Balance Preparation		
Component	**Final Pressure After Component Addition (kPa)**	**Weight of Refrigerant Added (g)**
Propane	22	0.40
HCFC-22	340	12.00
HFC-125	472	7.60

Table 2. Primary Calibration Standard Impurities

Component	Vapor Density, mg/ml	Volume Added, ml	mg Added (1)	ppm Added (2)	Total ppm Present (3)
methane	0.656	4.0	2.62	130	
23	2.895	3.5	10.13	500	
32	2.162	4.7	10.16	502	
ethene	1.147	4.0	4.58	226	
ethane	1.230	4.0	4.92	243	
13	4.331	2.4	10.39	513	
143a	3.511	2.9	10.18	503	
152a	2.772	3.7	10.25	506	
134a	4.279	2.4	10.26	507	
1301	6.205	1.7	10.54	521	
218	7.923	1.3	10.29	508	
propene	1.756	5.8	10.18	503	
12	5.077	2.0	10.15	501	
142b	4.248	2.4	10.19	503	
124	5.758	1.8	10.36	512	
133a	4.846	2.1	10.17	502	
21	4.367	2.4	10.48	517	
isobutane	2.452	4.2	10.29	508	
114	7.319	1.4	10.24	506	
n-butane	2.463	4.2	10.34	511	
Component	**Liquid Density, mg/µl**	**Volume Added, µl**	**mg Added (1)**	**ppm Added (2)**	**Total ppm Present (3)**
11	1.537	6.6	10.14	501	
123	1.526	6.7	10.22	505	
113	1.619	6.3	10.19	503	

(1) If necessary, correct the mg added for the purity of the calibration component previously established.

(2) Values shown are for illustration; exact values are determined in Part 33, Section 9.3, steps c and d.

(3) Column to be filled in (Part 33, Section 9.3 step g) after determining ppm present in the stock components (see Note 1 in Section 10).

Part 36

R-404 Purity GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Part 33, *General Procedure for the Determination of Purity of New and Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography* (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-404 blends.

Section 2. Limitations and Interferences

This method is calibrated only for those impurities commonly present in R-404A. This method will not detect any impurity that may elute within the comparatively large HFC-125, HFC-134a, and HFC-143a peak matrices. The contaminants HFC-23 and HFC-32 also elute together, but these may be separated if needed by adding a 0.30 m column section of Porapak-T to the end of the primary column.

Section 3. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1A and B. The data was obtained by analyzing an R-404 calibration mixture 7 times during one day by one operator.

Section 4. Tables

The following tables are to be used in conjunction with Part 33 of the *General Procedure for the Determination of Purity of Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography*.

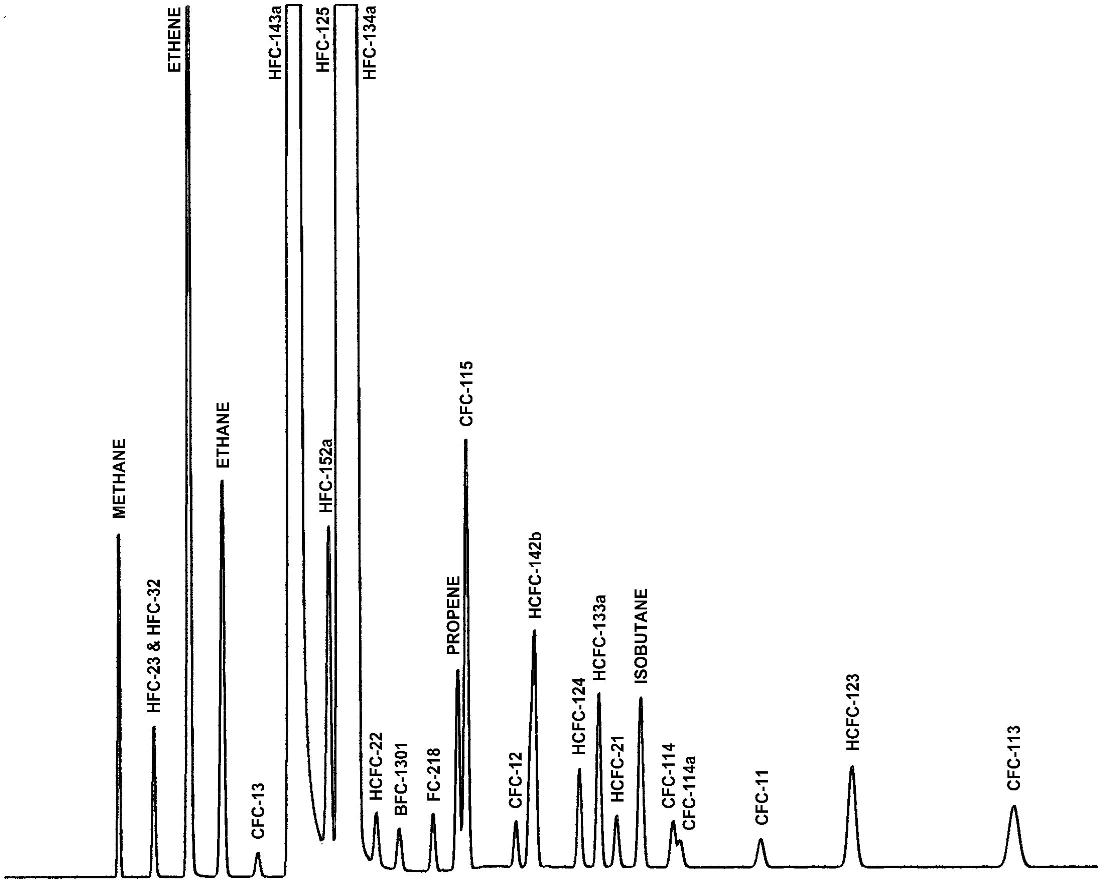


Figure 1. Gas Chromatogram of R-404A

Table 1A. Contaminant Statistical Parameters			
Component	**Detection Limit, ppm**	**Range Investigated, ppm**	**Precision at 95% Confidence Limit, ppm**
methane	1	120	3.1
23	10	520	6.7
32	4	520	7.9
ethene	1	520	5.1
ethane	1	230	2.5
13	10	520	9.4
22	7	520	12
1301	7	520	21
218	5	520	16
propene	1	130	2.1
115	6	4400	97
12	7	520	19
142b	2	520	89
124	3	520	82
133a	2	520	12
21	5	520	39
isobutane	1	130	3.0
114	4	350	4.6
114a	4	170	2.9
11	7	520	73
123	2	520	50

Table 1B. Component Statistical Parameters			
Component	**Detection Limit, ppm**	**Range Investigated, %**	**Precision at 95% Confidence Limit**
143a	4	52 %	0.62 %
125	4	44 %	0.61 %
134a	3	4 %	0.15 %

Table 1C. Blend Component Balance Preparation		
Component	**Final Pressure After Component Addition (kPa)**	**Weight of Refrigerant Added (g)**
R-134a	19	0.80
R-143a	304	10.4
R-125	458	8.80

Table 2. Primary Calibration Standard Impurities

Component	Vapor Density, mg/ml	Volume Added, ml	mg Added (1)	ppm Added (2)	Total ppm Present (3)
methane	0.656	4.0	2.62	130	
23	2.895	3.5	10.13	500	
32	2.162	4.7	10.16	502	
ethene	1.147	4.0	4.58	226	
ethane	1.230	4.0	4.92	243	
13	4.331	2.4	10.39	513	
152a	2.772	3.7	10.25	506	
22	3.606	2.9	10.45	516	
1301	6.205	1.7	10.54	521	
218	7.923	1.3	10.29	508	
propene	1.756	5.8	10.18	503	
115	6.486	1.6	10.37	512	
12	5.077	2.0	10.15	501	
142b	4.248	2.4	10.19	503	
124	5.758	1.8	10.36	512	
133a	4.846	2.1	10.17	502	
21	4.367	2.4	10.48	517	
isobutane	2.452	4.2	10.29	508	
114	7.319	1.4	10.24	506	
Component	**Liquid Density, mg/µl**	**Volume Added, µl**	**mg Added (1)**	**ppm Added (2)**	**Total ppm Present (3)**
11	1.537	6.6	10.14	501	
123	1.526	6.7	10.22	505	
113	1.619	6.3	10.19	503	

(1) If necessary, correct the mg added for the purity of the calibration component previously established.

(2) Values shown are for illustration; exact values are determined at Part 33, Section 9.3, steps c and d.

(3) Column to be filled in (Part 33, Section 9.3, step g) after determining ppm present in the stock components (see Note 1 in Section 10).

Part 37

R-405 GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Part 33, *General Procedure for the Determination of Purity of New and Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography* (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-405 blends.

Section 2. Limitations and Interferences

This method is calibrated only for those impurities commonly present in R-405A. This method will not detect any impurity that may elute within the comparatively large HCFC-22, HCFC-142b, HFC-152a, and FC-C318 peak matrices. For example, HFC-125 elutes on the far shoulder of the large HFC-152a peak and is therefore difficult to detect at low concentrations. Linearity of the HFC-125 response is marginal and limits the precision in the determination of this impurity. The contaminants HFC-23 and HFC-32 also elute together, but these may be separated if needed by adding a 0.30 m column section of Porapak-T to the end of the primary column.

Section 3. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1A and B. The data was obtained by analyzing an R-405 calibration mixture 7 times during one day by one operator.

Section 4. Tables

The following tables are to be used in conjunction with Part 33 of the *General Procedure for the Determination of Purity of Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography*.

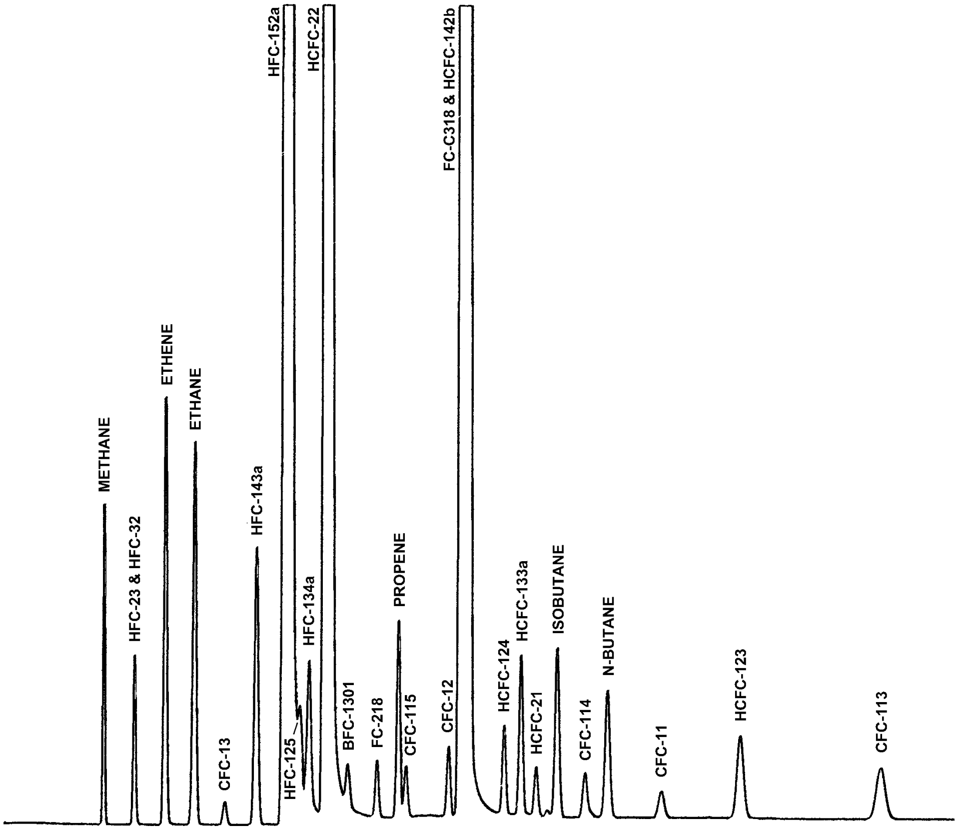


Figure 1. Gas Chromatogram of R-405

Table 1A. Contaminant Statistical Parameters			
Component	**Detection Limit, ppm**	**Range Investigated, ppm**	**Precision at 95% Confidence Limit, ppm**
methane	1	100	0.9
23	13	500	150
32	7	500	3.9
ethene	1	200	1.4
ethane	1	200	1.6
13	20	500	7.6
143a	2	500	3.9
134a	4	500	8.0
1301	16	500	3.0
218	9	500	2.8
propene	1	100	0.6
115	16	500	21
12	9	500	2.8
124	6	500	5.1
133a	3	500	5.8
21	10	500	10.
isobutane	1	100	1.1
114	8	500	5.9
n-butane	1	100	1.7
11	13	500	10.
123	4	500	13
113	4	500	21

Table 1B. Component Statistical Parameters			
Component	**Detection Limit, ppm**	**Range Investigated, %**	**Precision at 95% Confidence Limit**
152a	1	7 %	0.05 %
22	5	45 %	0.32 %
C318/142b	4	48 %	0.42 %

Table 1C. Blend Component Balance Preparation		
Component	**Final Pressure After Component Addition (kPa)**	**Weight of Refrigerant Added (g)**
FC-C318	105	9.00
R-142b	136	1.40
R-152a	173	1.10
R-22	394	8.5

Table 2. Primary Calibration Standard Impurities

Component	Vapor Density, mg/ml	Volume Added, ml	mg Added (1)	ppm Added (2)	Total ppm Present (3)
methane	0.656	4.0	2.62	130	
23	2.895	3.5	10.13	500	
32	2.162	4.7	10.16	502	
ethene	1.147	4.0	4.58	226	
ethane	1.230	4.0	4.92	243	
13	4.331	2.4	10.39	513	
143a	3.511	2.9	10.18	503	
125	5.004	2.1	10.50	519	
134a	4.279	2.4	10.26	507	
1301	6.205	1.7	10.54	521	
218	7.923	1.3	10.29	508	
propene	1.756	5.8	10.18	503	
115	6.486	1.6	10.37	512	
12	5.077	2.0	10.15	501	
124	5.758	1.8	10.36	512	
133a	4.846	2.1	10.17	502	
21	4.367	2.4	10.48	517	
isobutane	2.452	4.2	10.29	508	
114	7.319	1.4	10.24	506	
n-butane	2.463	4.2	10.34	511	
Component	**Liquid Density, mg/µl**	**Volume Added, µl**	**mg Added (1)**	**ppm Added (2)**	**Total ppm Present (3)**
11	1.537	6.6	10.14	501	
123	1.526	6.7	10.22	505	
113	1.619	6.3	10.19	503	

(1) If necessary, correct the mg added for the purity of the calibration component previously established.

(2) Values shown are for illustration; exact values are determined at Part 33, Section 9.3, steps c and d.

(3) Column to be filled in (Part 33, Section 9.3, step g) after determining ppm present in the stock components (see Note 1 in Section 10).

Part 38

R-406 Purity GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Part 33, *General Procedure for the Determination of Purity of New and Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography* (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-406 blends.

Section 2. Limitations and Interferences

This method is calibrated only for those impurities commonly present in R-406A. This method will not detect any impurity that may elute within the comparatively large HCFC-22, HCFC-142b, and isobutane peak matrices. The contaminants HFC-23 and HFC-32 also elute together, but these may be separated if needed by adding a 0.30 m column section of Porapak-T to the end of the primary column.

Section 3. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1A and B. The data was obtained by analyzing an R-406 calibration mixture 7 times during one day by one operator.

Section 4. Tables

The following tables are to be used in conjunction with Part 33 of the *General Procedure for the Determination of Purity of Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography*.

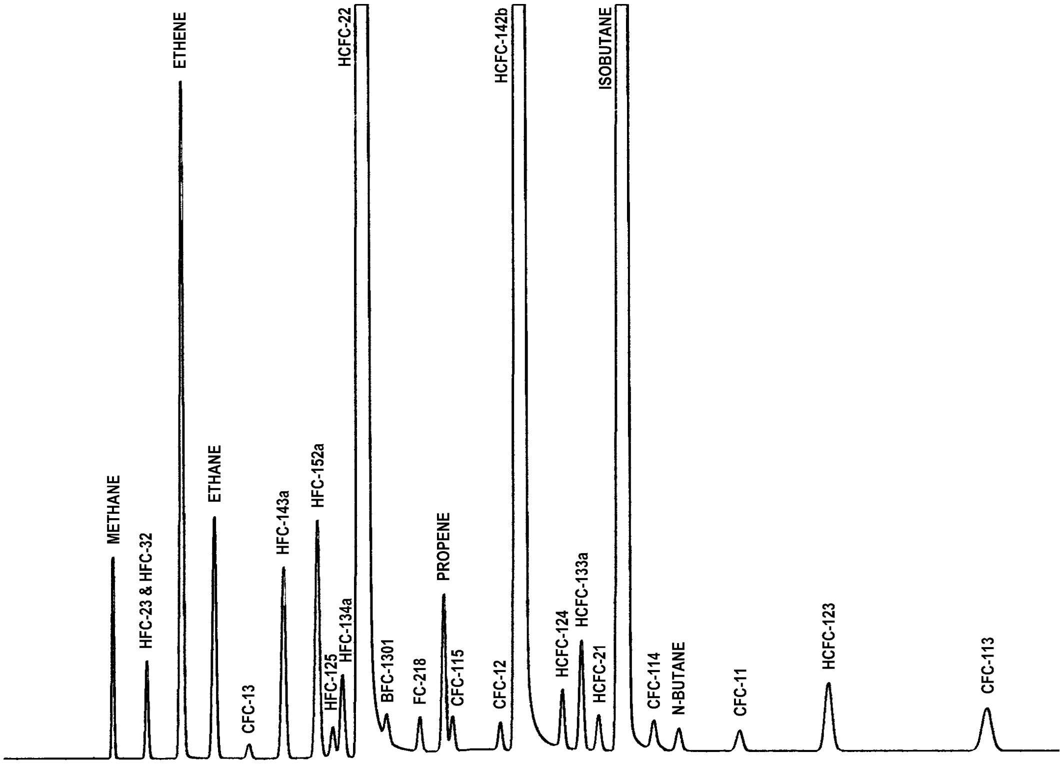


Figure 1. Gas Chromatogram of R-406

Table 1A. Contaminant Statistical Parameters

Component	Detection Limit, ppm	Range Investigated, ppm	Precision at 95% Confidence Limit, ppm
methane	1	100	3.3
23	11	450	24
32	4	450	9.0
ethene	1	450	15
ethane	1	200	6.5
13	13	450	18
143a	2	650	17
152a	1	450	30.
125	11	450	15
134a	2	450	20.
1301	11	450	6.4
218	6	450	18
propene	1	100	3.2
115	7	450	6.2
12	8	450	5.3
124	4	450	23
133a	2	450	14
21	5	450	4
114	6	450	10.
n-butane	3	100	2.9
11	7	450	6.2
123	2	450	5.0
113	2	450	7.1

Table 1B. Component Statistical Parameters

Component	Detection Limit, ppm	Range Investigated, %	Precision at 95% Confidence Limit
22	6	55 %	0.21 %
142b	4	41 %	0.35 %
isobutane	1	4 %	0.09 %

Table 1C. Blend Component Balance Preparation

Component	Final Pressure After Component Addition (kPa)	Weight of Refrigerant Added (g)
Isobutane	25	0.60
R-142b	166	6.15
R-22	371	8.25

Table 2. Primary Calibration Standard Impurities

Component	Vapor Density, mg/ml	Volume Added, ml	mg Added (1)	ppm Added (2)	Total ppm Present (3)
methane	0.656	4.0	2.62	130	
23	2.895	3.5	10.13	500	
32	2.162	4.7	10.16	502	
ethene	1.147	4.0	4.58	226	
ethane	1.230	4.0	4.92	243	
13	4.331	2.4	10.39	513	
143a	3.511	2.9	10.18	503	
152a	2.772	3.7	10.25	506	
125	5.004	2.1	10.50	519	
134a	4.279	2.4	10.26	507	
1301	6.205	1.7	10.54	521	
218	7.923	1.3	10.29	508	
propene	1.756	5.8	10.18	503	
115	6.486	1.6	10.37	512	
12	5.077	2.0	10.15	501	
124	5.758	1.8	10.36	512	
133a	4.846	2.1	10.17	502	
21	4.367	2.4	10.48	517	
114	7.319	1.4	10.24	506	
n-butane	2.463	4.2	10.34	511	
Component	**Liquid Density, mg/µl**	**Volume Added, µl**	**mg Added (1)**	**ppm Added (2)**	**Total ppm Present (3)**
11	1.537	6.6	10.14	501	
123	1.526	6.7	10.22	505	
113	1.619	6.3	10.19	503	

(1) If necessary, correct the mg added for the purity of the calibration component previously established.

(2) Values shown are for illustration; exact values are determined at Part 33, Section 9.3, steps c and d.

(3) Column to be filled in (Part 33, Section 9.3, step g) after determining ppm present in the stock components (see Note 1 in Section 10).

Part 39

R-407 Purity GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Part 33, *General Procedure for the Determination of Purity of New and Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography*. This GC Method Data Sheet is for use with R-407, blends of R-32, R-125, and R-134a.

Section 2. Limitations and Interferences

This method is calibrated only for those impurities commonly present in R-407 blends. This method will not detect any impurity that may elute within the comparatively large HFC-32, HFC-125, and HFC-134a peak matrices. Although this method only partially separates HFC-152a from HFC-125, the HFC-152a peak can be resolved by reducing the loop size to 100 µl.

Section 3. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1A and B. The data was obtained by analyzing an R-407 calibration mixture 7 times during one day by one operator.

Section 4. Tables

The following tables are to be used in conjunction with Part 33 of the *General Procedure for the Determination of Purity of Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography*.

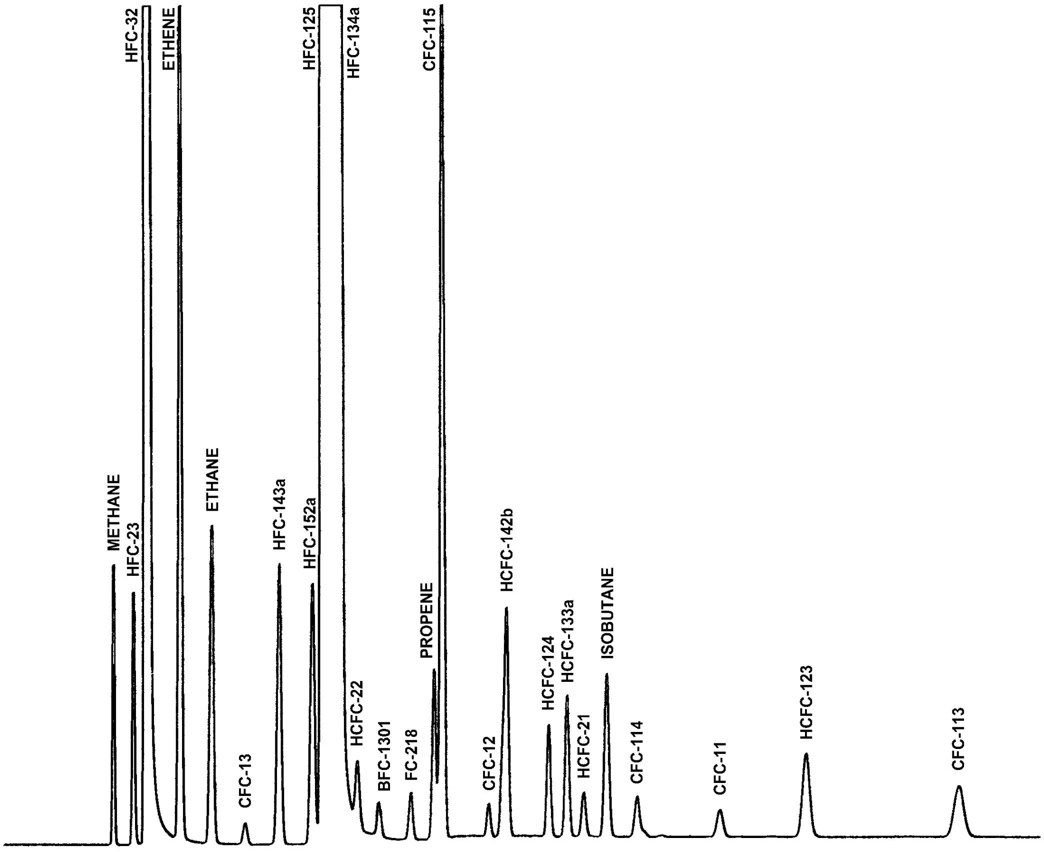


Figure 1. Gas Chromatogram of R-407

Table 1A. Contaminant Statistical Parameters			
Component	**Detection Limit, ppm**	**Range Investigated, ppm**	**Precision at 95% Confidence Limit, ppm**
methane	1	100	0.3
23	3	440	3.6
ethene	1	440	0.7
ethane	1	200	0.3
13	19	440	2.7
143a	2	440	30.
22	15	440	8.5
1301	14	440	11
218	10	440	5.5
propene	1	100	5.1
115	11	20,000	140
12	43	440	3.7
142b	3	440	3.3
124	5	440	1.5
133a	3	440	1.2
21	10	440	15
isobutane	1	100	0.4
114	8	440	3.2
11	11	440	13
123	3	440	5.3
113	4	440	11

Table 1B. Component Statistical Parameters			
Component	**Detection Limit, ppm**	**Range Investigated, %**	**Precision at 95% Confidence Limit**
32	3	20 %	0.05 %
125	4	40 %	0.14 %
134a	100	40 %	0.08 %

Table 1C. Blend Component Balance Preparation		
Component	**Final Pressure After Component Addition (kPa)**	**Weight of Refrigerant Added (g)**
R-134a	94	4.00
R-125	355	14.00
R-32	438	2.00

Table 2. Primary Calibration Standard Impurities					
Component	**Vapor Density, mg/ml**	**Volume Added, ml**	**mg Added (1)**	**ppm Added (2)**	**Total ppm Present (3)**
methane	0.656	4.0	2.62	130	
23	2.895	3.5	10.13	500	
ethene	1.147	4.0	4.58	226	
ethane	1.230	4.0	4.92	243	
13	4.331	2.4	10.39	513	
143a	3.511	2.9	10.18	503	
152a	2.772	3.7	10.25	506	
22	3.606	2.9	10.45	516	
1301	6.205	1.7	10.54	521	
218	7.923	1.3	10.29	508	
propene	1.756	5.8	10.18	503	
115	6.486	1.6	10.37	512	
12	5.077	2.0	10.15	501	
142b	4.248	2.4	10.19	503	
124	5.758	1.8	10.36	512	
133a	4.846	2.1	10.17	502	
21	4.367	2.4	10.48	517	
isobutane	2.452	4.2	10.29	508	
114	7.319	1.4	10.24	506	
Component	**Liquid Density, mg/µl**	**Volume Added, µl**	**mg Added (1)**	**ppm Added (2)**	**Total ppm Present (3)**
11	1.537	6.6	10.14	501	
123	1.526	6.7	10.22	505	
113	1.619	6.3	10.19	503	

(1) If necessary, correct the mg added for the purity of the calibration component previously established.

(2) Values shown are for illustration; exact values are determined at Part 33, Section 9.3, steps c and d.

(3) Column to be filled in (Part 33, Section 9.3, step g) after determining ppm present in the stock components (see Note 1 in Section 10).

Part 40

R-408 Purity GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Part 33, *General Procedure for the Determination of Purity of New and Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography* (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-408 blends.

Section 2. Limitations and Interferences

This method is calibrated only for those impurities commonly present in R-408A. This method will not detect any impurity that may elute within the comparatively large HCFC-22, HFC-125, and HFC-143a peak matrices. For example, HFC-134a elutes on the far shoulder of the large HFC-125 peak and is therefore difficult to detect at low concentrations. Linearity of the HFC-134a response is marginal and limits the precision in the determination of this impurity. The contaminants HFC-23 and HFC-32 also elute together, but these may be separated if needed by adding a 0.30 m column section of Porapak-T to the end of the primary column.

Section 3. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1A and B. The data was obtained by analyzing an R-408 calibration mixture 7 times during one day by one operator.

Section 4. Tables

The following tables are to be used in conjunction with Part 33 of the *General Procedure for the Determination of Purity of Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography*.

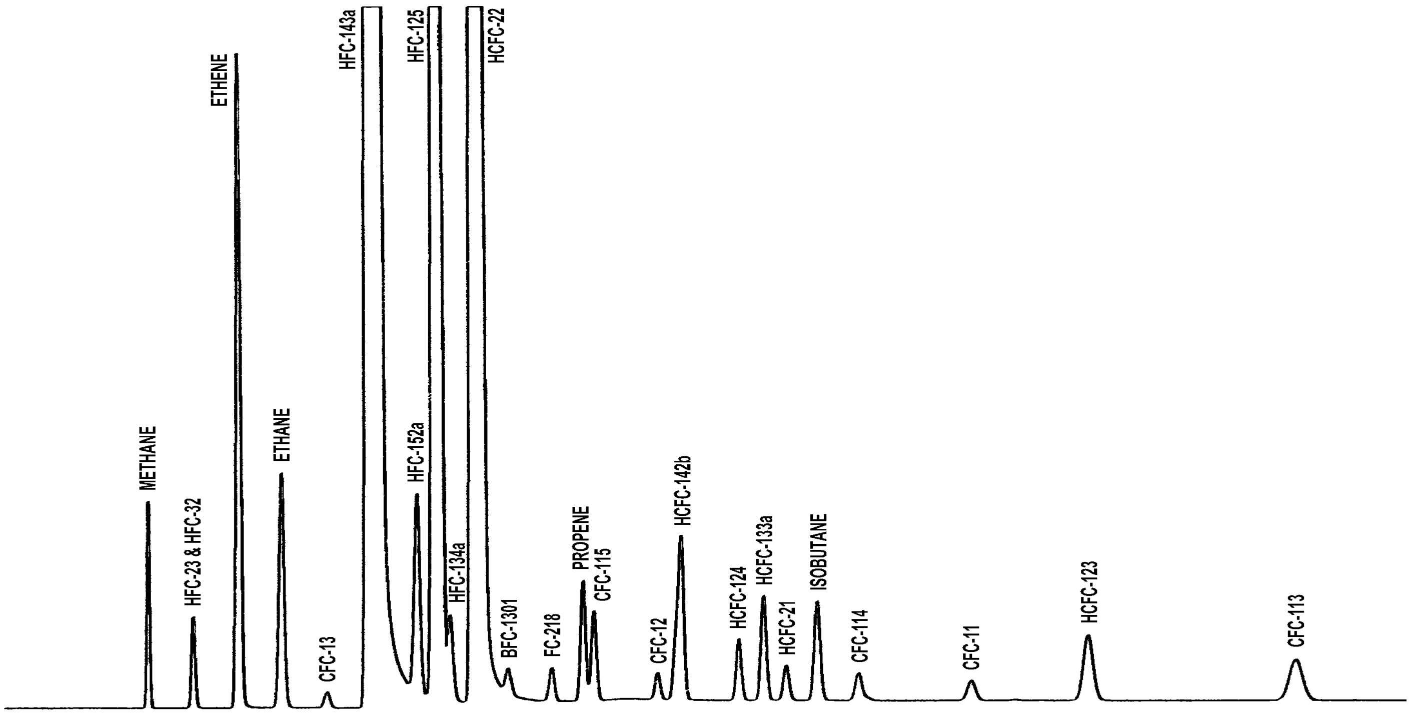


Table 1. Gas Chromatogram of R-408A

Table 1A. Contaminant Statistical Parameters

Component	Detection Limit, ppm	Range Investigated, ppm	Precision at 95% Confidence Limit, ppm
methane	1	100	0.6
23	10	430	2.1
32	4	430	1.8
ethene	1	430	2.6
ethane	1	200	1.6
13	11	430	7.5
152a	1	430	37
134a	100	430	3.4
1301	9	430	7.5
218	6	430	1.5
propene	1	100	6.8
115	7	1400	11
12	6	430	51
142b	1	430	4.3
124	4	430	2.3
133a	2	430	0.9
21	5	430	11
isobutane	1	100	0.9
114	5	430	0.8
11	7	430	12
123	2	430	4.6
113	2	430	7.9

Table 1B. Component Statistical Parameters

Component	Detection Limit, ppm	Range Investigated, %	Precision at 95% Confidence Limit
143a	3	46 %	0.06 %
125	5	7 %	0.10 %
22	6	47 %	0.17 %

Table 1C. Blend Component Balance Preparation

Component	Final Pressure After Component Addition (kPa)	Weight of Refrigerant Added (g)
R-22	254	9.40
R-143a	487	9.20
R-125	510	1.40

Table 2. Primary Calibration Standard Impurities

Component	Vapor Density, mg/ml	Volume Added, ml	mg Added (1)	ppm Added (2)	Total ppm Present (3)
methane	0.656	4.0	2.62	130	
23	2.895	3.5	10.13	500	
32	2.162	4.7	10.16	502	
ethene	1.147	4.0	4.58	226	
ethane	1.230	4.0	4.92	243	
13	4.331	2.4	10.39	513	
152a	2.772	3.7	10.25	506	
134a	4.279	2.4	10.26	507	
1301	6.205	1.7	10.54	521	
218	7.923	1.3	10.29	508	
propene	1.756	5.8	10.18	503	
115	6.486	1.6	10.37	512	
12	5.077	2.0	10.15	501	
142b	4.248	2.4	10.19	503	
124	5.758	1.8	10.36	512	
133a	4.846	2.1	10.17	502	
21	4.367	2.4	10.48	517	
isobutane	2.452	4.2	10.29	508	
114	7.319	1.4	10.24	506	
Component	**Liquid Density, mg/µl**	**Volume Added, µl**	**mg Added (1)**	**ppm Added (2)**	**Total ppm Present (3)**
11	1.537	6.6	10.14	501	
123	1.526	6.7	10.22	505	
113	1.619	6.3	10.19	503	

(1) If necessary, correct the mg added for the purity of the calibration component previously established.

(2) Values shown are for illustration; exact values are determined at Part 33, Section 9.3, steps c and d.

(3) Column to be filled in (Part 33, Section 9.3, step g) after determining ppm present in the stock components (see Note 1 in Section 10).

Part 41

R-409 Purity GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Part 33, *General Procedure for the Determination of Purity of New and Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography* (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-409 blends.

Section 2. Limitations and Interferences

This method is calibrated only for those impurities commonly present in R-409 blends. This method will not detect any impurity that may elute within the comparatively large HCFC-22, HCFC-124, and HCFC-142b peak matrices. The contaminants HFC-23 and HFC-32 also elute together, but these may be separated if needed by adding a 0.30 m column section of Porapak-T to the end of the primary column.

Section 3. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1A and B. The data was obtained by analyzing an R-409 calibration mixture 7 times during one day by one operator.

Section 4. Tables

The following tables are to be used in conjunction with Part 33 of the *General Procedure for the Determination of Purity of Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography*.

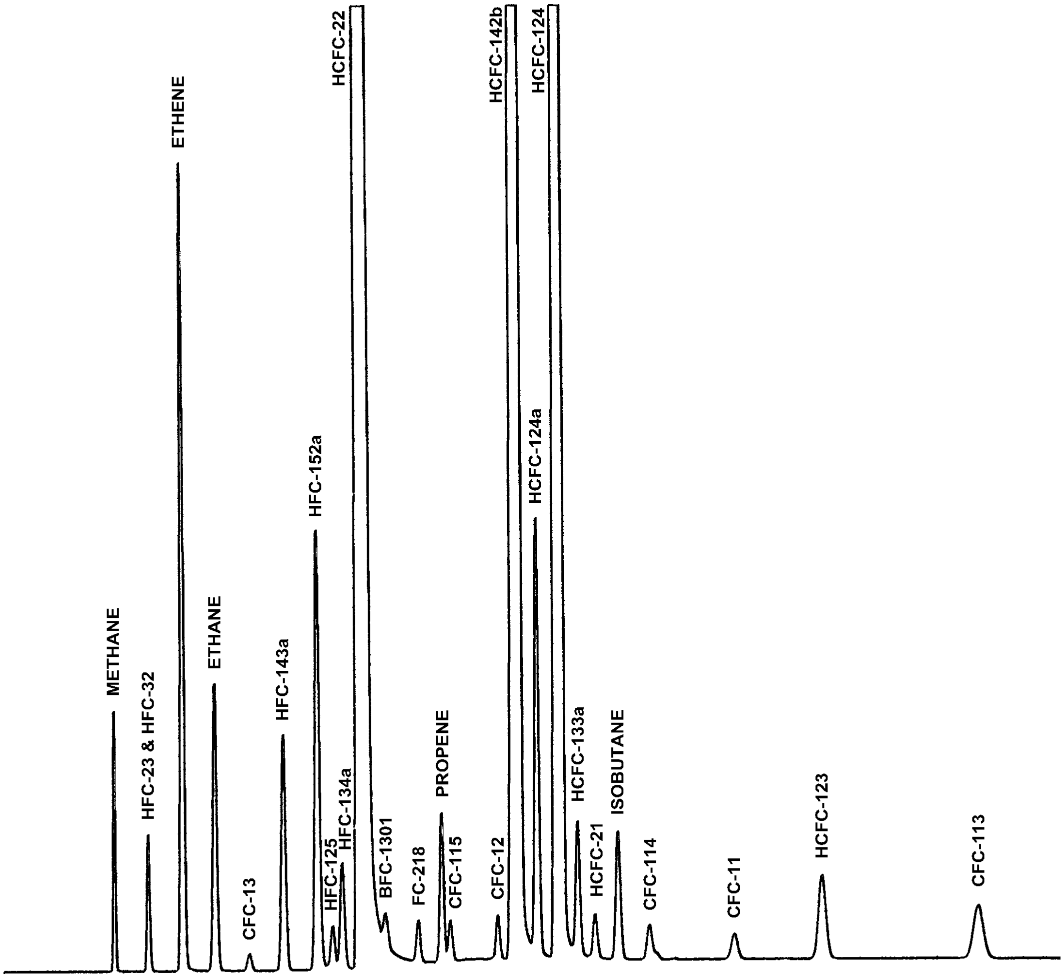


Figure 1. Gas Chromatogram of R-409

Table 1A. Contaminant Statistical Parameters			
Component	**Detection Limit, ppm**	**Range Investigated, ppm**	**Precision at 95% Confidence Limit, ppm**
methane	1	100	2.8
23	16	440	12
32	9	440	9.3
ethene	1	440	9.2
ethane	1	200	4.2
13	25	440	8.0
143a	3	440	9.2
152a	2	440	3.2
125	13	440	13
134a	5	440	7.0
1301	24	440	9.6
218	12	440	9.5
propene	1	100	0.5
115	14	440	27
12	14	440	2.7
133a	4	440	13
21	12	440	31
isobutane	1	100	1.6
114	10	440	1.7
11	12	440	42
123	3	440	41
113	3	440	66

Table 1B. Component Statistical Parameters			
Component	**Detection Limit, ppm**	**Range Investigated, %**	**Precision at 95% Confidence Limit**
22	6	60 %	0.34 %
142b	3	15 %	0.17 %
124	5	25 %	0.03 %

Table 1C. Blend Component Balance Preparation		
Component	**Final Pressure After Component Addition (kPa)**	**Weight of Refrigerant Added (g)**
R-142b	71	3.00
R-124	155	5.00
R-22	450	12.00

Table 2. Primary Calibration Standard Impurities

Component	**Vapor Density, mg/ml**	**Volume Added, ml**	**mg Added (1)**	**ppm Added (2)**	**Total ppm Present (3)**
methane	0.656	4.0	2.62	130	
23	2.895	3.5	10.13	500	
32	2.162	4.7	10.16	502	
ethene	1.147	4.0	4.58	226	
ethane	1.230	4.0	4.92	243	
13	4.331	2.4	10.39	513	
143a	3.511	2.9	10.18	503	
152a	2.772	3.7	10.25	506	
125	5.004	2.1	10.50	519	
134a	4.279	2.4	10.26	507	
1301	6.205	1.7	10.54	521	
218	7.923	1.3	10.29	508	
propene	1.756	5.8	10.18	503	
115	6.486	1.6	10.37	512	
12	5.077	2.0	10.15	501	
133a	4.846	2.1	10.17	502	
21	4.367	2.4	10.48	517	
isobutane	2.452	4.2	10.29	508	
114	7.319	1.4	10.24	506	
Component	**Liquid Density, mg/µl**	**Volume Added, µl**	**mg Added (1)**	**ppm Added (2)**	**Total ppm Present (3)**
11	1.537	6.6	10.14	501	
123	1.526	6.7	10.22	505	
113	1.619	6.3	10.19	503	

(1) If necessary, correct the mg added for the purity of the calibration component previously established.

(2) Values shown are for illustration; exact values are determined at Part 33, Section 9.3, steps c and d.

(3) Column to be filled in (Part 33, Section 9.3, step g) after determining ppm present in the stock components (see Note 1 in Section 10).

Part 42

R-410 Purity GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Part 33, *General Procedure for the Determination of Purity of New and Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography* (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-410 blends.

Section 2. Limitations and Interferences

This method is calibrated only for those impurities commonly present in R-410 blends. This method will not detect any impurity that may elute within the comparatively large HFC-32 and HFC-125 peak matrices. HFC-134a elutes on the far shoulder of the large HFC-125 peak, is poorly resolved and is difficult to detect at low concentrations.

Section 3. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1A and B. The data was obtained by analyzing an R-410 calibration mixture 7 times during one day by one operator.

Section 4. Tables

The following tables are to be used in conjunction with Part 33 of the *General Procedure for the Determination of Purity of Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography*.

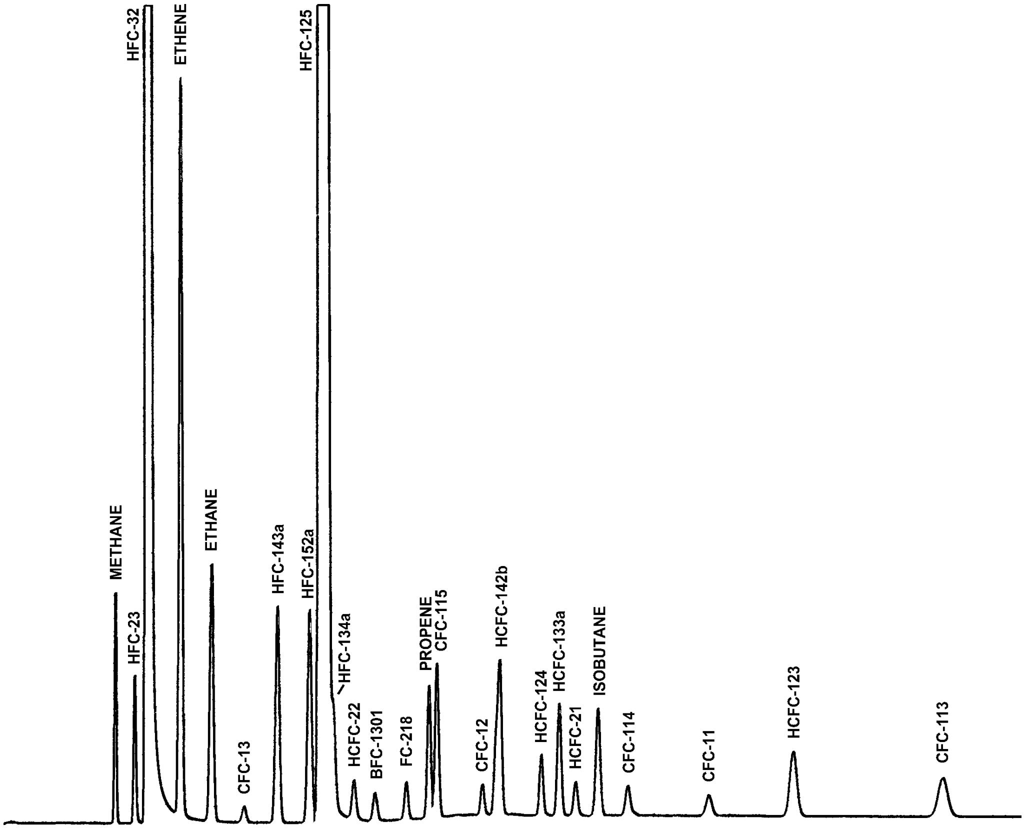


Figure 1. Gas Chromatogram of R-410

Table 1A. Contaminant Statistical Parameters			
Component	**Detection Limit, ppm**	**Range Investigated, ppm**	**Precision at 95% Confidence Limit, ppm**
methane	1	120	2.1
23	5	510	5.4
ethene	1	510	8.7
ethane	1	230	4.1
13	13	510	11
143a	3	550	7.3
152a	2	510	11
134a	100	510	17
22	7	510	14
1301	8	510	17
218	6	510	15
propene	1	130	1.6
115	6	1900	23
12	9	510	12
142b	1	510	5.8
124	4	510	6.7
133a	2	510	7.0
21	7	510	10.
isobutane	1	130	2.1
114	6	510	10.
11	8	510	23
123	2	510	5.2
113	3	510	6.4

Table 1B. Component Statistical Parameters			
Component	**Detection Limit, ppm**	**Range Investigated, %**	**Precision at 95% Confidence Limit**
32	7	50 %	0.38 %
125	5	50 %	0.25 %

Table 1C. Blend Component Balance Preparation		
Component	**Final Pressure After Component Addition (kPa)**	**Weight of Refrigerant Added (g)**
R-125	197	10.00
R-32	616	10.00

Table 2. Primary Calibration Standard Impurities

Component	Vapor Density, mg/ml	Volume Added, ml	mg Added (1)	ppm Added (2)	Total ppm Present (3)
methane	0.656	4.0	2.62	130	
23	2.895	3.5	10.13	500	
ethene	1.147	4.0	4.58	226	
ethane	1.230	4.0	4.92	243	
13	4.331	2.4	10.39	513	
143a	3.511	2.9	10.18	503	
152a	2.772	3.7	10.25	506	
134a	4.279	2.4	10.26	507	
22	3.606	2.9	10.45	516	
1301	6.205	1.7	10.54	521	
218	7.923	1.3	10.29	508	
propene	1.756	5.8	10.18	503	
115	6.486	1.6	10.37	512	
12	5.077	2.0	10.15	501	
142b	4.248	2.4	10.19	503	
124	5.758	1.8	10.36	512	
133a	4.846	2.1	10.17	502	
21	4.367	2.4	10.48	517	
isobutane	2.452	4.2	10.29	508	
114	7.319	1.4	10.24	506	
Component	**Liquid Density, mg/µl**	**Volume Added, µl**	**mg Added (1)**	**ppm Added (2)**	**Total ppm Present (3)**
11	1.537	6.6	10.14	501	
123	1.526	6.7	10.22	505	
113	1.619	6.3	10.19	503	

(1) If necessary, correct the mg added for the purity of the calibration component previously established.

(2) Values shown are for illustration; exact values are determined at Part 33, Section 9.3, steps c and d.

(3) Column to be filled in (Part 33, Section 9.3, step g) after determining ppm present in the stock components (see Note 1 in Section 10).

Part 43

R-411 Purity GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Part 33, *General Procedure for the Determination of Purity of New and Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography* (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-411 blends.

Section 2. Limitations and Interferences

This method is calibrated only for those impurities commonly present in R-411 blends. This method will not detect any impurity that may elute within the comparatively large HCFC-22, HFC-152a, and propene peak matrices. For example, HFC-125 elutes on the far shoulder of the large HFC-152a peak, is poorly resolved and is difficult to detect at low concentrations. The same difficulty is encountered measuring the CFC-115 contaminant because of its proximity to the large propene peak matrix. The contaminants HFC-23 and HFC-32 also elute together, but these may be separated if needed by adding a 0.30 m column section of Porapak-T to the end of the primary column.

Section 3. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1A and B. The data was obtained by analyzing an R-411 calibration mixture 7 times during one day by one operator.

Section 4. Tables

The following tables are to be used in conjunction with Part 33 of the *General Procedure for the Determination of Purity of Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography*.

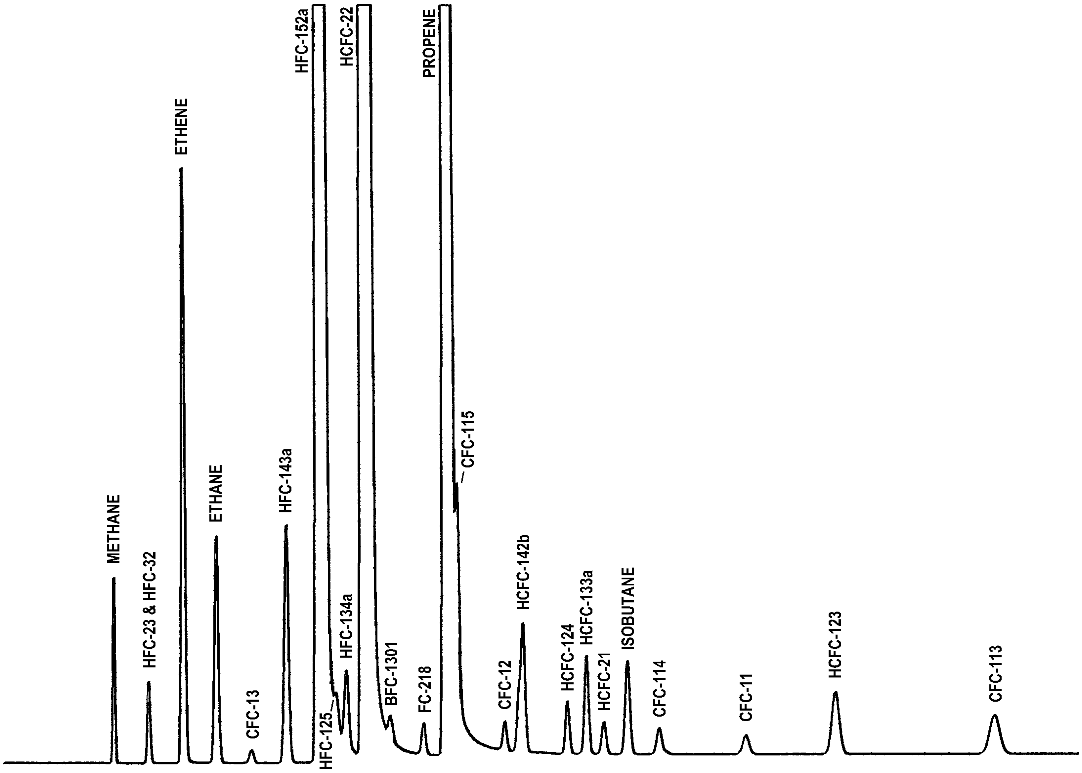


Figure 1. Gas Chromatogram of R-411

Table 1A. Contaminant Statistical Parameters

Component	Detection Limit, ppm	Range Investigated, ppm	Precision at 95% Confidence Limit, ppm
methane	1	100	12
23	7	420	36
32	5	420	17
ethene	1	420	12
ethane	1	200	6.5
13	1	420	30.
143a	2	420	19
134a	3	420	57
1301	14	420	14
218	6	420	14
115	10	420	25
12	9	420	5.3
142b	2	420	12
124	5	420	22
133a	2	420	25
21	7	420	22
isobutane	1	100	4.2
114	6	420	22
11	9	420	12
123	2	420	34
113	3	420	36

Table 1B. Component Statistical Parameters

Component	Detection Limit, ppm	Range Investigated, %	Precision at 95% Confidence Limit
152a	1	3 %	0.16 %
22	9	94 %	2.6 %
propene	1	3 %	0.06 %

Table 1C. Blend Component Balance Preparation

Component	Final Pressure After Component Addition (kPa)	Weight of Refrigerant Added (g)
Propene	35	0.60
R-152a	56	0.60
R-22	533	18.80

Table 2. Primary Calibration Standard Impurities

Component	Vapor Density, mg/ml	Volume Added, ml	mg Added (1)	ppm Added (2)	Total ppm Present (3)
methane	0.656	4.0	2.62	130	
23	2.895	3.5	10.13	500	
32	2.162	4.7	10.16	502	
ethene	1.147	4.0	4.58	226	
ethane	1.230	4.0	4.92	243	
13	4.331	2.4	10.39	513	
143a	3.511	2.9	10.18	503	
125	5.004	2.1	10.50	519	
134a	4.279	2.4	10.26	507	
1301	6.205	1.7	10.54	521	
218	7.923	1.3	10.29	508	
115	6.486	1.6	10.37	512	
12	5.077	2.0	10.15	501	
142b	4.248	2.4	10.19	503	
124	5.758	1.8	10.36	512	
133a	4.846	2.1	10.17	502	
21	4.367	2.4	10.48	517	
isobutane	2.452	4.2	10.29	508	
114	7.319	1.4	10.24	506	
Component	**Liquid Density, mg/µl**	**Volume Added, µl**	**mg Added (1)**	**ppm Added (2)**	**Total ppm Present (3)**
11	1.537	6.6	10.14	501	
123	1.526	6.7	10.22	505	
113	1.619	6.3	10.19	503	

(1) If necessary, correct the mg added for the purity of the calibration component previously established.

(2) Values shown are for illustration; exact values are determined in Part 33, Section 9.3, steps c and d.

(3) Column to be filled in (Part 33, Section 9.3, step g) after determining ppm present in the stock components (see Note 1 in Section 10).

Part 44

R-412 Purity GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Part 33, *General Procedure for the Determination of Purity of New and Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography* (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-412 blends.

Section 2. Limitations and Interferences

This method is calibrated only for those impurities commonly present in R-412A. This method will not detect any impurity that may elute within the comparatively large HCFC-22, HCFC-142b, and FC-218 peak matrices. The contaminants HFC-23 and HFC-32 also elute together, but these may be separated if needed by adding a 0.30 m column section of Porapak-T to the end of the primary column.

Section 3. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1A and B. The data was obtained by analyzing an R-412 calibration mixture 7 times during one day by one operator.

Section 4. Tables

The following tables are to be used in conjunction with Part 33 of the *General Procedure for the Determination of Purity of Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography*.

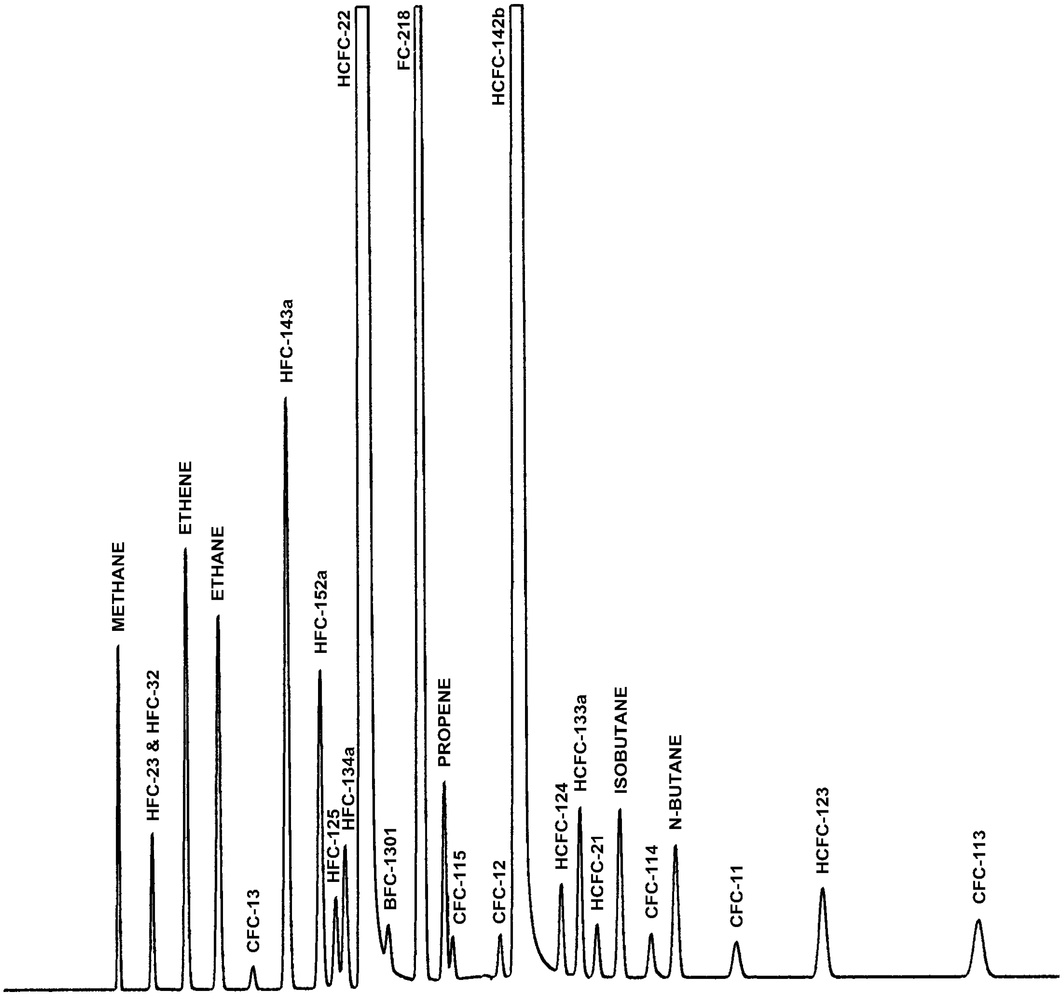


Figure 1. Gas Chromatogram of R-412

Table 1A. Contaminant Statistical Parameters			
Component	**Detection Limit, ppm**	**Range Investigated, ppm**	**Precision at 95% Confidence Limit, ppm**
methane	1	100	1.0
23	10	520	7.1
32	4	520	4.3
ethene	1	200	2.2
ethane	1	200	2.3
13	12	520	6.2
143a	1	700	9.2
152a	1	520	48
125	6	520	11
134a	2	520	6.9
1301	10	520	13
propene	1	150	7.4
115	6	520	16
12	7	520	19
124	4	520	5.2
133a	2	520	1.0
21	5	520	14
isobutane	1	150	0.3
114	5	520	3.9
n-butane	1	150	0.2
11	5	520	21
123	2	520	8.2
113	3	520	14

Table 1B. Component Statistical Parameters			
Component	**Detection Limit, ppm**	**Range Investigated, %**	**Precision at 95% Confidence Limit**
22	7	70 %	0.21 %
218	5	5 %	0.06 %
142b	2	25 %	0.08%

Table 1C. Blend Component Balance Preparation		
Component	**Final Pressure After Component Addition (kPa)**	**Weight of Refrigerant Added (g)**
FC-218	13	1.00
R-142b	129	5.00
R-22	475	14.00

Table 2. Primary Calibration Standard Impurities

Component	Vapor Density, mg/ml	Volume Added, ml	mg Added (1)	ppm Added (2)	Total ppm Present (3)
methane	0.656	4.0	2.62	130	
23	2.895	3.5	10.13	500	
32	2.162	4.7	10.16	502	
ethene	1.147	4.0	4.58	226	
ethane	1.230	4.0	4.92	243	
13	4.331	2.4	10.39	513	
143a	3.511	2.9	10.18	503	
152a	2.772	3.7	10.25	506	
125	5.004	2.1	10.50	519	
134a	4.279	2.4	10.26	507	
1301	6.205	1.7	10.54	521	
propene	1.756	5.8	10.18	503	
115	6.486	1.6	10.37	512	
12	5.077	2.0	10.15	501	
124	5.758	1.8	10.36	512	
133a	4.846	2.1	10.17	502	
21	4.367	2.4	10.48	517	
isobutane	2.452	4.2	10.29	508	
114	7.319	1.4	10.24	506	
n-butane	2.463	4.2	10.34	511	
Component	**Liquid Density, mg/µl**	**Volume Added, µl**	**mg Added (1)**	**ppm Added (2)**	**Total ppm Present (3)**
11	1.537	6.6	10.14	501	
123	1.526	6.7	10.22	505	
113	1.619	6.3	10.19	503	

(1) If necessary, correct the mg added for the purity of the calibration component previously established.

(2) Values shown are for illustration; exact values are determined at Part 33, Section 9.3, steps c and d.

(3) Column to be filled in (Part 33, Section 9.3, step g) after determining ppm present in the stock components (see Note 1 in Section 10).

Part 45

R-500 Purity GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Part 33, *General Procedure for the Determination of Purity of New and Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography* (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-500.

Section 2. Limitations and Interferences

This method is calibrated only for those impurities commonly present in R-500. This method will not detect any impurity that may elute within the comparatively large CFC-12 and HFC-152a peak matrices. For example, HFC-134a elutes on the far shoulder of the large HFC-152a peak and is therefore difficult to detect at low concentrations. Linearity of the HFC-134a response is marginal and limits the precision in the determination of this impurity. The contaminants HFC-23 and HFC-32 also elute together, but these may be separated if needed by adding a 0.30 m column section of Porapak-T to the end of the primary column.

Section 3. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1A and B. The data was obtained by analyzing an R-500 calibration mixture 7 times during one day by one operator.

Section 4. Tables

The following tables are to be used in conjunction with Part 33 of the *General Procedure for the Determination of Purity of Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography*.

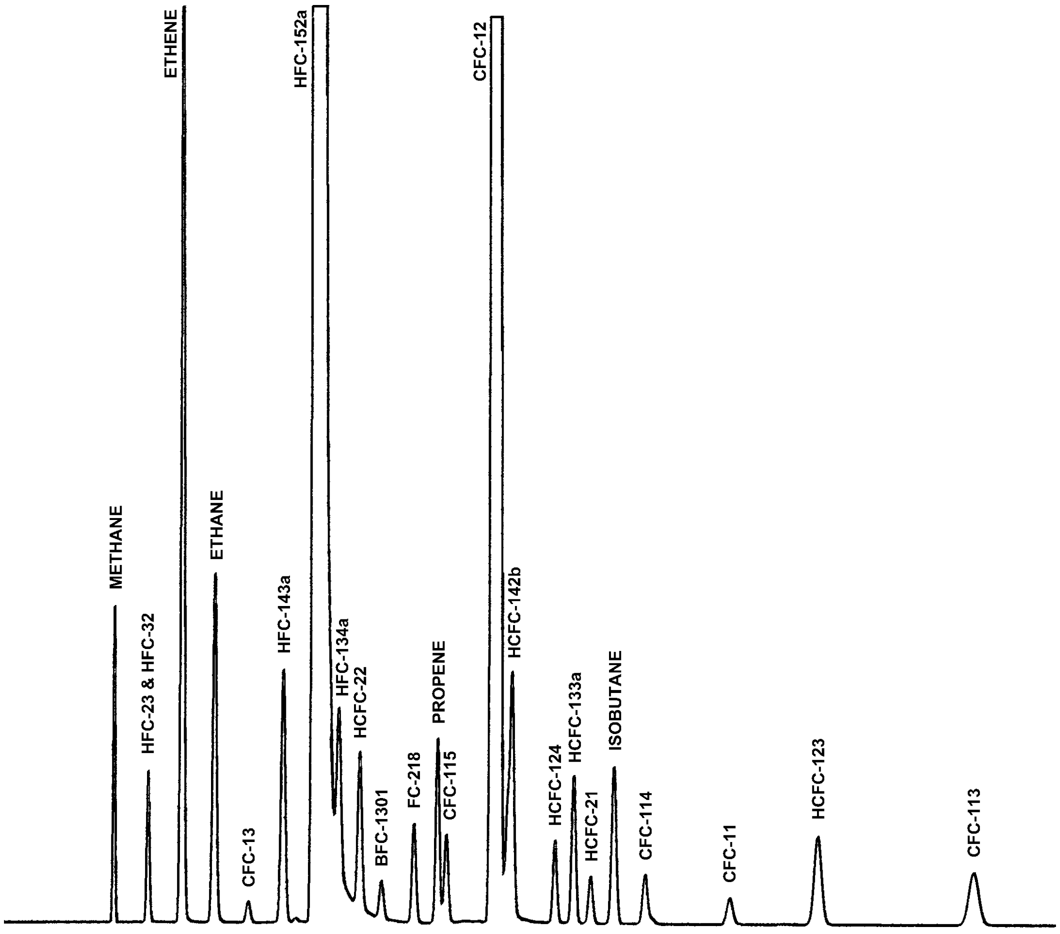


Figure 1. Gas Chromatogram of R-500

Table 1A. Contaminant Statistical Parameters

Component	Detection Limit, ppm	Range Investigated, ppm	Precision at 95% Confidence Limit, ppm
methane	2	100	1.1
23	7	440	8.5
32	4	440	3.3
ethene	1	440	2.3
ethane	1	200	0.9
13	9	440	5.0
143a	1	440	1.7
134a	3	440	1.7
22	5	440	3.1
1301	7	440	3.7
218	5	440	1.4
propene	1	100	4.5
115	6	440	8.5
142b	2	440	4.6
124	3	440	4.2
133a	2	440	5.4
21	5	440	12
isobutane	1	100	6.6
114	4	440	4.2
11	6	440	14
123	2	440	16
113	2	440	23

Table 1B. Component Statistical Parameters

Component	Detection Limit, ppm	Range Investigated, %	Precision at 95% Confidence Limit
152a	2	26.2 %	0.02 %
12	7	73.8 %	0.07 %

Table 1C. Blend Component Balance Preparation

Component	Final Pressure After Component Addition (kPa)	Weight of Refrigerant Added (g)
R-152a	185	5.24
R-12	441	14.76

Table 2. Primary Calibration Standard Impurities

Component	Vapor Density, mg/ml	Volume Added, ml	mg Added (1)	ppm Added (2)	Total ppm Present (3)
methane	0.656	4.0	2.62	130	
23	2.895	3.5	10.13	500	
32	2.162	4.7	10.16	502	
ethene	1.147	4.0	4.58	226	
ethane	1.230	4.0	4.92	243	
13	4.331	2.4	10.39	513	
143a	3.511	2.9	10.18	503	
134a	4.279	2.4	10.26	507	
22	3.606	2.9	10.45	516	
1301	6.205	1.7	10.54	521	
218	7.923	1.3	10.29	508	
propene	1.756	5.8	10.18	503	
115	6.486	1.6	10.37	512	
142b	4.248	2.4	10.19	503	
124	5.758	1.8	10.36	512	
133a	4.846	2.1	10.17	502	
21	4.367	2.4	10.48	517	
isobutane	2.452	4.2	10.29	508	
114	7.319	1.4	10.24	506	
Component	**Liquid Density, mg/µl**	**Volume Added, µl**	**mg Added (1)**	**ppm Added (2)**	**Total ppm Present (3)**
11	1.537	6.6	10.14	501	
123	1.526	6.7	10.22	505	
113	1.619	6.3	10.19	503	

(1) If necessary, correct the mg added for the purity of the calibration component previously established.

(2) Values shown are for illustration; exact values are determined at Part 33, Section 9.3, steps c and d.

(3) Column to be filled in (Part 33, Section 9.3, step g) after determining ppm present in the stock components (see Note 1 in Section 10).

Part 46

R-502 Purity GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Part 33, *General Procedure for the Determination of Purity of New and Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography* (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-502.

Section 2. Limitations and Interferences

This method is calibrated only for those impurities commonly present in R-502. This method will not detect any impurity that may elute within the comparatively large HCFC-22 and CFC-115 peak matrices. Propene and CFC-115 are poorly resolved which complicates the quantification of the propene. The contaminants HFC-23 and HFC-32 elute together, but these may be separated if needed by adding a 0.30 m column section of Porapak-T to the end of the primary column.

Section 3. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1A and B. The data was obtained by analyzing an R-502 calibration mixture 7 times during one day by one operator.

Section 4. Tables

The following tables are to be used in conjunction with Part 33 of the *General Procedure for the Determination of Purity of Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography*.

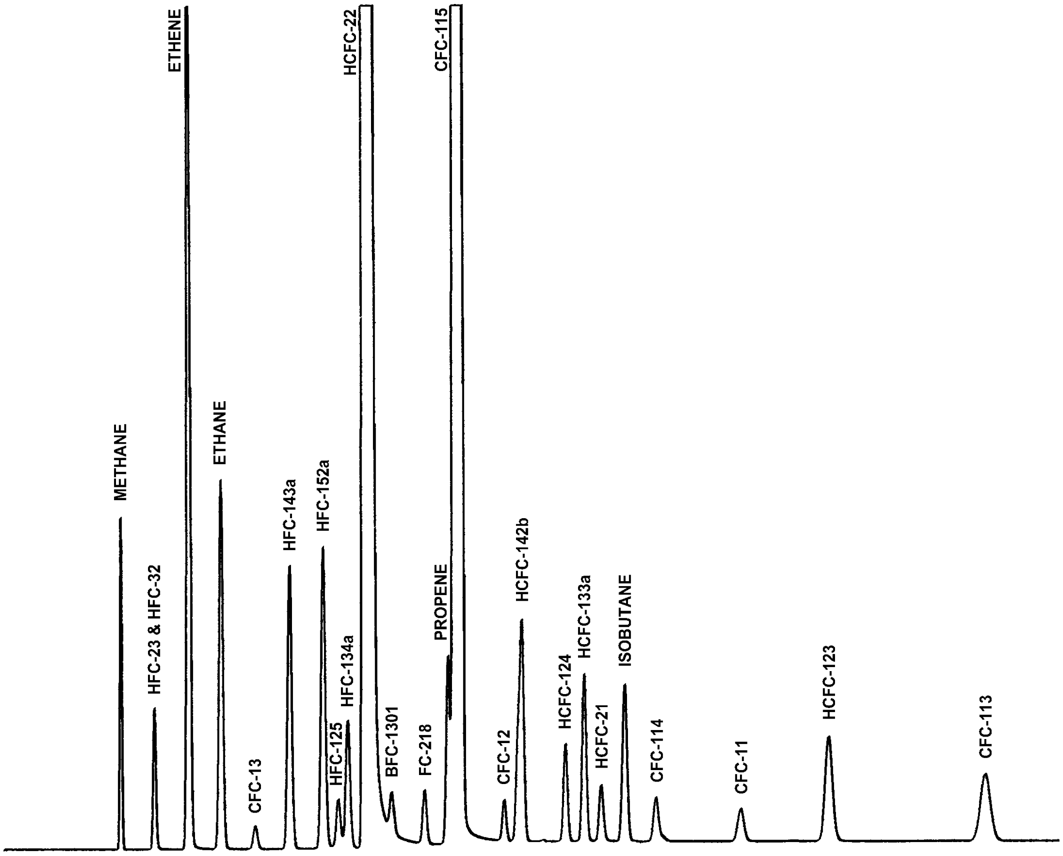


Figure 1. Gas Chromatogram of R-502

Table 1A. Contaminant Statistical Parameters

Component	Detection Limit, ppm	Range Investigated, ppm	Precision at 95% Confidence Limit, ppm
methane	1	100	8.4
23	5	440	13
32	3	440	15
ethene	1	440	8.7
ethane	1	200	3.5
13	8	440	11
143a	1	440	11
152a	1	440	11
125	5	440	14
134a	2	440	16
1301	21	440	31
218	5	440	14
propene	1	100	8.7
12	5	440	16
142b	1	440	11
124	2	440	11
133a	2	440	9.3
21	4	440	32
isobutane	1	100	0.9
114	4	440	9.5
11	9	440	74
123	2	440	11
113	3	440	21

Table 1B. Component Statistical Parameters

Component	Detection Limit, ppm	Range Investigated, %	Precision at 95% Confidence Limit
22	5	48.8 %	0.11 %
115	5	51.2 %	0.10 %

Table 1C. Blend Component Balance Preparation

Component	Final Pressure After Component Addition (kPa)	Weight of Refrigerant Added (g)
R-115	156	10.24
R-22	405	9.76

Table 2. Primary Calibration Standard Impurities

Component	Vapor Density, mg/ml	Volume Added, ml	mg Added (1)	ppm Added (2)	Total ppm Present (3)
methane	0.656	4.0	2.62	130	
23	2.895	3.5	10.13	500	
32	2.162	4.7	10.16	502	
ethene	1.147	4.0	4.58	226	
ethane	1.230	4.0	4.92	243	
13	4.331	2.4	10.39	513	
143a	3.511	2.9	10.18	503	
152a	2.772	3.7	10.25	506	
125	5.004	2.1	10.50	519	
134a	4.279	2.4	10.26	507	
1301	6.205	1.7	10.54	521	
218	7.923	1.3	10.29	508	
propene	1.756	5.8	10.18	503	
12	5.077	2.0	10.15	501	
142b	4.248	2.4	10.19	503	
124	5.758	1.8	10.36	512	
133a	4.846	2.1	10.17	502	
21	4.367	2.4	10.48	517	
isobutane	2.452	4.2	10.29	508	
114	7.319	1.4	10.24	506	
Component	**Liquid Density, mg/µl**	**Volume Added, µl**	**mg Added (1)**	**ppm Added (2)**	**Total ppm Present (3)**
11	1.537	6.6	10.14	501	
123	1.526	6.7	10.22	505	
113	1.619	6.3	10.19	503	

(1) If necessary, correct the mg added for the purity of the calibration component previously established.

(2) Values shown are for illustration; exact values are determined at Part 33, Section 9.3, steps c and d.

(3) Column to be filled in (Part 33, Section 9.3, step g) after determining ppm present in the stock components (see Note 1 in Section 10).

Part 47

R-503 Purity GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Part 33, *General Procedure for the Determination of Purity of New and Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography* (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-503.

Section 2. Limitations and Interferences

This method is calibrated only for those impurities commonly present in R-503. This method will not detect any impurity that may elute within the comparatively large CFC-13 and HFC-23 peak matrices. For example, HFC-32 cannot be detected using this method as it elutes within the large HFC-23 component peak matrix.

Section 3. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1A and B. The data was obtained by analyzing an R-503 calibration mixture 7 times during one day by one operator.

Section 4. Tables

The following tables are to be used in conjunction with Part 33 of the *General Procedure for the Determination of Purity of Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography*.

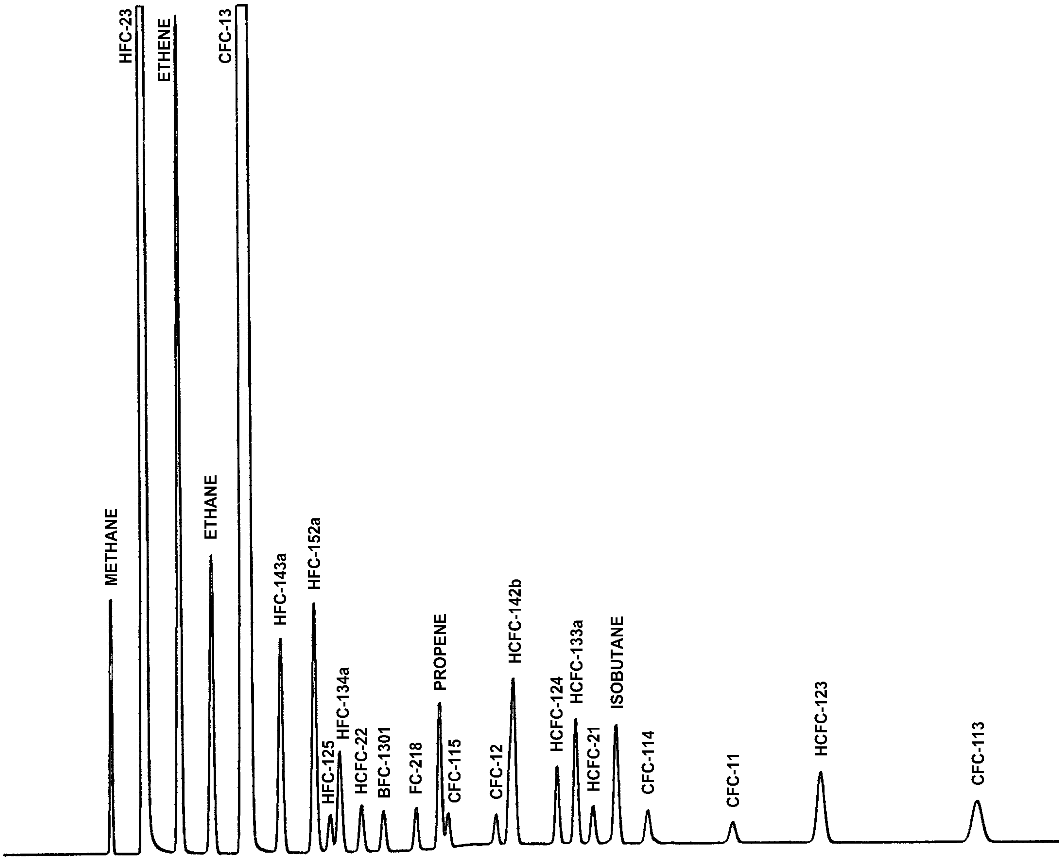


Figure 1. Gas Chromatogram of R-503

Table 1A. Contaminant Statistical Parameters

Component	Detection Limit, ppm	Range Investigated, ppm	Precision at 95% Confidence Limit, ppm
methane	1	100	0.9
ethene	1	440	3.9
ethane	1	200	1.6
143a	2	440	15
152a	1	440	4.2
125	7	440	14
134a	2	440	5.6
22	5	440	20.
1301	7	440	2.5
218	5	440	3.9
propene	1	100	3.1
115	7	440	20.
12	8	440	1.0
142b	2	440	10.
124	3	440	19
133a	2	440	16
21	5	440	7.9
isobutane	1	100	1.7
114	5	440	9.1
11	7	440	20
123	2	440	14
113	3	440	15

Table 1B. Component Statistical Parameters

Component	Detection Limit, ppm	Range Investigated, %	Precision at 95% Confidence Limit
23	11	40.1 %	0.07 %
13	10	59.9 %	0.09 %

Table 1C. Blend Component Balance Preparation

Component	Final Pressure After Component Addition (kPa)	Weight of Refrigerant Added (g)
R-13	272	11.98
R-23	533	8.02

Table 2. Primary Calibration Standard Impurities

Component	Vapor Density, mg/ml	Volume Added, ml	mg Added (1)	ppm Added (2)	Total ppm Present (3)
methane	0.656	4.0	2.62	130	
ethene	1.147	4.0	4.58	226	
ethane	1.230	4.0	4.92	243	
143a	3.511	2.9	10.18	503	
152a	2.772	3.7	10.25	506	
125	5.004	2.1	10.50	519	
134a	4.279	2.4	10.26	507	
22	3.606	2.9	10.45	516	
1301	6.205	1.7	10.54	521	
218	7.923	1.3	10.29	508	
propene	1.756	5.8	10.18	503	
115	6.486	1.6	10.37	512	
12	5.077	2.0	10.15	501	
142b	4.248	2.4	10.19	503	
124	5.758	1.8	10.36	512	
133a	4.846	2.1	10.17	502	
21	4.367	2.4	10.48	517	
isobutane	2.452	4.2	10.29	508	
114	7.319	1.4	10.24	506	
Component	**Liquid Density, mg/µl**	**Volume Added, µl**	**mg Added (1)**	**ppm Added (2)**	**Total ppm Present (3)**
11	1.537	6.6	10.14	501	
123	1.526	6.7	10.22	505	
113	1.619	6.3	10.19	503	

(1) If necessary, correct the mg added for the purity of the calibration component previously established.

(2) Values shown are for illustration; exact values are determined at Part 33, Section 9.3, steps c and d.

(3) Column to be filled in (Part 33, Section 9.3, step g) after determining ppm present in the stock components (see Note 1 in Section 10).

Part 48

R-507 Purity GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Part 33, *General Procedure for the Determination of Purity of New and Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography* (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-507.

Section 2. Limitations and Interferences

This method is calibrated only for those impurities commonly present in R-507A. This method will not detect any impurity that may elute within the comparatively large HFC-125 and HFC-143a peak matrices. For example, HFC-134a elutes on the far shoulder of the large HFC-125 peak and is therefore difficult to detect at low concentrations. Because of this poor resolution, the precision of the HFC-134a impurity is comparitively large. The contaminants HFC-23 and HFC-32 also elute together, but these may be separated if needed by adding a 0.30 m column section of Porapak-T to the end of the primary column.

Section 3. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1A and B. The data was obtained by analyzing an R-507 calibration mixture 7 times during one day by one operator.

Section 4. Tables

The following tables are to be used in conjunction with Part 33 of the *General Procedure for the Determination of Purity of Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography*.

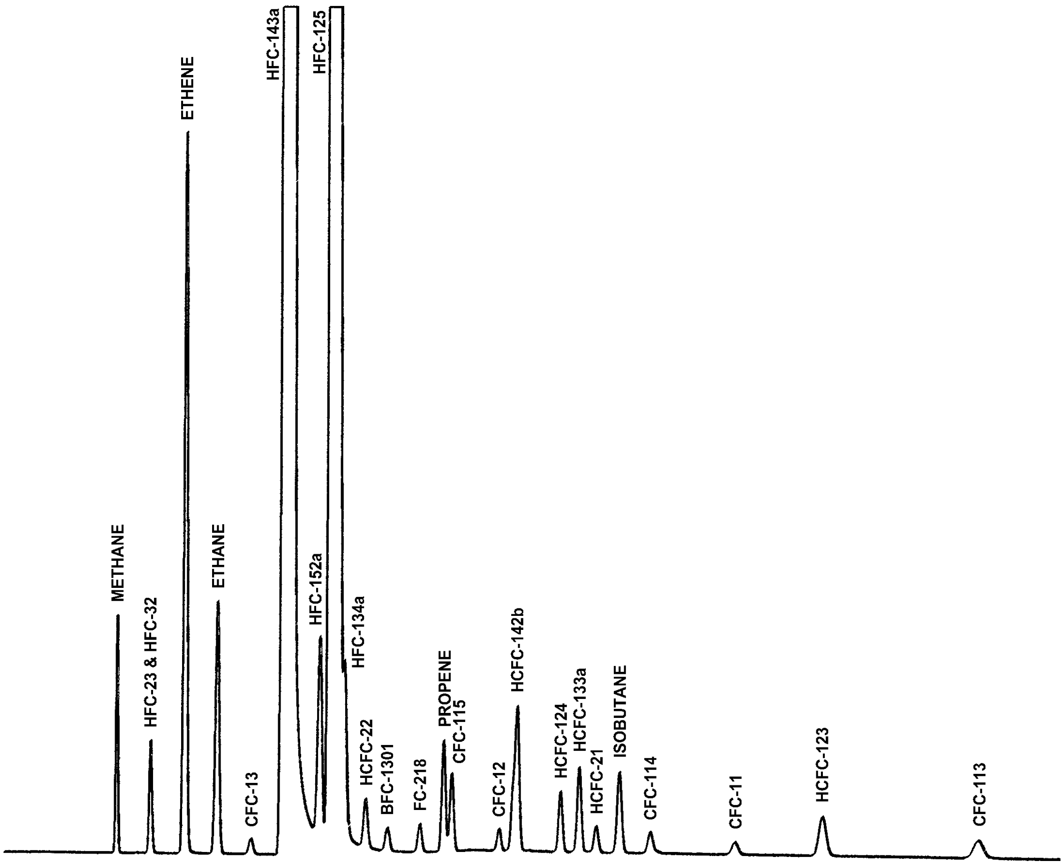


Figure 1. Gas Chromatogram of R-507

Table 1A. Contaminant Statistical Parameters			
Component	**Detection Limit, ppm**	**Range Investigated, ppm**	**Precision at 95% Confidence Limit, ppm**
methane	1	100	0.5
23	17	450	12
32	8	450	3.3
ethene	1	450	1.8
ethane	1	200	0.8
13	23	450	5.4
134a	100	450	5.3
22	15	450	29
1301	16	450	4.3
218	11	450	0.9
propene	1	100	0.5
115	17	3400	110
12	17	450	4.3
142b	3	450	6.6
124	7	450	1.9
133a	4	450	3.3
21	11	450	13
isobutane	1	100	1.1
114	10	450	3.9
11	12	450	9.6
123	4	450	11
113	5	450	14

Table 1B. Component Statistical Parameters			
Component	**Detection Limit, ppm**	**Range Investigated, %**	**Precision at 95% Confidence Limit**
143a	4	50 %	0.05 %
125	5	50 %	0.07 %

Table 1C. Blend Component Balance Preparation		
Component	**Final Pressure After Component Addition (kPa)**	**Weight of Refrigerant Added (g)**
R-143a	276	10.00
R-125	453	10.00

Table 2. Primary Calibration Standard Impurities

Component	**Vapor Density, mg/ml**	**Volume Added, ml**	**mg Added (1)**	**ppm Added (2)**	**Total ppm Present (3)**
methane	0.656	4.0	2.62	130	
23	2.895	3.5	10.13	500	
32	2.162	4.7	10.16	502	
ethene	1.147	4.0	4.58	226	
ethane	1.230	4.0	4.92	243	
13	4.331	2.4	10.39	513	
152a	2.772	3.7	10.25	506	
134a	4.279	2.4	10.26	507	
22	3.606	2.9	10.45	516	
1301	6.205	1.7	10.54	521	
218	7.923	1.3	10.29	508	
propene	1.756	5.8	10.18	503	
115	6.486	1.6	10.37	512	
12	5.077	2.0	10.15	501	
142b	4.248	2.4	10.19	503	
124	5.758	1.8	10.36	512	
133a	4.846	2.1	10.17	502	
21	4.367	2.4	10.48	517	
isobutane	2.452	4.2	10.29	508	
114	7.319	1.4	10.24	506	
Component	**Liquid Density, mg/µl**	**Volume Added, µl**	**mg Added (1)**	**ppm Added (2)**	**Total ppm Present (3)**
11	1.537	6.6	10.14	501	
123	1.526	6.7	10.22	505	
113	1.619	6.3	10.19	503	

(1) If necessary, correct the mg added for the purity of the calibration component previously established.

(2) Values shown are for illustration; exact values are determined at Part 33, Section 9.3, steps c and d.

(3) Column to be filled in (Part 33, Section 9.3, step g) after determining ppm present in the stock components (see Note 1 in Section 10).

Part 49

R-508 Purity GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Part 33, *General Procedure for the Determination of Purity of New and Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography* (hereafter referred to as General Procedure). This GC Method Data Sheet is for use with R-508.

Section 2. Limitations and Interferences

This method is calibrated only for those impurities commonly present in R-508 blends. This method will not detect any impurity that may elute within the comparatively large HFC-23 and FC-116 peak matrices. For example, HFC-32 cannot be detected using this method as it elutes within the large HFC-23 component peak matrix. In addition, ethane cannot be detected as it elutes within the FC-116 component peak.

Section 3. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1A and B. The data was obtained by analyzing an R-508 calibration mixture 7 times during one day by one operator.

Section 4. Tables

The following tables are to be used in conjunction with Part 33 of the *General Procedure for the Determination of Purity of Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography*.

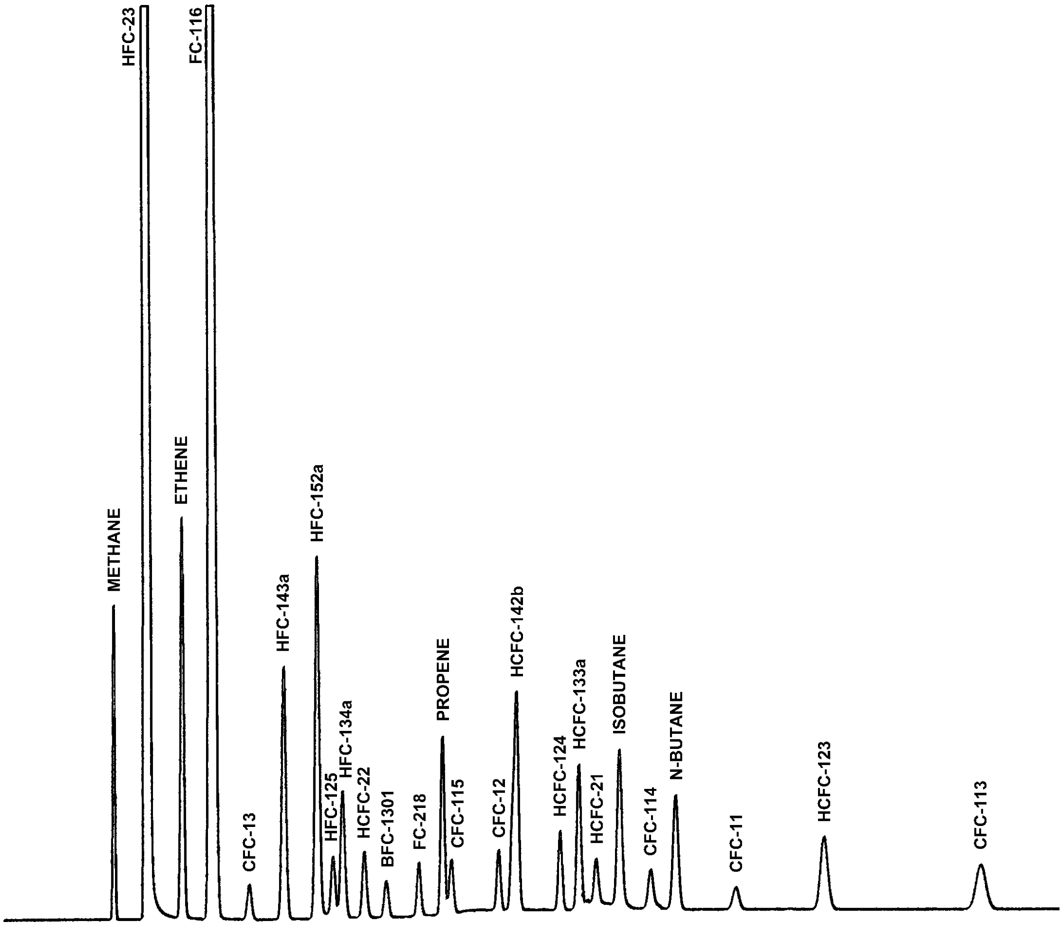


Figure 1. Gas Chromatogram of R-508

Table 1A. Contaminant Statistical Parameters			
Component	**Detection Limit, ppm**	**Range Investigated, ppm**	**Precision at 95% Confidence Limit, ppm**
methane	1	100	0.4
ethene	1	200	1.0
13	14	450	2.0
143a	2	450	3.0
152a	2	450	5.3
125	8	450	41
134a	4	450	2.0
22	12	450	31
1301	14	450	3.6
218	11	450	1.5
propene	1	100	6.0
115	13	450	21
12	16	450	2.4
142b	3	450	4.9
124	7	450	3.9
133a	3	450	5.2
21	8	450	5.4
isobutane	1	100	2.6
114	9	450	4.8
n-butane	1	100	2.4
11	15	450	12
123	4	450	11
113	5	450	7.8

Table 1B. Component Statistical Parameters			
Component	**Detection Limit, ppm**	**Range Investigated, %**	**Precision at 95% Confidence Limit**
23	8	39 %	0.12 %
116	98	61 %	1.7 %

Table 1C. Blend Component Balance Preparation		
Component	**Final Pressure After Component Addition (kPa)**	**Weight of Refrigerant Added (g)**
R-23	266	7.80
R-116	465	12.20

Table 2. Primary Calibration Standard Impurities

Component	**Vapor Density, mg/ml**	**Volume Added, ml**	**mg Added (1)**	**ppm Added (2)**	**Total ppm Present (3)**
methane	0.656	4.0	2.62	130	
ethene	1.147	4.0	4.58	226	
13	4.331	2.4	10.39	513	
143a	3.511	2.9	10.18	503	
152a	2.772	3.7	10.25	506	
125	5.004	2.1	10.50	519	
134a	4.279	2.4	10.26	507	
22	3.606	2.9	10.45	516	
1301	6.205	1.7	10.54	521	
218	7.923	1.3	10.29	508	
propene	1.756	5.8	10.18	503	
115	6.486	1.6	10.37	512	
12	5.077	2.0	10.15	501	
142b	4.248	2.4	10.19	503	
124	5.758	1.8	10.36	512	
133a	4.846	2.1	10.17	502	
21	4.367	2.4	10.48	517	
isobutane	2.452	4.2	10.29	508	
114	7.319	1.4	10.24	506	
n-butane	2.463	4.2	10.34	511	
Component	**Liquid Density, mg/µl**	**Volume Added, µl**	**mg Added (1)**	**ppm Added (2)**	**Total ppm Present (3)**
11	1.537	6.6	10.14	501	
123	1.526	6.7	10.22	505	
113	1.619	6.3	10.19	503	

(1) If necessary, correct the mg added for the purity of the calibration component previously established.

(2) Values shown are for illustration; exact values are determined at Part 33, Section 9.3, steps c and d.

(3) Column to be filled in (Part 33, Section 9.3, step g) after determining ppm present in the stock components (see Note 1 in Section 10).

Part 50

R-509 Purity GC Method Data Sheet

Section 1. Scope

This GC Method Data Sheet is for use in conjunction with Part 33, *General Procedure for the Determination of Purity of New and Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography*. This GC Method Data Sheet is for use with R-509.

Section 2. Limitations and Interferences

This method is calibrated only for those impurities commonly present in R-509A. This method will not detect any impurity that may elute within the comparatively large HCFC-22 and FC-218 peak matrices. The contaminants HFC-23 and HFC-32 also elute together, but these may be separated if needed by adding a 0.30 m column section of Porapak-T to the end of the primary column.

Section 3. Sensitivity, Precision, and Accuracy

Statistical parameters for each impurity are listed in Table 1A and B. The data was obtained by analyzing an R-509 calibration mixture 7 times during one day by one operator.

Section 4. Tables

The following tables are to be used in conjunction with Part 33 of the *General Procedure for the Determination of Purity of Reclaimed Refrigerant Blends and Azeotropes by Gas Chromatography.*

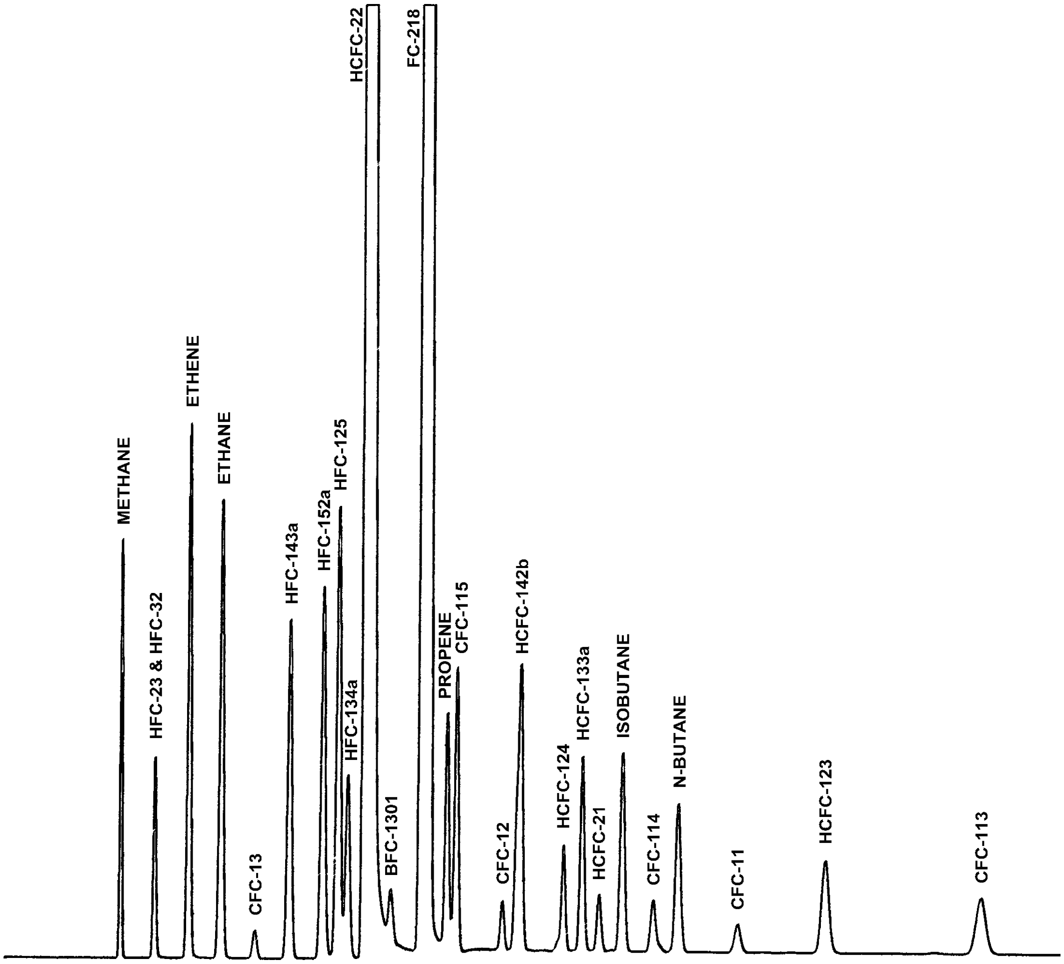


Figure 1. Gas Chromatogram of R-509

Table 1A. Contaminant Statistical Parameters			
Component	**Detection Limit, ppm**	**Range Investigated, ppm**	**Precision at 95% Confidence Limit, ppm**
methane	1	100	0.9
23	8	450	8.3
32	3	450	4.8
ethene	1	200	1.4
ethane	1	200	1.9
13	9	450	2.7
143a	1	450	4.8
152a	1	450	5.4
125	4	450	5.2
134a	2	450	3.6
1301	7	450	4.5
propene	1	100	0.5
115	6	450	12
12	8	450	1.3
142b	1	450	83
124	3	450	7.3
133a	2	450	7.6
21	4	450	6.0
isobutane	1	100	1.2
114	4	450	4.9
n-butane	1	100	1.2
11	6	450	17
123	2	450	5.2
113	2	450	13

Table 1B. Component Statistical Parameters			
Component	**Detection Limit, ppm**	**Range Investigated, %**	**Precision at 95% Confidence Limit**
22	5	44 %	0.01 %
218	5	56 %	0.12 %

Table 1C. Blend Component Balance Preparation		
Component	**Final Pressure After Component Addition (kPa)**	**Weight of Refrigerant Added (g)**
FC-218	140	11.20
R-22	363	8.80

Table 2. Primary Calibration Standard Impurities

Component	Vapor Density, mg/ml	Volume Added, ml	mg Added (1)	ppm Added (2)	Total ppm Present (3)
methane	0.656	4.0	2.62	130	
23	2.895	3.5	10.13	500	
32	2.162	4.7	10.16	502	
ethene	1.147	4.0	4.58	226	
ethane	1.230	4.0	4.92	243	
13	4.331	2.4	10.39	513	
143a	3.511	2.9	10.18	503	
152a	2.772	3.7	10.25	506	
125	5.004	2.1	10.50	519	
134a	4.279	2.4	10.26	507	
1301	6.205	1.7	10.54	521	
propene	1.756	5.8	10.18	503	
115	6.486	1.6	10.37	512	
12	5.077	2.0	10.15	501	
142b	4.248	2.4	10.19	503	
124	5.758	1.8	10.36	512	
133a	4.846	2.1	10.17	502	
21	4.367	2.4	10.48	517	
isobutane	2.452	4.2	10.29	508	
114	7.319	1.4	10.24	506	
n-butane	2.463	4.2	10.34	511	
Component	**Liquid Density, mg/µl**	**Volume Added, µl**	**mg Added (1)**	**ppm Added (2)**	**Total ppm Present (3)**
11	1.537	6.6	10.14	501	
123	1.526	6.7	10.22	505	
113	1.619	6.3	10.19	503	

(1) If necessary, correct the mg added for the purity of the calibration component previously established.

(2) Values shown are for illustration; exact values are determined at Part 33, Section 9.3, steps c and d.

(3) Column to be filled in (Part 33, Section 9.3, step g) after determining ppm present in the stock components (see Note 1 in Section 10).