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# CHEMICAL AND THERMAL STABILITY OF REFRIGERANT-LUBRICANT MIXTURES WITH METALS

Final Report

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## ABSTRACT

This report presents the results of a sealed tube stability study on twenty-one refrigerant-lubricant mixtures selected from the following groupings:

- HFCs R-32, R-125, R-134, R-134a, R-143a, and R-152a with one or more lubricants selected from among three pentaerythritol esters and three polyalkylene glycols. All lubricants were carefully pre-dried to 25 ppm or less moisture content.
- HCFCs R-22, R-123, R-124, and R-142b, as well as CFC R-11, with one or more lubricants selected from among two mineral oils and one alkylbenzene fluid.

Each test mixture was aged at three temperature levels.

Primary results obtained were:

1. HFCs tested, as well as R-22, are very stable and do not undergo chemical or thermal decompositions after 14 days at temperatures up to 200°C (392°F).
2. HCFCs R-124 and R-142b have stability properties intermediate between above HFCs and the currently widely used CFC R-12.
3. HCFC R-123 while more reactive than any of the above, still offers a stability improvement by a factor of ten over R-11, the CFC refrigerant it is to replace in low pressure chiller applications.
4. All pentaerythritol ester and all polyalkylene glycol fluids showed some changes of chemical structure after exposure to 200°C (392°F), and in some cases after exposure to the lower temperatures of 150°C (300°F) and 175°C (347°F).

This report supersedes previously published quarterly reports DE/CE/23810-2A and DE/CE/23810-3A.

# SCOPE

The scope of ARTI MCLR Project 650-50200 encompasses the determination of the chemical and thermal stability of twenty-one refrigerant-lubricant combinations. The test methodology chosen was the sealed tube stability test. Refrigerants and lubricants are heated at elevated temperatures in the presence of a steel catalyst. Test results are based on chemical and physical measurements designed to assess the changes that occur to the test materials during thermal aging.

Refrigerants tested as part of the project consisted of six HFCs, four HCFCs, and one CFC; the latter was only included to establish base line information. Lubricants tested were three polyglycols, three polyolesters, two mineral oils and one alkylbenzene fluid.

The goal of the project was an assessment of the general stability properties of generically selected refrigerant-lubricant mixtures, rather than endorsement (or opposite thereof) of any of the specific materials tested. Aging tests were conducted over temperatures ranging from 105°C (220°F) to 200°C (392°F) depending on the specific materials combinations tested. As it will be apparent from the results, not all materials tested perform equally well at the higher temperatures. On the other hand, not all refrigerant systems require materials capable of 200°C performance. Chemical and thermal stability of refrigerant-oil mixtures is but one of a number of properties necessary for reliable performance in refrigeration and air-conditioning systems.

# SIGNIFICANT RESULTS

## INTRODUCTION

A total of twenty-one refrigerant-mixtures were tested during this study. They were:

- R-22/Mineral Oil-ISO VG32 (150 SSU viscosity)
- R-32/Pentaerythritol Ester (mixed acid I)
- R-32/Polypropylene Glycol (butyl monoether)
- R-123/Mineral Oil-ISO VG32 (150 SSU viscosity)
- R-124/Alkylbenzene - ISO VG32 (150 SSU viscosity)
- R-125/Polypropylene Glycol (butyl monoether)
- R-125/Modified Polyglycol
- R-125/Pentaerythritol Ester (mixed acid I)
- R-134a/Polypropylene Glycol (butyl monoether)
- R-134a/Polypropylene Glycol Diol
- R-134a/Modified Polyglycol
- R-134a/Pentaerythritol Ester (mixed acid II)
- R-134a/Pentaerythritol Ester (mixed acid I)
- R-134a/Pentaerythritol Ester (100 cSt)
- R-142b/Alkylbenzene - ISO VG32 (150 SSU viscosity)
- R-143a/Pentaerythritol Ester (mixed acid I)
- R-152a/Alkylbenzene - ISO VG32 (150 SSU viscosity)
- R-134/Pentaerythritol Ester (mixed acid I)
- R-11/Mineral Oil-ISO VG32 (150 SSU viscosity)
- R-11/White Naphthenic Mineral Oil (ISO VG45)
- R-123/White Naphthenic Mineral Oil (ISO VG45)

Test results are presented in the form of individual data tables for each refrigerant-lubricant mixture. These tables are consolidated in Appendix A (Tables A-1 through A-21). Following each data table are the gas chromatograms for the test samples covered in each table (Figures A-1 to A-42).

Listed in the data tables is the following information:

- contents of the sealed tubes reported in each table
- aging conditions
- visual observations on sealed tube contents after aging, including change of oil (liquid phase) color expressed in units of the ASTM Standard D1500 color scale, condition of the steel catalyst, formation of insoluble materials, etc.
- chemical analysis results on aged tubes, including gas chromatography (GC) of the vapor phase tube contents, chloride

or fluoride ion content, and oil analysis results for those mixtures containing polyglycol or polyol ester lubricants: total acid number (TAN), infrared (IR) spectroscopy, and size exclusion chromatography (SEC).

Under "Chemical Analysis" the column following chloride/ fluoride ion results is labeled "% Refrigerant Reacted". This value was calculated from the measured halide ion content by making the assumption that each halide ion generated equals the destruction of one refrigerant molecule. Based on past experience, this is a reasonable assumption at least at low and intermediate levels of reactivity. It is not valid for very reactive systems where more than one halide atom has been removed from refrigerant molecules.

Figures A-1 to A-42 are copies of the chromatograms obtained on the vapor content of aged sealed tubes. There are four chromatograms for each refrigerant-lubricant mixture: unused refrigerant and vapor samples aged at the three test temperatures.

Oil analysis of aged synthetic lubricants consisted of IR spectroscopy for the polyol ester samples, and SEC for the polyalkylene glycol samples. Copies of IR spectra and SEC chromatograms can be found in Appendix B (Figures B-1 to B-22).

Aged sealed tubes and their individual steel catalysts (both aged and unaged) were photographed for all test sets. Copies of photographs from those test sets that exhibited significant visual changes after aging are shown in Appendix D (Figures D-1 to D-8). Appendix D is included only with the permanently bound copies of this report.

All test materials used in this study are referred to by generic designations only. Cross references from generic to commercial designations for all lubricants tested are shown in Appendix C. Also included in Appendix C are the results of purity measurements obtained on all of the refrigerants and lubricants, as received by us. This includes GC analysis of refrigerants, and water, copper and iron content, as well as the total acid number of lubricants.

## DISCUSSION OF RESULTS

Discussion of results is arranged in the order HFC/pentaerythritol (PE) ester mixtures, HFC/polyalkylene glycol (PAG) mixtures, and HCFCs.

### HFC/PE Ester Mixtures

Table 1, page 6, is a summary of results taken from the individual data tables for the eight mixtures listed. The R-152a/alkybenzene mixture has been included in this ester result table to avoid an extra classification for just one HFC/alkylbenzene mixture.

The data listed in Table 1 do not show any evidence of significant refrigerant decomposition. This holds for all test mixtures and at all aging conditions. Fluoride contents are very low and very close to the "zero" value for the test method. Values of "% Refrigerant Decomposition" for the titration blanks performed during the analyses are in the range of 0.001 to 0.002%. The actual fluoride concentrations measured (after subtraction of the titration blank) are of the same low order of magnitude.

GC analyses (Appendix A) for all Table 1 mixtures lend additional support to the above findings. Again, there is no evidence of any measurable refrigerant decomposition for any of the test mixtures.

The only changes of any significance observed with the HFC-ester mixtures are on the aged lubricants. Acid numbers of aged oils increased after all of the 200°C (392°F) tests. The increases range from 0.49 to 1.69 units (ASTM Method D664 "Test of Neutralization Number by Potentiometric Titration"). Some of the mixtures also had acid number increases at 150°C (300°F) and at 175°C (347°F). The consistent acid number increases at the highest temperature are believed to be indicative of the thermal stability limits of the fluids tested.

Reasons for the randomly occurring changes at lower temperatures are not quite apparent, except in the case of the R-134a/100 cSt ester mixture. Here, in addition to the acid number changes, IR spectral changes occurred at all test temperatures. The observed IR changes are typical of C=O absorption (formation of free acids) and C-O absorption (free alcohol functions). In addition, GC analysis of 200°C tubes (Figure A-28) detected the presence of $CO_2$ which is a product of the decarboxylation of ester linkages.

TABLE 1: STABILITY RESULTS HFC/PE ESTER LUBRICANTS

| TEST SAMPLE | DATA TABLE | AGING TEMP. (°C) | % REFRIGERANT REACTED (calc. from F ion data) | LUBRICANT ACID NUMBER (mg KOH/g) |
|---|---|---|---|---|
| R-32/PE | A-2 | 150 (300°F) | 0.002 | 0.00 |
| (mixed acid) | | 175 (347°F) | 0.001 | 0.00 |
| | | 200 (392°F) | 0.001 | 1.67 |
| R-125/PE | A-8 | 150 | 0.005 | 0.00 |
| (mixed acid I) | | 175 | 0.005 | 0.48 |
| | | 200 | 0.005 | 1.09 |
| R-134a/PE | A-12 | 150 | 0.000 | 0.00 |
| (mixed acid 11) | | 175 | 0.000 | 0.00 |
| | | 200 | 0.000 | 0.67 |
| R-134a/PE | A-13 | 150 | 0.001 | 0.00 |
| (mixed acid I) | | 175 | 0.001 | 0.00 |
| | | 200 | 0.001 | 0.74 |
| R-134a/PE | A-14 | 150 | 0.001 | 0.94 |
| (100 cSt) | | 175 | 0.001 | 0.63 |
| | | 200 | 0.004 | 0.99 |

Note: Aged lubricant from this mixture showed significant IR spectrum changes after aging at all temperatures.

| | | | | |
|---|---|---|---|---|
| R-143a/PE | A-16 | 150 | 0.001 | 0.51 |
| (mixed acid I) | | 175 | 0.002 | 1.07 |
| | | 200 | 0.001 | 0.99 |
| R-134/PE | A-18 | 150 | 0.001 | 0.59 |
| (mixed acid I) | | 175 | 0.000 | 0.83 |
| | | 200 | 0.000 | 1.69 |
| R-152a/ALKYL- | A-17 | 150 | 0.000 | Not applicable |
| -BENZENE | | 175 | 0.001 | Not applicable |
| | | 200 | 0.000 | Not applicable |

The only other IR spectral changes observed on polyol esters were minor changes after aging at 200°C (392°F) with the R-125/PE (mixed acid I) mixture.

The overall conclusions from the HFC/PE ester tests are as follows:

1. All HFCs tested are stable at the test conditions used.
2. The high viscosity ester (100 cSt), tested with R-134a, exhibited signs of molecular changes at all three test temperatures. It is unknown at this point whether these observed changes will impact on full scale equipment performance.
3. The other two PE esters tested, mixed acid I and mixed acid II, showed little, if any, changes except for consistent increases in acid numbers after aging at 200°C (392°F). In other words, these two esters appear to be quite stable to at least 175°C (347°F).
4. All tests were performed with esters having very low moisture levels in the 25 ppm range. Results reported here may or may not be representative of the stability of PE esters in the "real world", where much higher water contents are expected to be the norm.
5. No observable reactions occurred with the R-152a/ alkylbenzene mixture.

## HFC/PAG Mixtures

Table 2, page 8, summarizes the test results from mixtures containing PAG lubricants. Again, there is no evidence of HFC decomposition by either GC or fluoride ion analysis under any of the three test conditions.

However, size exclusion chromatography (SEC) suggests changes occurred to the structure of the PAG molecules. This was observed to varying degrees with all three PAG lubricants tested, and at all test temperatures. As was noted under the polyol esters, the significance of these observed changes on the actual performance of the PAG lubricants is not clear at the present time. The absence of HFC decomposition products suggests that PAG molecular changes do not give rise to refrigerant instability. The most likely effects, if any, of the PAG changes would be viscosity decreases, which impact lubrication performance, and/or the formation of high molecular weight solids and sludges, which can cause systems flow restrictions.

TABLE 2: STABILITY RESULTS HFC-PAG LUBRICANTS

| TEST SAMPLE | DATA TABLE | AGING TEMP. (°C) | % REFRIGERANT REACTED (calc. from F data) | Size Exclusion Chromatography |
|---|---|---|---|---|
| R-32/PPG BUTYL MONOETHER | A-3 | 150 (300°F) | 0.000 | Minor changes in PAG structure (all tests) |
| | | 175 (347°F) | 0.002 | |
| | | 200 (392°F) | 0.004 | |
| R-125/PPG BUTYL MONOETHER | A-6 | 150 | 0.001 | Changes in PAG structure (all tests) |
| | | 175 | 0.002 | |
| | | 200 | 0.002 | |
| R-125/MODIFIED POLYGLYCOL | A-7 | 150 | 0.007 | Changes in PAG structure (all tests) |
| | | 175 | 0.004 | |
| | | 200 | 0.005 | |
| R-134a/PPG BUTYL MONOETHER | A-9 | 150 | 0.001 | Changes in PAG structure (all tests) |
| | | 175 | 0.001 | |
| | | 200 | 0.001 | |
| R-134a/PPG DIOL | A-10 | 150 | 0.000 | Significant changes in PAG structure (all tests) |
| | | 175 | 0.001 | |
| | | 200 | 0.000 | |
| R-134a/MODIFIED POLYGLYCOL | A-11 | 150 | 0.001 | Significant changes in PAG structure (all tests) |
| | | 175 | 0.001 | |
| | | 200 | 0.001 | |

The test results on HFC-PAG mixtures lead to the following conclusions:

1. All HFCs tested are stable at the test conditions used.
2. Based on SEC analysis all PAGs tested exhibited varying degrees of molecular change. Again, the impact, if any, of these observed changes on full scale systems performance is unknown.
3. Also unknown at this point are the effects of moisture contents higher than the low 25 ppm level present in the test work reported here.

HCFC/Hydrocarbon Lubricant Mixtures

Table 3, page 10, summarizes the test results from the HCFC containing mixtures. It is apparent that, with the possible exception of R-22, the HCFCs tested are more reactive under the test conditions used than any of the previously discussed HFCs. Both chloride ion and vapor phase GC analyses support this contention in all cases. However, in comparison to CFCs, such as R-12, both R-124 and R-142b exhibit improved chemical and thermal stability properties. Hence, the use of these HCFCs in full scale systems should not present chemical liability questions beyond those that the industry has accepted historically with R-12 and other CFCs.

The fourth HCFC tested, R-123, is significantly more reactive than are any of the "new" refrigerants included in this project, especially at temperatures above 150°C. It must be noted, however, that R-123 is a low pressure refrigerant and will be used as a replacement for R-11 in low pressure systems. There, it will never be exposed to the higher range temperatures of the sealed tube tests.

The GC results obtained on the vapor phase contents of aged R-123 sealed tubes show the expected decomposition path for this refrigerant, namely

$$\underset{\text{R-123}}{CF_3CHCl_2} \longrightarrow \underset{\text{R-133a}}{CF_3CH_2Cl} \longrightarrow \underset{\text{R-143a}}{CF_3CH_3}$$

Unfortunately, pure samples of R-133a were not available for a quantitative calibration of GC results. All R-133a values listed in the tables are uncorrected readouts and are approximate values only.

TABLE 3: STABILITY RESULTS HCFC-HYDROCARBON LUBRICANTS

| TEST SAMPLE | DATA TABLE | AGING TEMP. (°C) | % REFRIGERANT REACTED (calc. from C1 data) | GC RESULTS |
|---|---|---|---|---|
| R-22/Mineral Oil (ISO VG32-150 SSU) | A-1 | 150 (300°F) | 0.001 | No changes |
| | | 175 (347°F) | 0.003 | No changes |
| | | 200 (392°F) | 0.004 | No changes |
| R-123/Mineral Oil (ISO VG32-150 SSU) | A-4 | 105 (220°F) | 0.045/0.032 | No changes |
| | | 150 | 1.56/2.71 | R-133a: 1.8/3.4% |
| | | 160 (320°F) | 26.0 | R-133a: 14.0%<br>R-143a: 0.45% |
| | | 175 | 40.5 | R-133a: 44.5%<br>R-143a: 2.0% |
| R-124/Alkylbenzene (ISO VG32) | A-5 | 150 | 0.008 | No changes |
| | | 175 | 0.014 | No changes |
| | | 200 | 0.052 | No changes |
| R-142b/Alkylbenzene (ISO VG32) | A-15 | 150 | 0.011 | No changes |
| | | 175 | 0.015 | No changes |
| | | 200 | 0.169 | No measurable changes |

For Comparison with Past Practices:

| | | | | |
|---|---|---|---|---|
| R-12/Mineral Oil | | 175 | 0.2 (typical value)(1) | R-22 |
| R-12/Alkylbenzene | | 175 | 0.1 (typical value)(1) | R-22 |

(1) "Sealed Tube Stability Test Results: Alternative Refrigerants" Carrier Corporation, Syracuse, NY, September 1989

Because of the relatively limited high temperature capability of R-123, a direct comparison was established for this refrigerant with the R-11 low pressure CFC refrigerant. Table 4, page 12, summarizes the results obtained from the two refrigerants after aging at 105, 150, and 160°C (220, 300, and 320°F) .

The data show that R-123 is approximately ten times more stable than is R-11. Therefore, the use of R-123 as an R-11 replacement is expected to cause fewer, if any, chemical and/or thermal stability problems than does R-11 now in equivalent applications. The reactivity of both refrigerants depends on oil quality, especially at the lower sealed tube temperatures. At 150°C (300°F) and above most, if not all, R-11 was destroyed after 14 days. In addition, all chlorine was removed from some of the R-11 molecules resulting in $CO_2$ formation (GC analysis). R-123 was not subject to such destructive action under the same test conditions. As a rough comparison, R-123 exhibits chemical activity at 105°C (220°F) similar to that of R-12 at 175°C (347°F) .

The HCFC tests lead to the following conclusions:

1. R-22 has excellent stability, at least up to 200°C (392°F), similar to the HFCs tested under this program.

2. R-124 and R-142b are slightly more reactive than the HFCs, but are less reactive than R-12 and similar CFCs.

3. While R-123 was the most reactive new refrigerant tested during this program, it is still significantly less reactive than is R-11 - by a factor of approximately ten. Therefore, the replacement of R-11 by R-123 in low pressure refrigeration equipment will cause fewer, if any, chemical problems than may exist now.

## CONCLUSIONS

The following summary conclusions can be drawn from the results reported here:

1. All HFCs tested, as well as R-22, are very stable materials and do not undergo significant chemical reactions or thermal decomposition at temperatures up to 200°C (392°F).

TABLE 4: STABILITY COMPARISON R-123 VS. R-11

| TEST SAMPLE | DATA TABLE | AGING TEMP. (°C) | %,REFRIGERANT REACTED (calc from C1 data) | GC RESULTS |
|---|---|---|---|---|
| R-123/Mineral Oil (ISO VG32) | A-4 | 105 (220°F) | 0.045/0.032 | No changes |
| | | 150 (300°F) | 1.56/2.71 | R-133a: 1.8/3.4% |
| | | 160 (320°F) | 26.0 | R-133a: 14.0%, R-143a: 0.45% |
| R-11/Mineral Oil (ISO VG32) | A-19 | 105 | 0.46 | No measurable change |
| | | 150 | 72.0 | R-21: 72%, R-31: <0.5% |
| | | 160 | 87.0 | R-11: 0%, R-21: 47.8% R-31: 17.2% $CO_2$: 31.4% |
| R-11/White Naphthenic Oil (ISO VG46) | A-20 | 105 | 0.014 | No changes |
| | | 150 | 45.2 | R-21: 17.8%, R-31: 1.37% $CO_2$ |
| | | 160 | 97.0 | Trace R-11, R-21: 75.0%, R-31: 10.4%, $CO_2$ :13.7% |
| R-123/White Naphthenic Oil (ISO VG46) | A-21 | 105 | 0.002 | No changes |
| | | 150 | 5.84 | R-133a: 3.9% R-143a: 0.08% |
| | | 160 | 8.85 | R-133a: 10.9% R-143a: 0.38% $CO_2$ |

2. Two HCFCs tested, R-124 and R-142b, were found to be intermediate in reactivity between the above HFCs and CFCs, such as R-12.

3. While R-123 is significantly more reactive than is any of the "new" refrigerants tested here, it still offers a stability improvement by a factor of ten over R-11, the CFC refrigerant it is intended to replace in low pressure chiller applications.

4. The two low viscosity (ISO VG22 and VG32) PE esters included in the program exhibited some increases in acid number, especially after aging at 200°C (392°F). The high viscosity ester (ISO 100) tested exhibited additional evidence of molecular changes during aging. Formation of $CO_2$ at 200°C (392°F) indicates decarboxylation of the ester at that temperature.

5. All polyalkylene glycol lubricants exhibited some signs of molecular change after aging. The significance of these changes has not been established.

# TEST PROCEDURES

## SEALED TUBE TESTS

All sealed tube tests were conducted in heavy walled glass tubes, 6.35 mm I.D. and 20 cm long (¼" x 8") with one end sealed. After thorough cleaning with toluene and drying at 150°C (300°F) the tubes were necked 5 cm (2") from the open end. This gives finished sealed tubes with an internal free volume of 7.5-8.0 ml (about 0.5 $in^3$).

Metal coupons were cut from valve steel (Sandvik Steel Company), 6.3 mm (¼") wide and 32 mm (1.25") long. After cleaning with hexane the coupons were stored submerged in this solvent. One coupon was added to each tube immediately prior to necking. After necking of the tubes 1.0 ml of lubricant was added using a calibrated syringe. Refrigerant equal to the lubricant weight was next condensed from a gas handling manifold into the liquid nitrogen cooled tubes. After sealing and flame annealing of the seals the finished tubes were inspected for any visible faults. Acceptable tubes were placed into individual slots in large aluminum "blocks" of 12.7 cm (0.5") OD.

Three sealed tubes were prepared for each test set, that is, for every refrigerant-lubricant combination at each aging temperature. The aluminum blocks were heated in large convection ovens for 14 days at their respective temperatures. Oven cavities as well as internal temperatures of the blocks were monitored throughout the aging tests.

## EVALUATION OF AGING TESTS

Aged sealed tubes were evaluated by combinations of the following methods:

### Visual Inspection

After completion of aging sealed tubes were inspected visually. Recorded were:

- changes in color of the liquid phase scaled per ASTM Standard D1500
- appearance of insoluble reaction products, if any
- appearance of the metal coupons.

In addition, a photographic record was prepared of each test set consisting (from left to right in photograph) of a clean unused steel coupon, the aged coupon, and the sealed tube. The aged coupons for the photographs came from the GC analysis tubes. Photographs were taken as quickly as possible after opening of the tubes to minimize any effects of air oxidation on the appearance of metal surfaces, a precaution especially important for coupons from HCFC tests. Depending on the amount of chloride ion present, many of the HCFC exposed coupons corroded badly upon exposure to air.

## Gas Chromatographic (GC) Analysis

All GC analyses were performed on a Gow-Mac Series 550P Thermal Conductivity Detector instrument using a Poropak "Q" column of 2.44 m (8′) length. Gas samples were injected through a six-way Valco gas sample valve. Aged sealed tubes to be analyzed were connected to the gas handling manifold with a "tube breaker" assembly. After freezing of the tube contents in liquid nitrogen, the tube tips (scratched with a sharp file) were broken off. The tubes were then warmed slowly and all volatile tube contents were expanded into the manifold, which has a free volume of 1058 ml (64.4 $in^3$). A sample of gas from this volume was injected through the sample valve into the GC column. The vapor pressure of the gas sample normally was in the 100 to 200 torr range, depending on the vapor pressure of the test refrigerant.

The column was operated under the following conditions:

Column and injector temperatures: 120°C (R-32 which eluted at 0.55-0.60 min. was also analyzed at 100°C [0.7 min peak] for better resolution of peak(s) below 0.5 min.);
Detector temperature: 200°C; Detector current: 150 mA;
Carrier gas: Helium; Gas flow rate: 27 ml/min;
Sample size: 500 microliter

GC results, i.e. peak retention times, peak areas and peak heights, and area percent for each measurable peak in a chromatogram, were obtained on a Hewlett-Packer Model 3390A Integrator.

GC elution peaks of known identities are reported by their chemical designations, while those unknown to us are referred to by their peak retention times.

## Chloride and Fluoride Ion Determinations

Chloride and/or fluoride ion contents of sealed tubes were determined with Orion "Ion Selective Electrodes" coupled to a Fisher "Accumet pH Ion Analyzer".

Theory predicts and past experience has shown that the chloride atoms of CFC and HCFC molecules are significantly more reactive than are their fluoride atoms. Therefore, HCFC containing mixtures were analyzed only for chloride ion content in the belief that this is a more sensitive indicator of chemical reactivity. All HFC containing mixtures were of course analyzed for fluoride ion. These determinations were performed in duplicate for every test set. All tube contents, i.e. refrigerant, lubricant, metal coupon, and all glass tube fragments were included in the halide ion determinations.

Results are reported in micrograms of halide ion. From this value we computed "% Refrigerant Reacted" based on the assumption that each halide ion found is the product of the removal of one halide atom from a refrigerant molecule. This assumption is not valid for very reactive systems, where more than one halide atom is removed from refrigerant molecules. R-123 at elevated temperature levels belongs to this latter category.

## Oil Analysis

The type of analyses performed varied according to the lubricant tested, as follows:

Hydrocarbon Lubricants. Visual observations are recorded and color photographs were prepared to document the color changes of the aged fluids and the formation of decomposition products such as oil sludges, etc. This is consistent with past practices where sealed tube analysis by chemical methods has generally been confined to the analysis of refrigerants only.

Pentaerythritol Esters. The primary evaluation was the determination of acid numbers following the procedures proscribed in ASTM Method D664 "Test of Neutralization Number by Potentiometric Titration".

Where sufficient sample was available an Infrared (IR) spectrum was prepared. The spectrum of the aged oil was then compared to that of unused lubricant, shown as "Differential Spectra" in the Appendix B figures. All spectra were obtained from neat oil samples on NaCl plates.

Note: The interpretation of the IR spectra is limited to statements about gross spectral changes. A more detailed analysis requires an understanding of ester composition as well as that of potential decomposition products, which is beyond the scope of this project.

Polyalkylene Glycols. Size exclusion chromatography (SEC) was used to assess gross molecular changes of aged PAG lubricants. This technique, even more so than IR analysis, greatly benefits from detailed knowledge of the composition of the base materials and that of their potential decomposition products - again a task beyond the scope of this project.

All chromatograms were obtained using chloroform as eluent. This solvent was selected to avoid interference from residual refrigerant. The columns used were two 300 mm length sets of "Phenogel Linear Mix", with an I.D. of 2 mm. Detection was by refractive index.

## COMPLIANCE WITH AGREEMENT

All work performed during this project was in full compliance with the requirements of the original Work Statement or as amended during the course of this project.

The specific changes requested by ARTI were as follows,

(1) the substitution of R-134 for the originally proposed, but unavailable, E-134.

(2) the addition of four sets of sealed tube stability tests involving R-11 and R-123 using the identical test protocol as defined in the work statement, except for the substitution of the 175°C aging temperature by 160°C.

## PRINCIPAL INVESTIGATOR EFFORT

The principal investigator participated in the following Contract activities,

- the procurement and purity analysis of all refrigerant and lubricant samples
- scheduling and supervision of sealed tube preparation
- scheduling and supervision of analysis of aged tubes
- evaluation of test results
- preparation of monthly progress letters, abstracts for presentations at Purdue Compressor Conference and at ASHRAE/ARI Winter Meeting and Show in Chicago in January of 1993, two quarterly progress reports and the final report.

# APPENDIX A

TABLE A-1: STABILITY OF R-22 WITH MINERAL OIL (150 SSU)

SEALED TUBE CONTENTS:

VALVE STEEL STRIP
1.0 CC (0.9 G) OF TEST LUBRICANT
0.9 G OF R-22

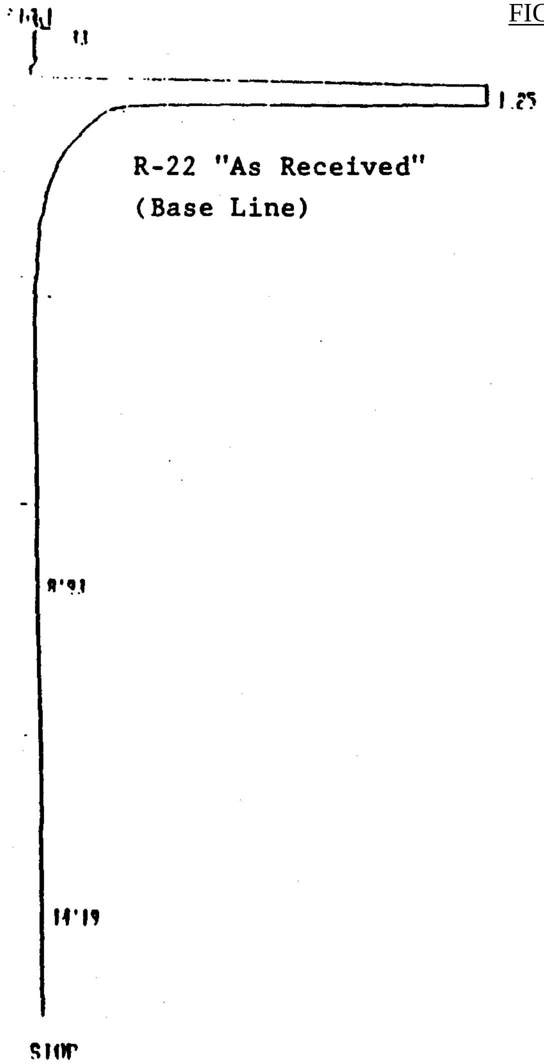
AGING SCHEDULE:

14 DAYS AT TEMPERATURES SHOWN

TEST RESULTS:

| AGING TEMP, °C (°F) | VISUAL APPEARANCE OF AGED TUBES | | CHEMICAL ANALYSIS | | |
|---|---|---|---|---|---|
| | COLOR CHANGE OF OIL (ASTM) | OTHER VISUAL OBSERVATIONS | GC | CL ION (UG) | %R-22 REACTED |
| 150 (300) | FROM 2.5 To 3.0 | STEEL: NO CHANGE | NO CHANGE | 2.5 | 0.001 |
| 175 (347) | FROM 2.5 To 3.0 | STEEL: NO CHANGE | NO CHANGE | 10.0 | 0.003 |
| 200 (392) | FROM 2.5 To 3.0 | DARKENING OF STEEL | NO CHANGE | 16.7 | 0.004 |

FIGURE A-1

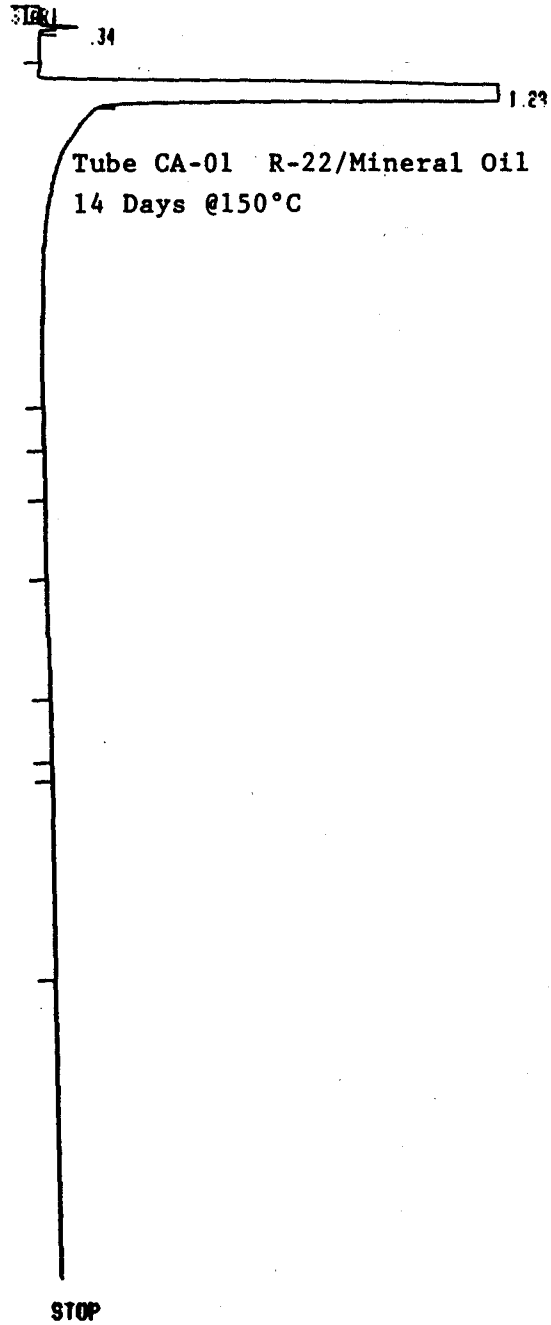



RUN # 13 JAN/16/92 14:57:36

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 945 | BB | 0.053 | 0.035 |
| 1.25 | 2726000 | PB | 0.155 | 99.965 |

TOTAL AREA= 2727000
MUL FACTOR= 1.0000E+00

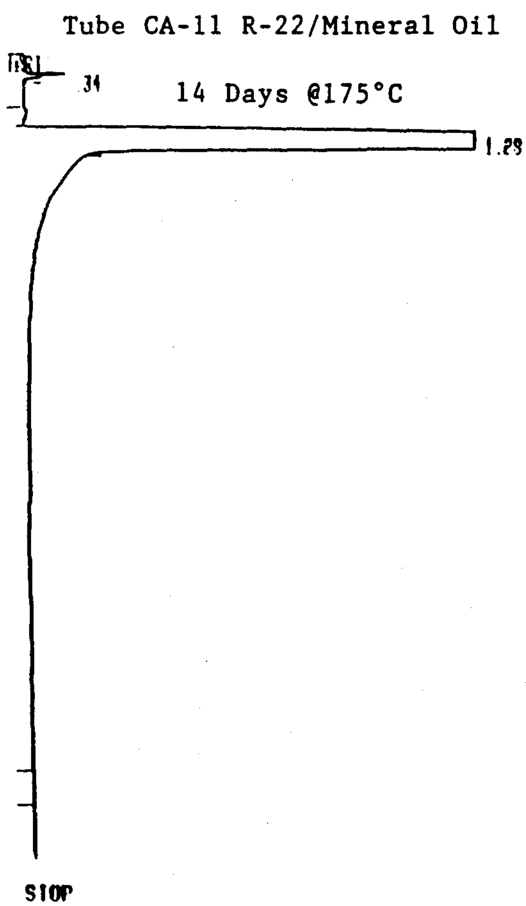



RUN # 11 FEB/26/92 14:19:20

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 2126 | BB | 0.053 | 0.126 |
| 1.28 | 1683000 | PB | 0.140 | 99.874 |

FIGURE A-2

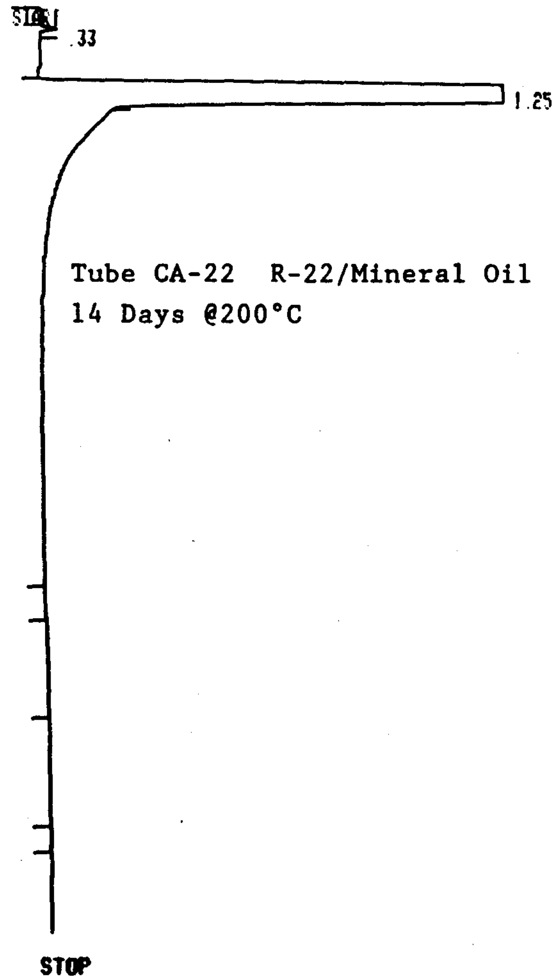



RUN # 12 FEB/26/92 14:56:53

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 2301 | BB | 0.052 | 0.135 |
| 1.28 | 1704800 | PB | 0.140 | 99.865 |

TOTAL AREA= 1707100
MUL FACTOR= 1.0000E+00




RUN # 16 APR/15/92 15:59:43

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 920 | BB | 0.051 | 0.053 |
| 1.25 | 1730200 | PB | 0.139 | 99.947 |

TOTAL AREA= 1731100
MUL FACTOR= 1.0000E+00

TABLE A-2: STABILITY OF R-32 WITH PENTAERYTHRITOL ESTER (MIXED ACID I)

SEALED TUBE CONTENTS:

VALVE STEEL STRIP
1.0 cc (1.0 G) OF TEST LUBRICANT
1.0 G OF R-32

AGING SCHEDULE:

14 DAYS AT TEMPERATURES SHOWN

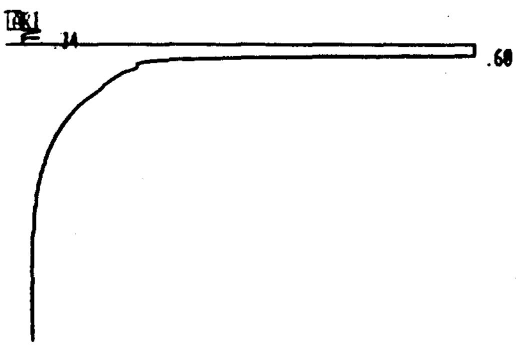
TEST RESULTS:

| | VISUAL APPEARANCE | | CHEMICAL ANALYSIS | | | | |
|---|---|---|---|---|---|---|---|
| AGING TEMP. °C, (°F) | OIL COLOR CHANGE (ASTM) | OTHER VISUAL CHANGES | GC | F ION (UG) | %R-32 REACTED | OIL ACID NO. | OIL IR (DIFFERENTIAL SCAN) |
| 150 (300) | NO CHANGE AT 2.5 | STEEL: NO CHANGE | NO CHANGE | 6.0 | 0.002 | 0.00 | NO SIGNIFICANT CHANGES |
| 175 (347) | NO CHANGE AT 2.5 | STEEL: NO CHANGE | NO CHANGE | 4.7 | 0.001 | 0.00 | NO SIGNIFICANT CHANGES |
| 200 (392) | FROM 2.5 TO 3.0 | STEEL: NO CHANGE | NO CHANGE | 4.2 | 0.001 | 1.67 | NO SIGNIFICANT CHANGES |

(FLUORIDE ANALYSIS BLANK: 3.1)

FIGURE A-3

R-32 "As Received"




STOP

RUN # 2 FEB/19/92 08:56:24

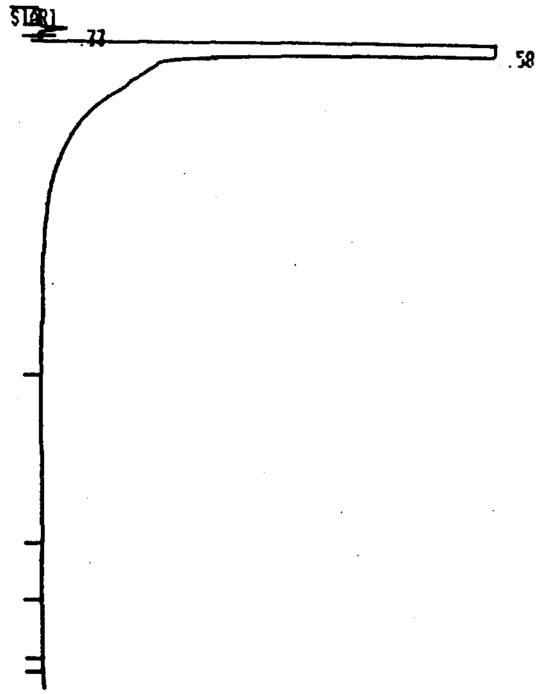
AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 1063 | BB | 0.053 | 0.045 |
| 0.60 | 2367700 | PB | 0.080 | 99.955 |

TOTAL AREA= 2368800
MUL FACTOR= 1.0000E+00

Tube CB-01 R-32/PE Ester (mixed acid I)

14 Days @ 150°C




STOP

RUN # 3 MAR/18/92 11:06:37

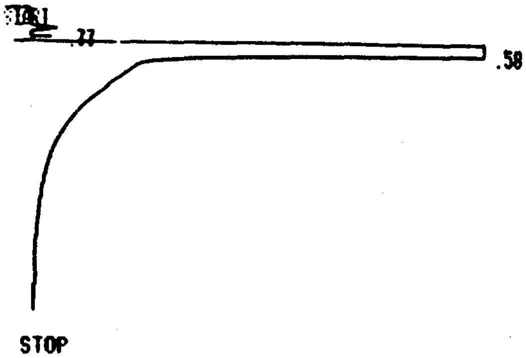
AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 1506 | BB | 0.050 | 0.059 |
| 0.58 | 2553000 | PB | 0.081 | 99.941 |

TOTAL AREA= 2554500
MUL FACTOR= 1.0000E+00

FIGURE A-4

Tube CB-12
R-32/PE Ester (mixedacid I)
14 Days @ 175°C



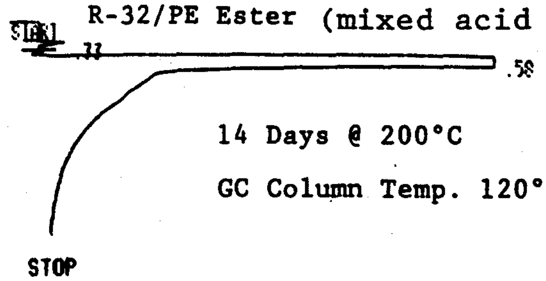

RUN # 4 MAR/18/92 11:40:26

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 1035 | PB | 0.038 | 0.040 |
| 0.58 | 2579000 | PB | 0.081 | 99.960 |

TOTAL AREA= 2580000
MUL FACTOR= 1.0000E+00

Tube CB-21
R-32/PE Ester (mixed acid I)



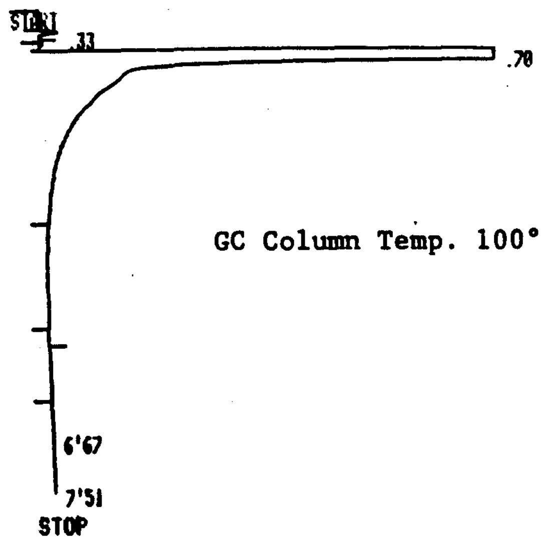

14 Days @ 200°C

GC Column Temp. 120°C

RUN # 3 APR/01/92 09:00:36

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 1234 | BB | 0.048 | 0.051 |
| 0.58 | 2400000 | PB | 0.079 | 99.949 |




GC Column Temp. 100°C

RUN # 4 APR/01/92 09:13:42

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 966 | BB | 0.051 | 0.045 |
| 0.70 | 2141000 | PB | 0.088 | 99.955 |

TOTAL AREA= 2142000
MUL FACTOR= 1.0000E+00

TABLE A-3: STABILITY OF R-32 WITH POLYPROPYLENE GLYCOL (BUTYL MONOETHER)

SEALED TUBE CONTENTS:

VALVE STEEL STRIP
1.0 cc (1.0 g) OF TEST LUBRICANT
1.0 g OF R-32

AGING SCHEDULE:

14 DAYS AT TEMPERATURES SHOWN

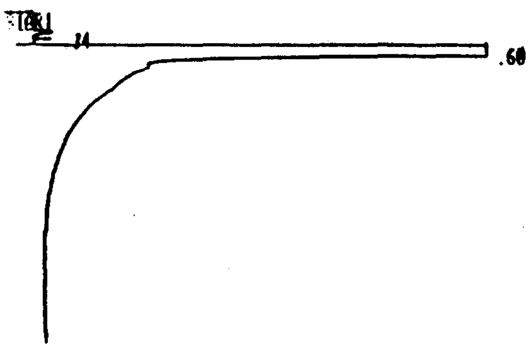
TEST RESULTS:

| | VISUAL APPEARANCE | | CHEMICAL ANALYSIS | | | |
|---|---|---|---|---|---|---|
| AGING TEMP. °C (°F) | OIL COLOR CHANGE (ASTM) | OTHER VISUAL CHANGES | GC | F ION (UG) | %R-32 REACTED | SIZE EXCLUSION CHROMATOGRAPHY OF OIL |
| 150 (300) | NO CHANGE AT 2.0 | STEEL: NO CHANGE | NO CHANGE | 0.2 | 0.000 | MINOR CHANGES IN STRUCTURE OF PAG |
| 175 (347) | NO CHANGE AT 2.5 | STEEL: NO CHANGE | NO CHANGE | 5.4 | 0.002 | MINOR CHANGES IN STRUCTURE |
| 200 (392) | NO CHANGE AT 2.5 | STEEL: NO CHANGE | NO CHANGE | 14.1 | 0.004 | MINOR CHANGES IN STRUCTURE |

(FLUORIDE ANALYSIS BLANK: 3.1)

FIGURE A-05

R-32 "As Received"



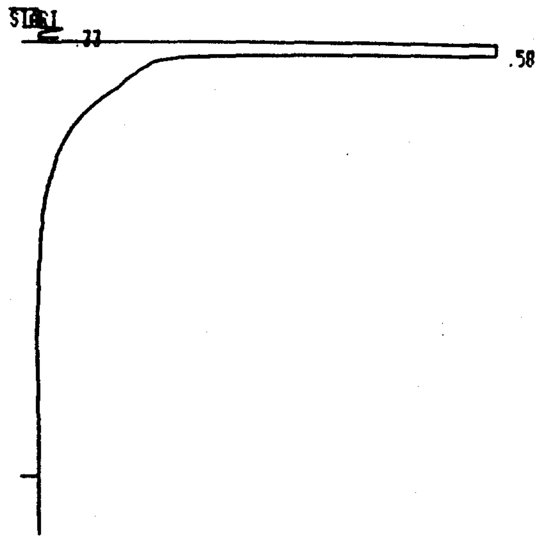

RUN # 2 FEB/19/92 08:56:24

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 1063 | BB | 0.053 | 0.045 |
| 0.60 | 2367700 | PB | 0.080 | 99.955 |

TOTAL AREA= 2368800
MUL FACTOR= 1.0000E+00

Tube CC-01 R32/PPG (butyl mono-ether)
14 Days @ 150°C




RUN # 2 APR/15/92 09:25:55

AREA%

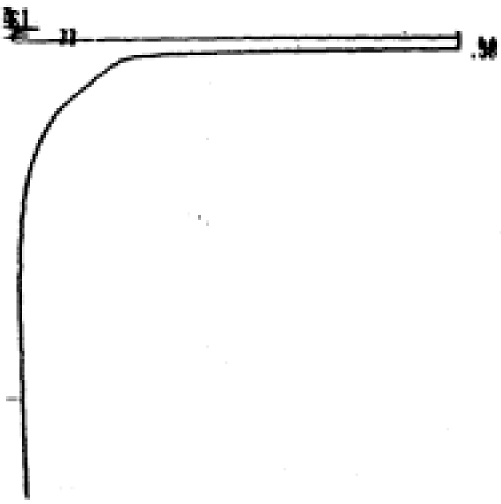
| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 1081 | BB | 0.045 | 0.043 |
| 0.58 | 2527000 | PB | 0.080 | 99.957 |

TOTAL AREA= 2528100
MUL FACTOR= 1.0000E+00

FIGURE A-06

Tube CC-11 R-32/PPG

14 Days @175°C




RUN # 3 APR/15/92 10:01:40

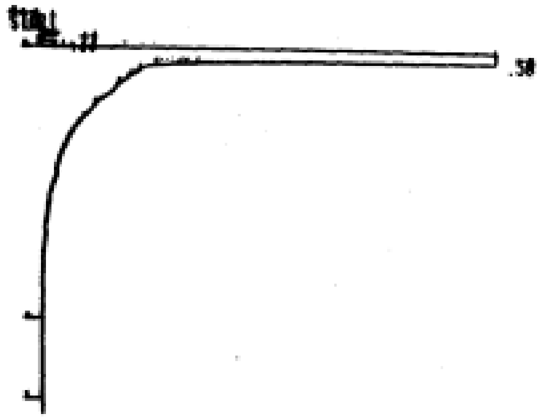
AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 1530 | BB | 0.050 | 0.061 |
| 0.50 | 2514400 | PB | 0.080 | 99.939 |

TOTAL AREA= 2515900
MUL FACTOR= 1.0000E+00

Tube CC-22 R32/PPG

14 Days @200°C




RUN # 4 APR/15/92 10:31:12

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 1067 | PB | 0.049 | 0.047 |
| 0.50 | 2202100 | PB | 0.070 | 99.953 |

TOTAL AREA= 2203200
MUL FACTOR= 1.0000E+00

TABLE A-4: STABILITY OF R-123 WITH MINERAL OIL (ISO VG32)

SEALED TUBE CONTENTS:

VALVE STEEL STRIP
1.0 CC (1.0 G) OF TEST LUBRICANT
0.9 G OF R-123

AGING SCHEDULE:

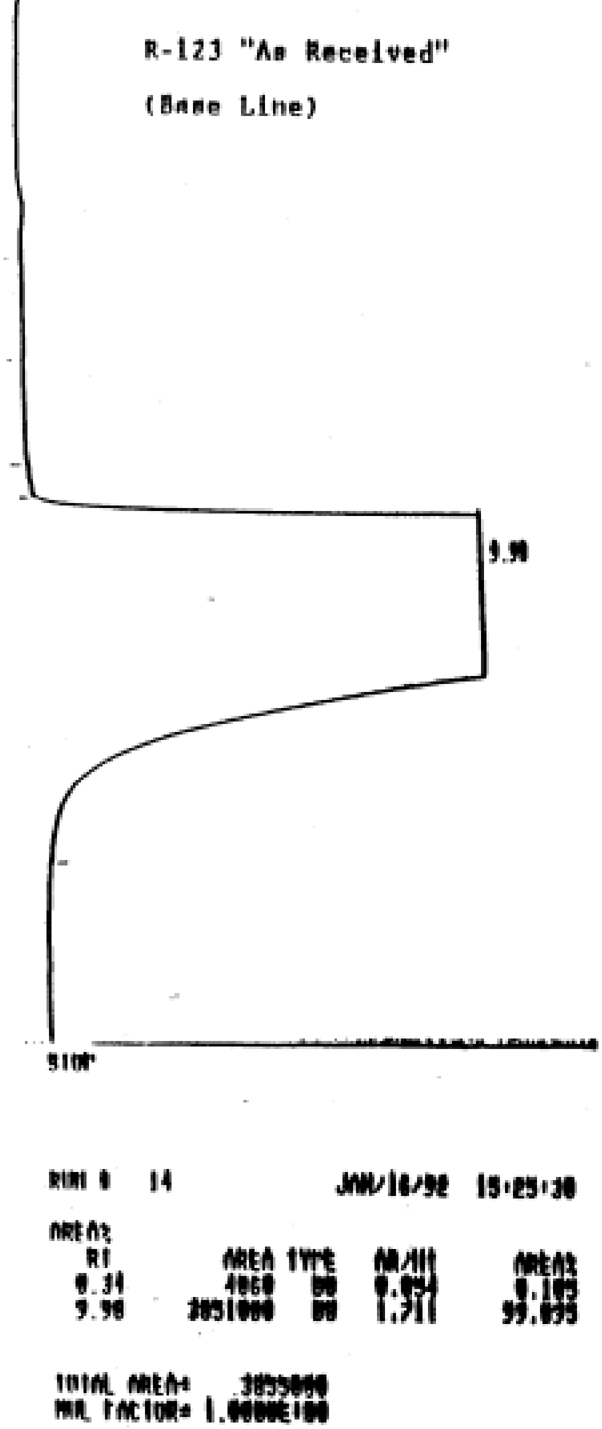
14 DAYS AT TEMPERATURES SHOWN

TEST RESULTS:

| | VISUAL APPEARANCE OF TUBES | | CHEMICAL ANALYSIS | | |
|---|---|---|---|---|---|
| AGING TEMP. °C, (°F) | OIL COLOR CHANGE (ASTM) | OTHER VISUAL OBSERVATIONS | GC - % OF R-123 REACTED (UNCORRECTED), PRODUCTS FOUND | CL ION (UG) | %R-123 REACTED (FROM CL ION) |
| 105 (220) | FROM 2.5 TO 3.0 | STEEL: BROWN STAIN | NO CHANGE | 98.7 | 0.045 |
| | FROM 2. 5 TO 3.0 | STEEL: BROWN | NO CHANGE | 65.8 | 0.032 |
| 150 (300) | FROM 2.5 TO 7.0 | STEEL: ETCHED | 1.8 R-133A | 3,260 | 1.56 |
| | FROM 2.5 TO 7.5 | STEEL: LIGHT SLUDGE DEPOSITS | 3.4 R-133A | 5,660 | 2.71 |
| 160 (320) | FROM 2.5 TO >8.0 (BLACK) | BLACK SLUDGE, STEEL: SLUDGE DEPOSITS | 14.0 R-133A<br>0.45 R-143A | 54,400 | 26.0 |
| 175 (347) | FROM 2.5 TO >8.0 (BLACK) | BLACK SLUDGE, STEEL: ETCHED, SLUDGE DEPOSITS | 44.5 R-133A<br>2.0 R-143A | 169,440 | 40.5 |

NOTE: SEE PHOTOGRAPHIC RECORD OF VISUAL OBSERVATIONS IN APPENDIX D

FIGURE A-7


R-123 "As Received"
(Base Line)
9.98
STOP
RUN # 14
AREA%
RT
AREA TYPE
AR/HT
AREA%
TOTAL AREA=
MUL FACTOR= 1.0000E+00

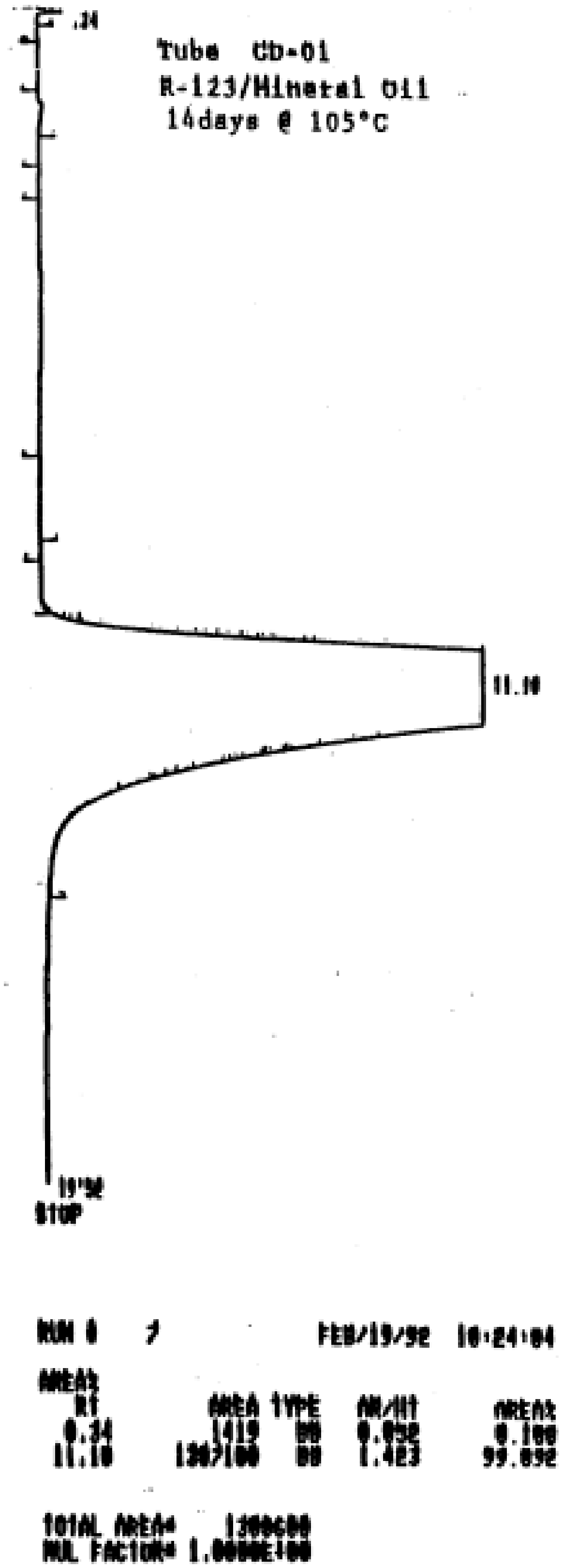
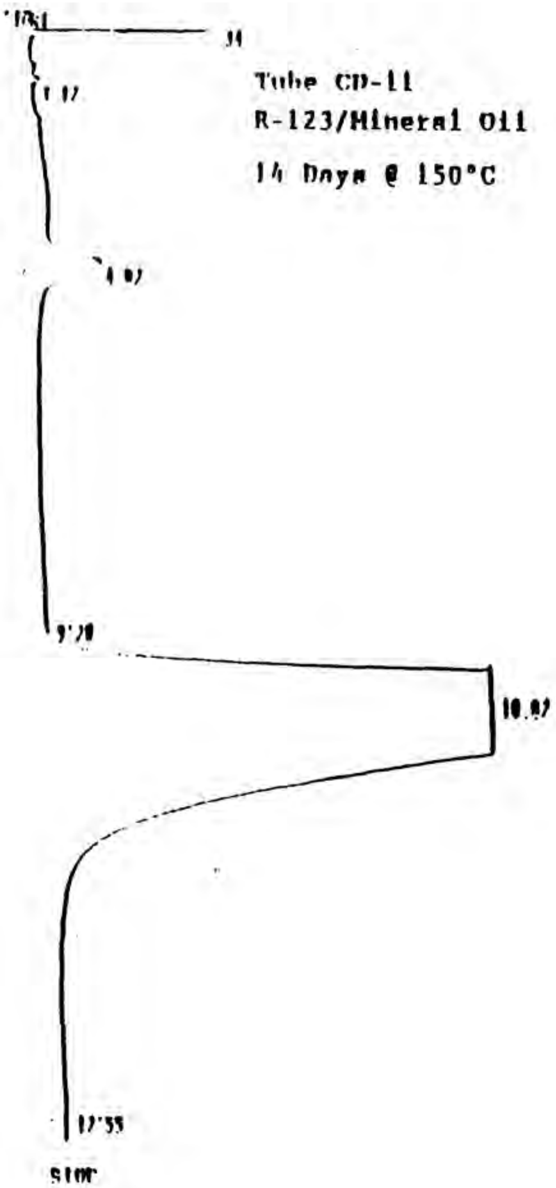

Tube Cb-01
R-123/Mineral Oil
14days @ 105°C
11.10
STOP
AREA%
RT
AREA TYPE
AR/HT
AREA%
0.34
11.10
TOTAL AREA=
MUL FACTOR= 1.0000E+00

FIGURE A-8

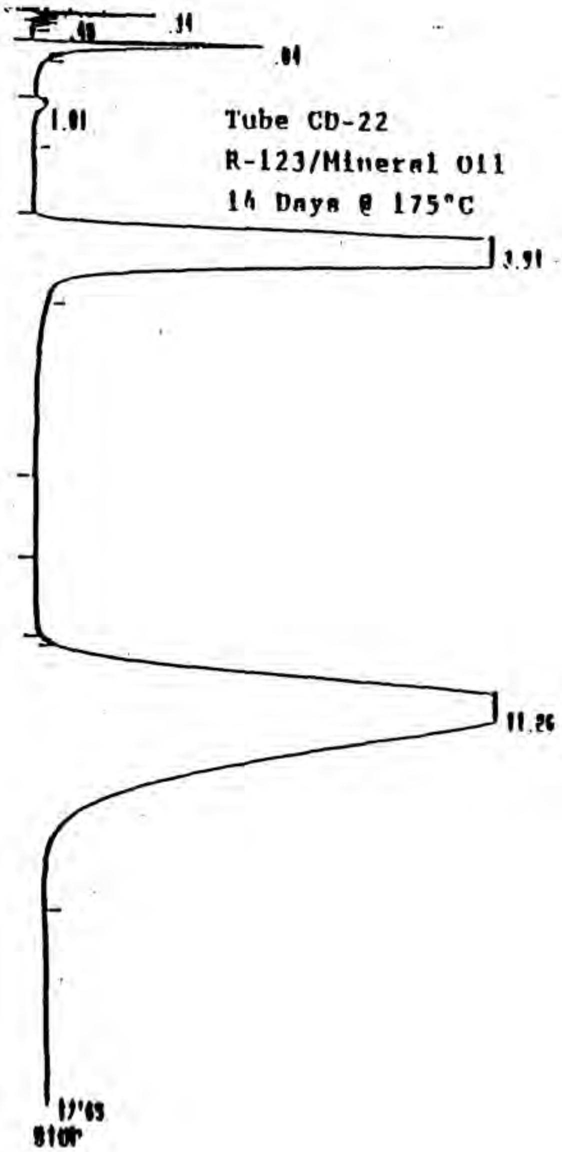



RUN # 3 FEB/26/92 10:02:42

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 9670 | PB | 0.050 | 0.663 |
| 1.17 | 795 | PB | 0.102 | 0.054 |
| 4.07 | 25297 | BB | 0.400 | 1.726 |
| 10.07 | 1424700 | BB | 1.473 | 97.557 |

TOTAL AREA= 1460400
MUL FACTOR= 1.0000E+00



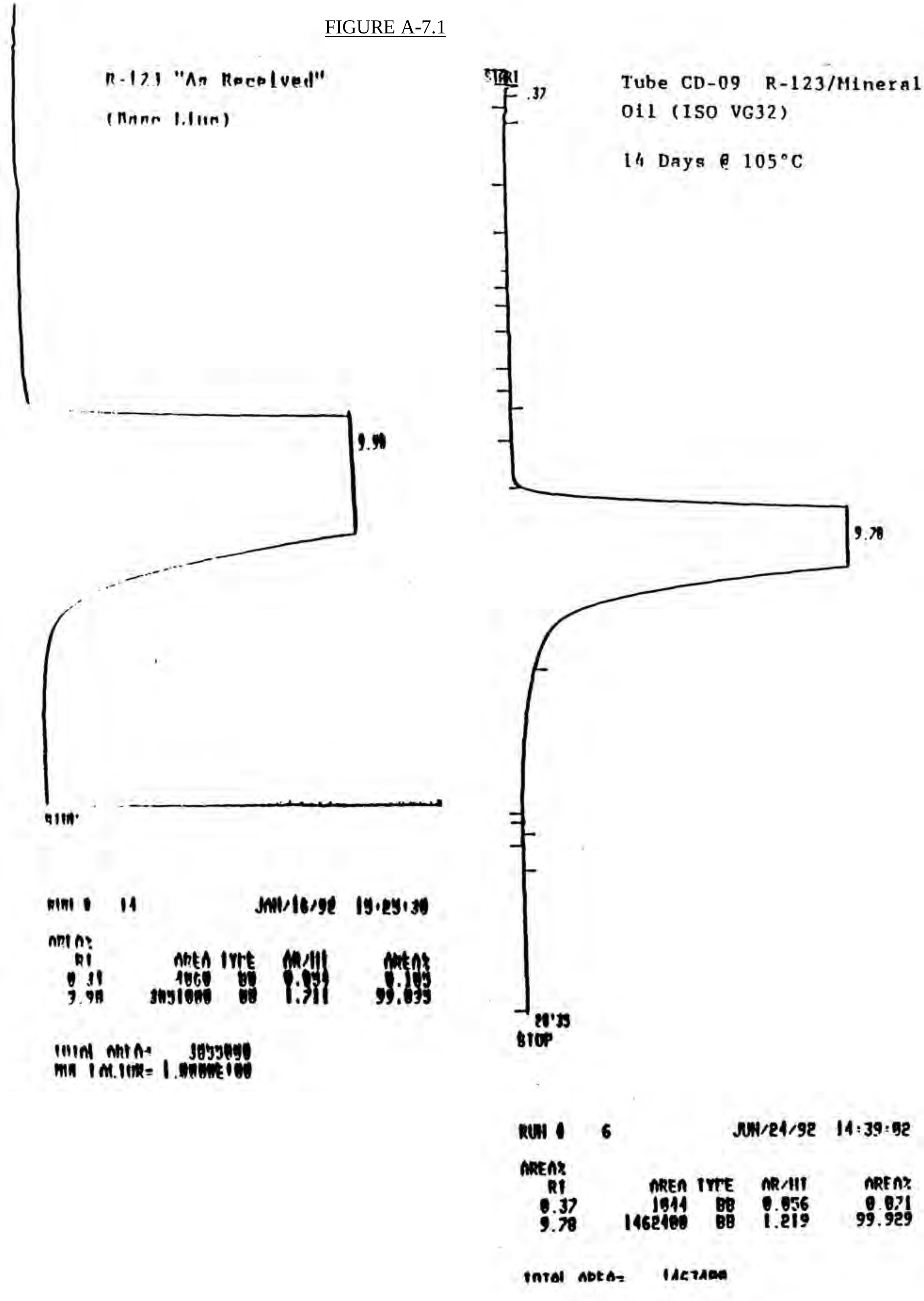

RUN # 7 FEB/26/92 19:47:11

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 6912 | BV | 0.052 | 0.520 |
| 0.49 | 1427 | VB | 0.033 | 0.111 |
| 0.84 | 25817 | PB | 0.107 | 2.001 |
| 1.01 | 2790 | BB | 0.202 | 0.216 |
| 3.94 | 543500 | PB | 0.410 | 42.127 |
| 11.26 | 709810 | BB | 1.359 | 55.018 |

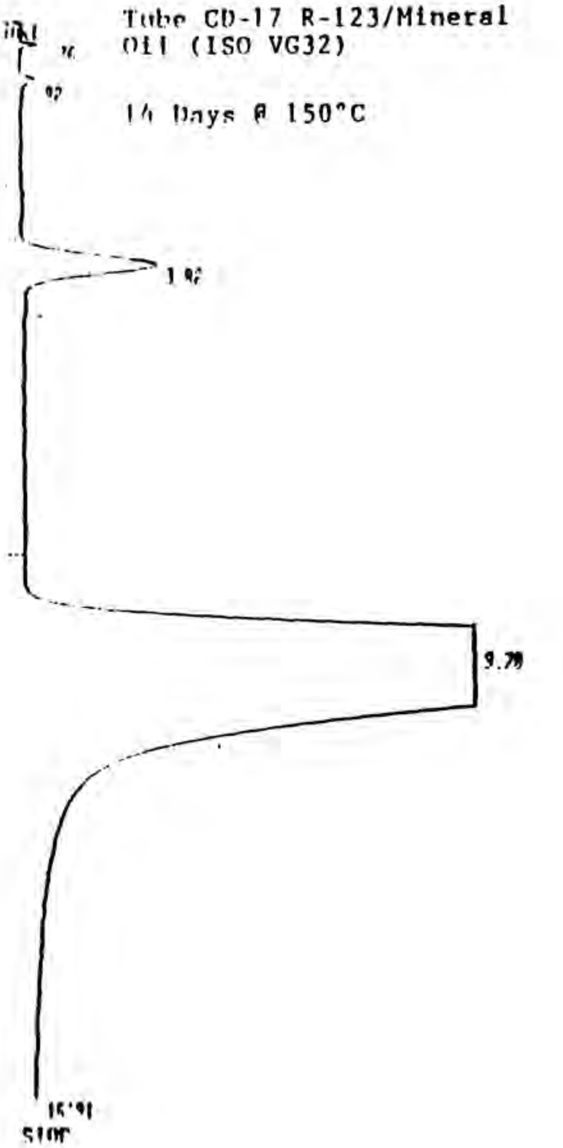
TOTAL AREA= 1290100
MUL FACTOR= 1.0000E+00

FIGURE A-7.1



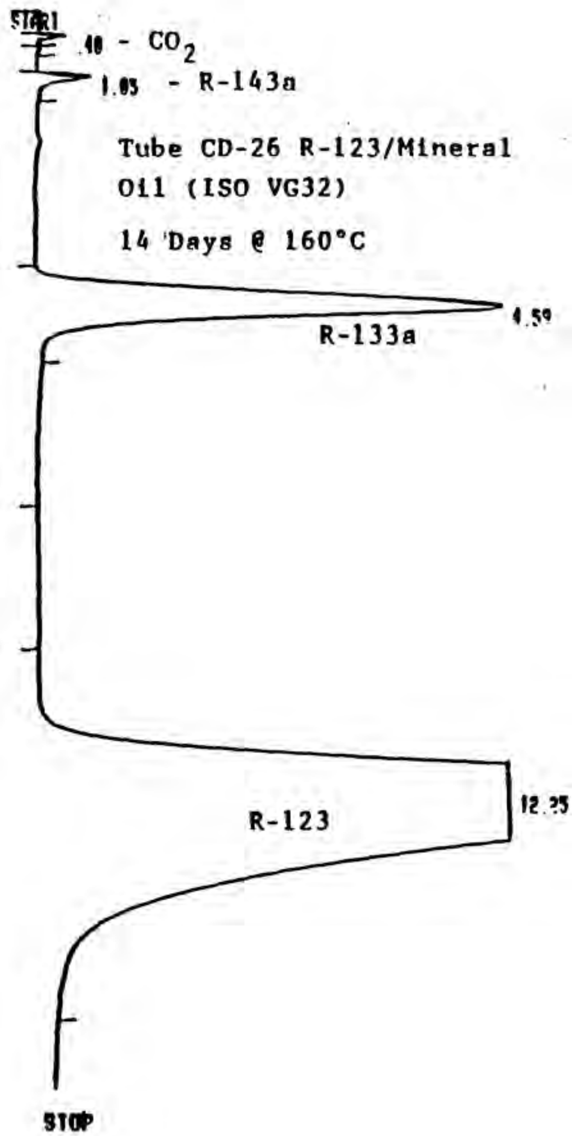
"As Received"
9.90
Tube CD-09 R-123/Mineral
Oil (ISO VG32)
14 Days @ 105°C
START
.37
9.78
STOP
RUN # 6
JUN/24/92 14:39:02
AREA%
RT AREA TYPE AR/HT AREA%
0.37 1044 BB 0.056 0.071
9.78 1462400 BB 1.219 99.929

FIGURE A8.1




| RUN # 7 | | | JUN/24/92 | 15:23:44 |
|---|---|---|---|---|
| AREA% | | | | |
| RT | AREA | TYPE | AR/HT | AREA% |
| 0.36 | 1059 | PB | 0.053 | 0.070 |
| 0.92 | 1724 | PB | 0.115 | 0.114 |
| 1.92 | 51000 | BB | 0.352 | 3.375 |
| 9.78 | 1457400 | BB | 1.222 | 96.441 |

TOTAL AREA= 1511200
MUL FACTOR= 1.0000E+00




| RUN # 2 | | | JUL/22/92 | 15:34:55 |
|---|---|---|---|---|
| AREA% | | | | |
| RT | AREA | TYPE | AR/HT | AREA% |
| 0.40 | 1700 | BB | 0.061 | 0.113 |
| 1.05 | 6777 | PB | 0.121 | 0.448 |
| 4.59 | 212500 | PB | 0.436 | 14.034 |
| 12.25 | 1293100 | PB | 1.449 | 85.405 |

TOTAL AREA= 1514100
MUL FACTOR= 1.0000E+00

TABLE A-5: STABILITY OF R-124 WITH ALKYLBENZENE OIL

SEALED TUBE CONTENTS:

VALVE STEEL STRIP
1.0 cc (0.9 G) OF TEST LUBRICANT
0.9 G OF R-124

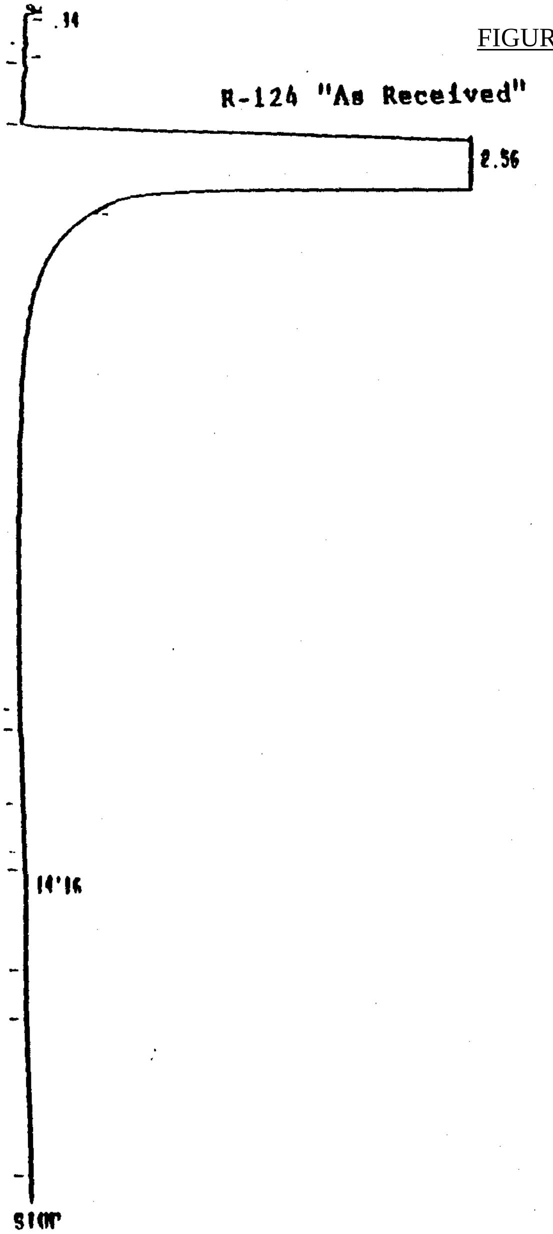
AGING SCHEDULE:

14 DAYS AT TEMPERATURES SHOWN

TEST RESULTS:

| | VISUAL APPEARANCE OF TUBES | | CHEMICAL ANALYSIS | | |
|---|---|---|---|---|---|
| AGING TEMP, °C (°F) | OIL COLOR CHANGE (ASTM) | OTHER VISUAL OBSERVATIONS | GC | CL ION (UG) | %R-124 REACTED |
| 150 (300) | NO CHANGE AT 2.0 | STEEL: NO CHANGE | NO CHANGE | 18.4 | 0.008 |
| 175 (347) | FROM 2.0 TO 2.5 | STEEL: NO CHANGE | NO CHANGE | 36.1 | 0.015 |
| 200 (392) | FROM 2.0 TO 3.0 | STEEL: BROWN STAINS | NO MEASURABLE CHANGE | 122.7 | 0.052 |

FIGURE A-9

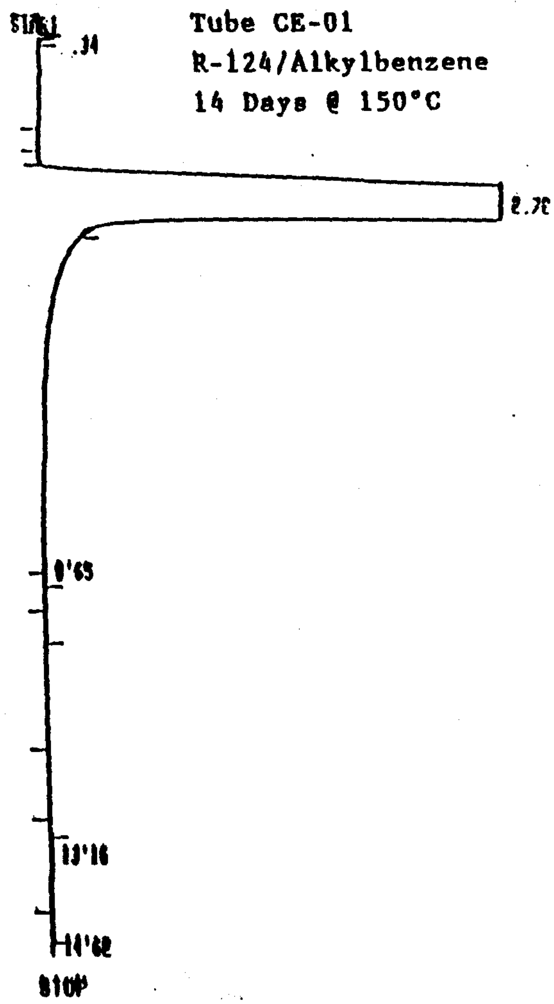



RUN # 26 JAN/17/92 15:59:17

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.31 | 965 | BB | 0.054 | 0.025 |
| 2.56 | 3050100 | PB | 0.380 | 99.975 |

TOTAL AREA= 3051100
MUL FACTOR= 1.0000E+00




RUN # 9 FEB/26/92 11:34:49

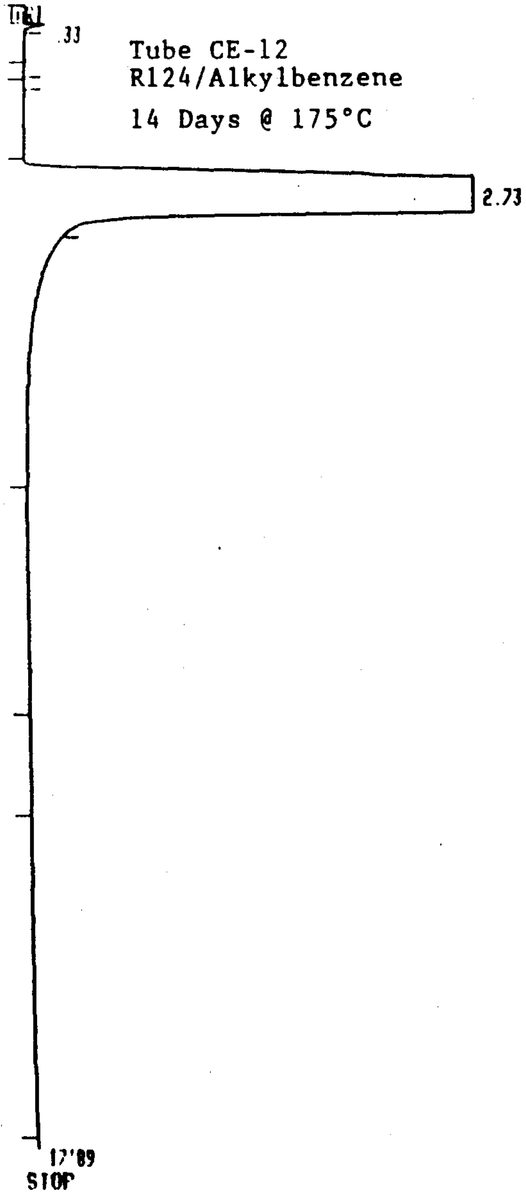
AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 1217 | BB | 0.053 | 0.086 |
| 2.72 | 1423000 | PB | 0.322 | 99.915 |

TOTAL AREA= 1424200
MUL FACTOR= 1.0000E+00

FIGURE A-10

Tube CE-12
R124/Alkylbenzene
14 Days @ 175°C



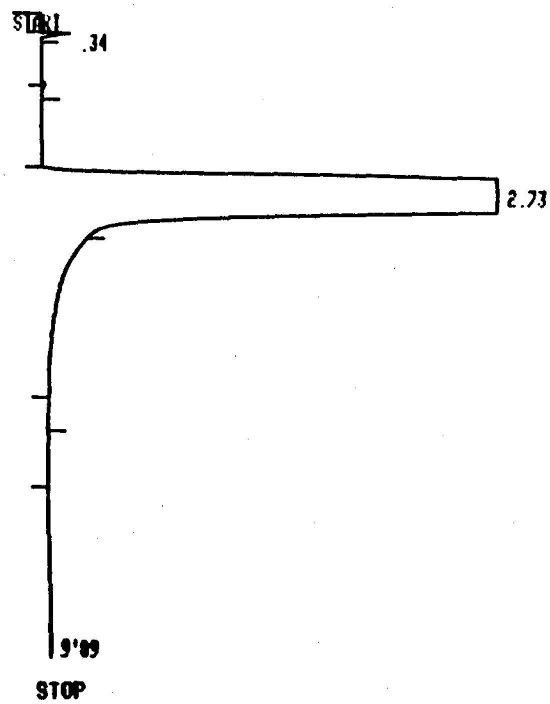

RUN # 10 FEB/26/92 12:19:26

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 1221 | BB | 0.054 | 0.090 |
| 2.73 | 1354600 | PB | 0.322 | 99.910 |

TOTAL AREA= 1355900
MUL FACTOR= 1.0000E+00

Tube CE-21
R-124/Alkylbenzene
14 Days @ 200°C




RUN # 5 APR/01/92 09:49:55

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 1545 | BB | 0.051 | 0.111 |
| 2.73 | 1386000 | PB | 0.323 | 99.889 |

TOTAL AREA= 1387500
MUL FACTOR= 1.0000E+00

TABLE A-6: STABILITY OF R-125 WITH POLYPROPYLENE GLYCOL (BUTYL MONOETHER)

SEALED TUBE CONTENTS:

VALVE STEEL STRIP
1.0 cc (1.0 g) OF TEST LUBRICANT
1.0 g OF R-125

AGING SCHEDULE:

14 DAYS AT TEMPERATURES SHOWN

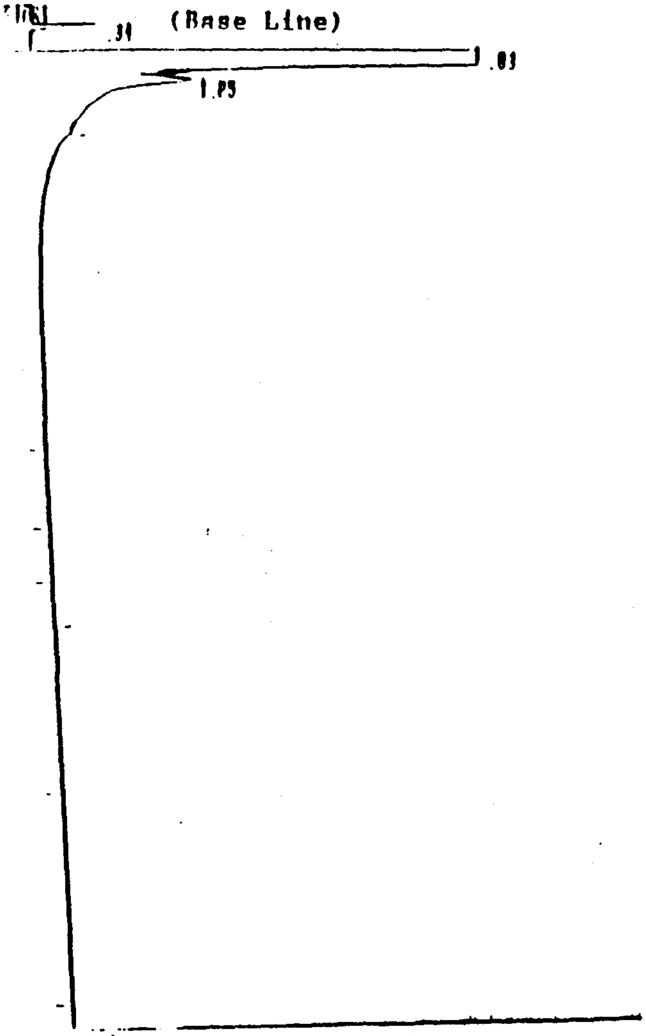
TEST RESULTS:

| | VISUAL APPEARANCE | | CHEMICAL ANALYSIS | | | |
|---|---|---|---|---|---|---|
| AGING TEMP. °C (°F) | OIL COLOR CHANGE (ASTM) | OTHER VISUAL CHANGES | GC | F ION (UG) | %R-125 REACTED | SIZE EXCLUSION CHROMATOGRAPHY OF OIL |
| 150 (300) | NO CHANGE AT 2.5 | STEEL: NO CHANGE | ELUENT AT 0.62 MIN. | 1.6 | 0.001 | CHANGES IN PAG STRUCTURE |
| 175 (347) | NO CHANGE AT 2.5 | STEEL: NO CHANGE | ELUENT AT 0.62 MIN. | 3.0 | 0.002 | CHANGES IN PAG STRUCTURE |
| 200 (392) | NO CHANGE AT 2.5 | STEEL: BLACKENED | NO CHANGE | 3.1 | 0.002 | CHANGES IN PAG STRUCTURE |

(FLUORIDE ANALYSIS BLANK: 3.6)

FIGURE A-11

R-125 "As Received"
(Base Line)




RUN # 25 JAN/17/92 14:49:04

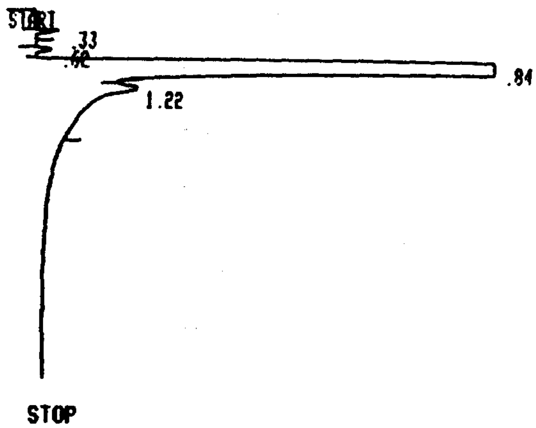
AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.31 | 3599 | BB | 0.050 | 0.106 |
| 0.83 | 3390200 | PB | 0.117 | 99.894 |
| 1.25 | 0 | BB | 0.000 | 0.000 |

TOTAL AREA= 3393800
MUL FACTOR= 1.0000E+00

Tube CF-01 R-125/PPG (monobutyl-ether)

14 Days @ 150°C




RUN # 5 APR/15/92 10:48:37

AREA%

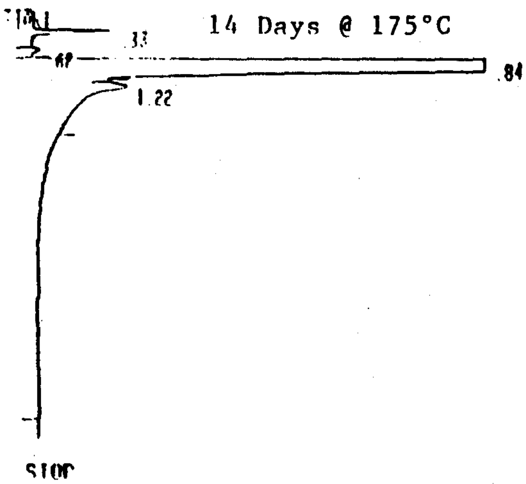
| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 1130 | BB | 0.049 | 0.069 |
| 0.62 | 911 | BP | 0.057 | 0.056 |
| 0.84 | 1626600 | PB | 0.100 | 99.847 |
| 1.22 | 453 | BB | 0.016 | 0.028 |

TOTAL AREA= 1629100
MUL FACTOR= 1.0000E+00

FIGURE A-12

Tube CF-11 R-125/PPG (monobutyl ether) Tube CF-21

14 Days @ 175°C



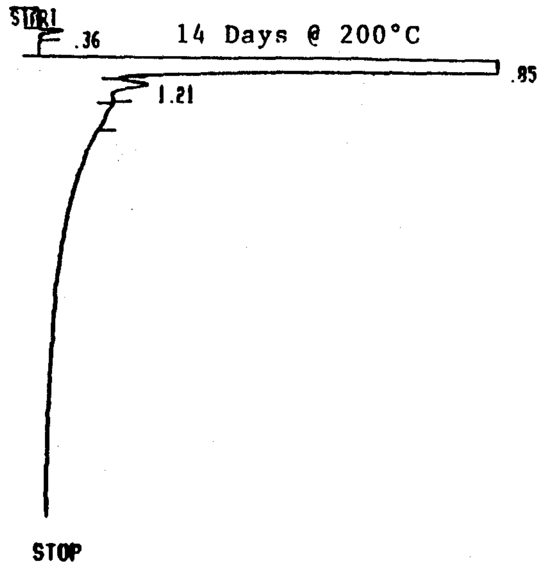

RUN # 6 APR/15/92 11:07:09

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 4151 | PB | 0.050 | 0.256 |
| 0.62 | 486 | PB | 0.056 | 0.030 |
| 0.84 | 1618400 | PB | 0.100 | 99.696 |
| 1.22 | 459 | BB | 0.017 | 0.028 |

TOTAL AREA= 1623500
MUL FACTOR= 1.0000E+00

14 Days @ 200°C




RUN # 11 JUN/11/92 10:11:34

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.36 | 1095 | PB | 0.047 | 0.062 |
| 0.85 | 1773600 | PB | 0.097 | 99.751 |
| 1.21 | 3334 | PB | 0.101 | 0.188 |

TOTAL AREA= 1778100
MUL FACTOR= 1.0000E+00

TABLE A-7: STABILITY OF R-125 WITH MODIFIED POLYGLYCOL

SEALED TUBE CONTENTS:

VALVE STEEL STRIP
1.0 cc (1.0 g) OF TEST LUBRICANT
1.0 g OF R-125

AGING SCHEDULE:

14 DAYS AT TEMPERATURES SHOWN

TEST RESULTS:

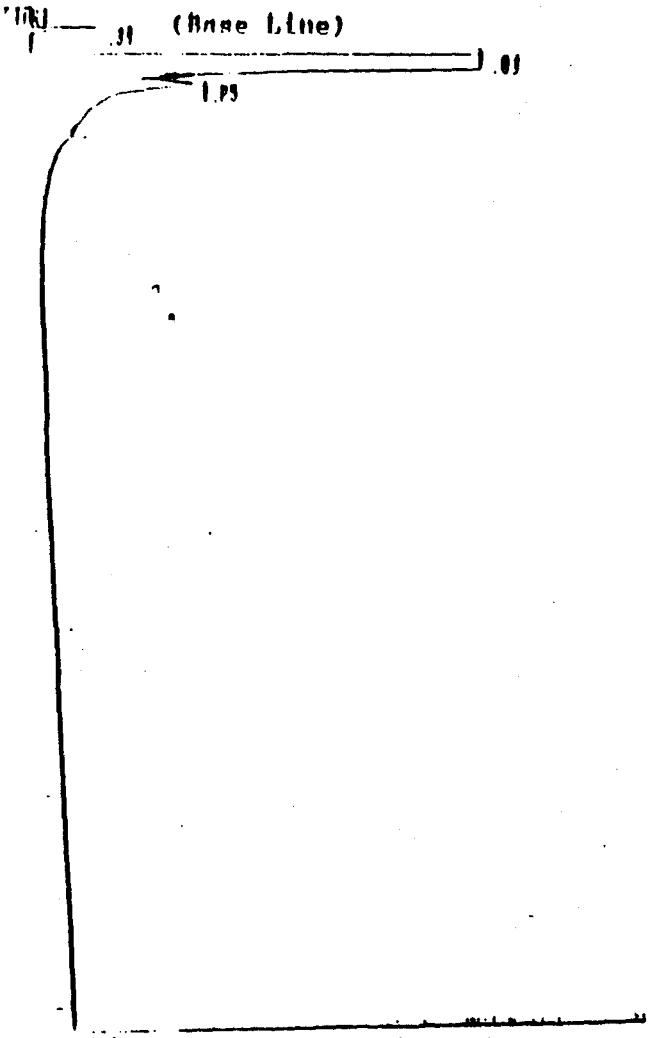
| | VISUAL APPEARANCE | | CHEMICAL ANALYSIS | | | |
|---|---|---|---|---|---|---|
| AGING TEMP. °C, (°F) | OIL COLOR CHANGE (ASTM) | OTHER VISUAL CHANGES | GC | F ION (UG) | %R-125 REACTED | SIZE EXCLUSION CHROMATOGRAPHY OF OIL |
| 150 (300) | FROM 2.5 TO 3.0 | STEEL: NO CHANGE | NO CHANGE | 11.0 | 0.007 | CHANGES IN PAG STRUCTURE |
| 175 (347) | FROM 2.5 TO 3.5 | STEEL: NO CHANGE | NO CHANGE | 7.1 | 0.004 | CHANGES IN PAG STRUCTURE |
| 200 (392) | FROM 2.5 TO 4.0 | STEED.: NO CHANGE | NO CHANGE | 8.5 | 0.005 | CHANGES IN PAG STRUCTURE |

(FLUORIDE ANALYSIS BLANK: 8.2)

FIGURE A-13

Gas Chromatograms R-125/Modified Polyglycol

R-125 "As Received"




RUN # 29 JAN/17/92 14:49:04

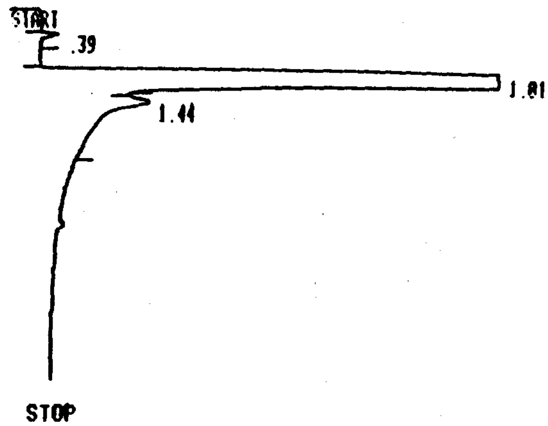
AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.31 | 3599 | BB | 0.050 | 0.106 |
| 0.83 | 3390200 | PB | 0.112 | 99.894 |
| 1.29 | 0 | BB | 0.000 | 0.000 |

TOTAL AREA= 3393800
MUL FACTOR= 1.0000E+00

Tube CG-01 (14 days @ 150°C)

14 Days @ 150°C




RUN # 19 JUL/22/92 10:36:38

AREA%

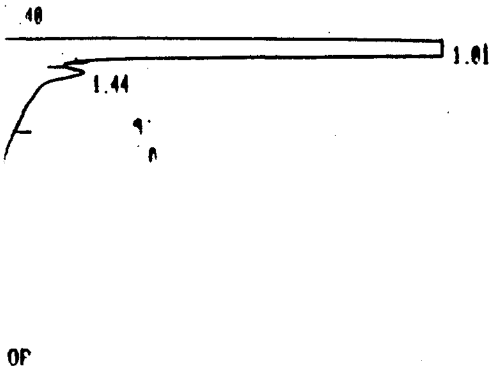
| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.39 | 1074 | PB | 0.059 | 0.058 |
| 1.01 | 1856700 | PB | 0.114 | 99.927 |
| 1.44 | 285 | BB | 0.009 | 0.015 |

TOTAL AREA= 1858100
MUL FACTOR= 1.0000E+00

FIGURE A-14

Gas Chromatograms R-125/Modified Polyglycol

Tube CG-11 (14 days @175°C)



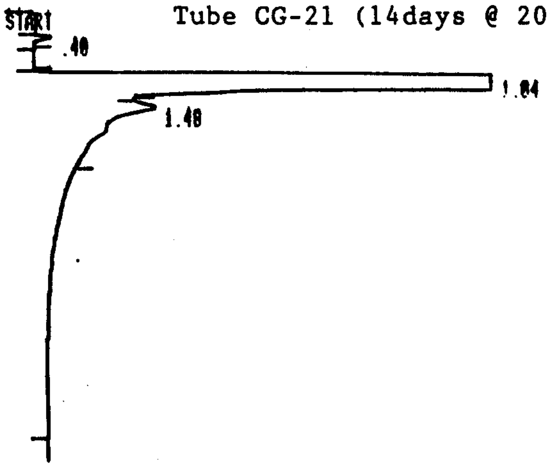

| IN # | 20 | | JUL/22/92 | 11:04:51 |
|---|---|---|---|---|

'EA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.40 | 944 | PB | 0.056 | 0.052 |
| 1.01 | 1815500 | PB | 0.114 | 99.948 |
| 1.44 | 0 | BB | 0.000 | 0.000 |

)TAL AREA= 1816500
JL FACTOR= 1.0000E+00

Tube CG-21 (14days @ 200°C)




| RUN # | 4 | | JUL/23/92 | 15:45:24 |
|---|---|---|---|---|

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.40 | 1202 | PB | 0.065 | 0.065 |
| 1.04 | 1850100 | PB | 0.116 | 99.833 |
| 1.40 | 1892 | BB | 0.061 | 0.102 |

TOTAL AREA= 1853200
MUL FACTOR= 1.0000E+00

TABLE A-8: STABILITY OF R-125 WITH PENTAERYTHRITOL ESTER (MIXED ACID I)

SEALED TUBE CONTENTS:

VALVE STEEL STRIP
1.0 CC (1.0 G) OF TEST LUBRICANT
1.0 G OF R-125

AGING SCHEDULE:

14 DAYS AT TEMPERATURES SHOWN

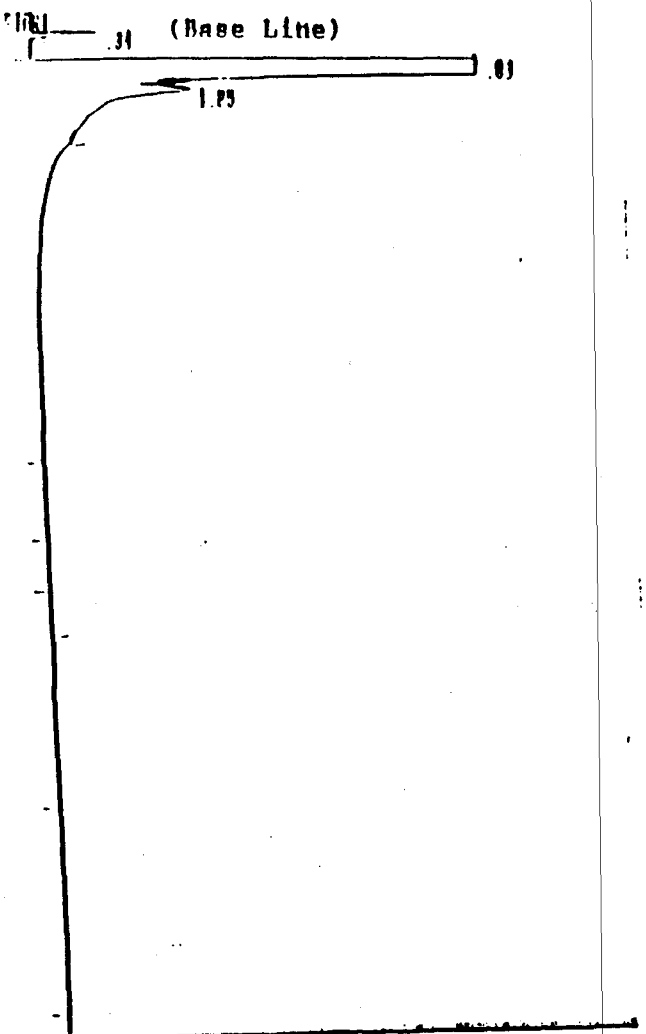
TEST RESULTS:

| | VISUAL APPEARANCE | | CHEMICAL ANALYSIS | | | | |
|---|---|---|---|---|---|---|---|
| AGING TEMP. °C, (°F) | OIL COLOR CHANGE (ASTM) | OTHER VISUAL CHANGES | GC | F ION (UG) | %R-125 REACTED | OIL ACID NO. | OIL IR (DIFFERENTIAL SCAN) |
| 150 (300) | FROM 2.5 TO 3.0 | STEEL: NO CHANGE | NO CHANGE | 8.2 | 0.005 | 0.00 | NO SIGNIFICANT CHANGES |
| 175 (347) | FROM 2.5 TO 3.0 | STEEL: NO CHANGE | NO CHANGE | 7.7 | 0.005 | 0.48 | NO SIGNIFICANT CHANGES |
| 200 (392) | FROM 2.5 TO 3.0 | STEEL: NO CHANGE | NO CHANGE | 7.7 | 0.005 | 1.09 | CHANGES IN 1200--1300 $CM^{-1}$ REGION |

(FLUORIDE ANALYSIS BLANK: 3.1)

FIGURE A-15

R-125 "As Received"
(Base Line)



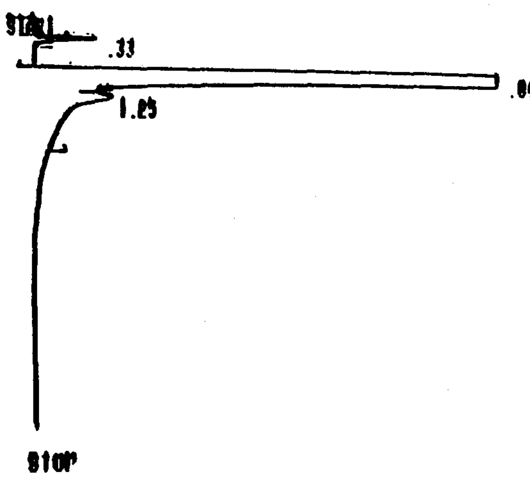

RUN # 25 JAN/17/92 14:49:04

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 3599 | BB | 0.050 | 0.106 |
| 0.03 | 3390200 | PB | 0.117 | 99.894 |
| 1.25 | 0 | BB | 0.000 | 0.000 |

TOTAL AREA= 3393800
MUL FACTOR= 1.0000E+00

Tube CH-01
R-125/PE Ester (mixed acid I)
14 Days @ 150°C




RUN # 9 MAR/04/92 09:52:28

AREA%

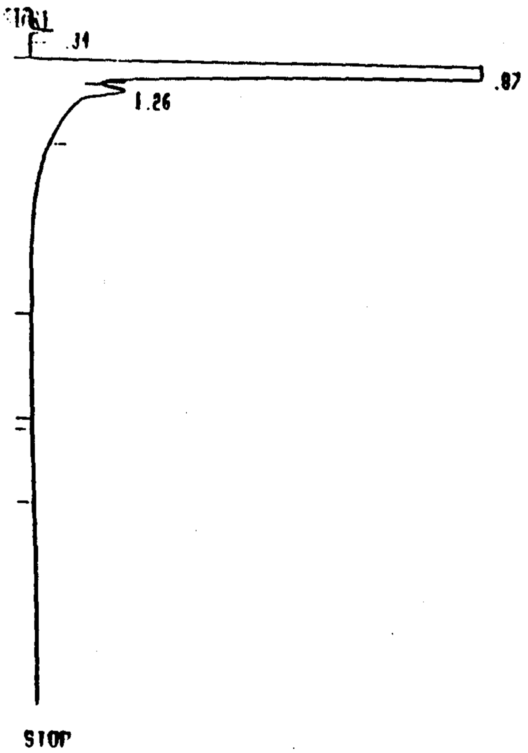
| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 3291 | BB | 0.051 | 0.221 |
| 0.06 | 1403400 | PB | 0.100 | 99.779 |
| 1.25 | 0 | BB | 0.000 | 0.000 |

TOTAL AREA= 1406700
MUL FACTOR= 1.0000E+00

FIGURE A-16

Tube CH-11 R-125/PE Ester (mixed acid I)

14 Days @ 175°C




RUN # 11 MAR/04/92 10:34:15

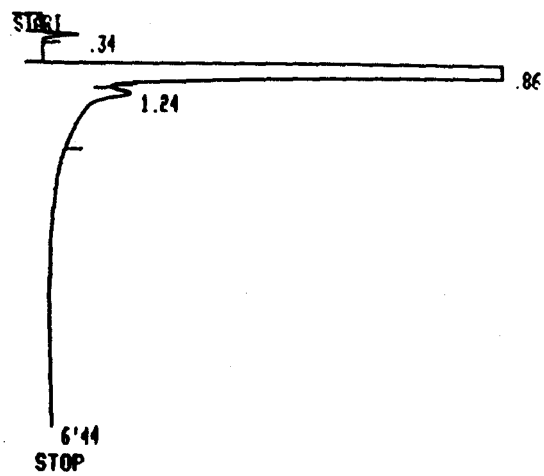
AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 1314 | BB | 0.054 | 0.080 |
| 0.87 | 1647800 | PB | 0.102 | 99.917 |
| 1.26 | 50 | BB | 0.002 | 0.003 |

TOTAL AREA= 1649200
MUL FACTOR= 1.0000E+00

Tube CH-21
R-125/PE Ester (mixed acid I)

14 Days @ 200°C




RUN # 6 APR/01/92 10:36:21

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 1900 | BB | 0.051 | 0.121 |
| 0.86 | 1569900 | PB | 0.101 | 99.879 |
| 1.24 | 0 | BB | 0.000 | 0.000 |

TOTAL AREA= 1571800
MUL FACTOR= 1.0000E+00

TABLE A-9: STABILITY OF R-134A WITH POLYPROPYLENE GLYCOL (BUTYL MONOETHER)

SEALED TUBE CONTENTS:

VALVE STEEL STRIP
1.0 cc (1.0 G) OF TEST LUBRICANT
1.0 G OF R-134A

AGING SCHEDULE:

14 DAYS AT TEMPERATURES SHOWN

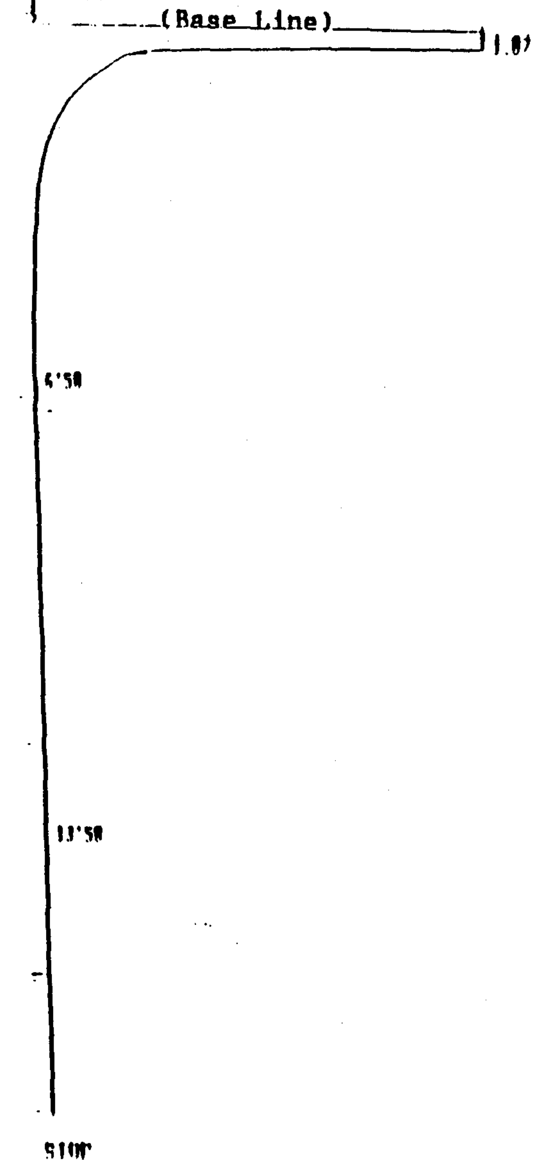
TEST RESULTS:

| | VISUAL APPEARANCE | | CHEMICAL ANALYSIS | | | |
|---|---|---|---|---|---|---|
| AGING TEMP °C (°F) | OIL COLOR CHANGE (ASTM) | OTHER VISUAL CHANGES | GC | F ION (UG) | %R-134A REACTED | SIZE EXCLUSION CHROMATOGRAPHY OF OIL |
| 150 (300) | NO CHANGE AT 2.5 | STEEL: NO CHANGE | NO CHANGE | 1.2 | 0.001 | CHANGES IN PAG STRUCTURE |
| 175 (347) | NO CHANGE AT 2.5 | STEEL: NO CHANGE | NO CHANGE | 1.2 | 0.001 | CHANGES IN PAG STRUCTURE |
| 200 (392) | FROM 2.5 TO 3.0 | STEEL: DARKENED | NO CHANGE | 1.1 | 0.001 | CHANGES IN PAG STRUCTURE |

(FLUORIDE ANALYSIS BLANK: 3.1)

FIGURE A-17

R-134a "As Received"




RUN # 11 JAN/16/92 14:02:49

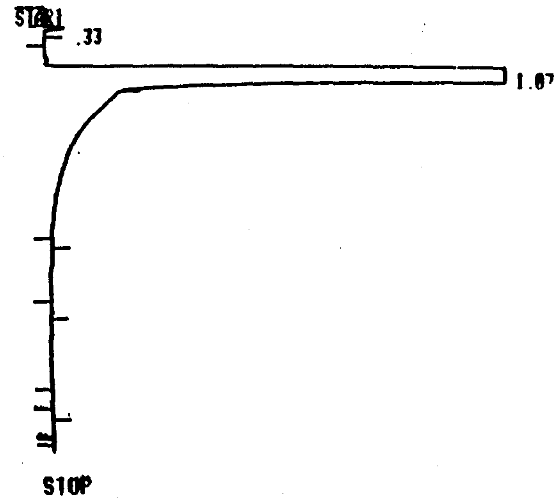
AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 1812 | BB | 0.052 | 0.065 |
| 1.07 | 2000200 | PB | 0.140 | 99.935 |

TOTAL AREA= 2002009
MUL FACTOR= 1.0000E+00

Tube CI-01 R134a/PPG (Butyl-monoether)

14 Days @ 150°C




RUN # 7 APR/15/92 11:33:09

AREA%

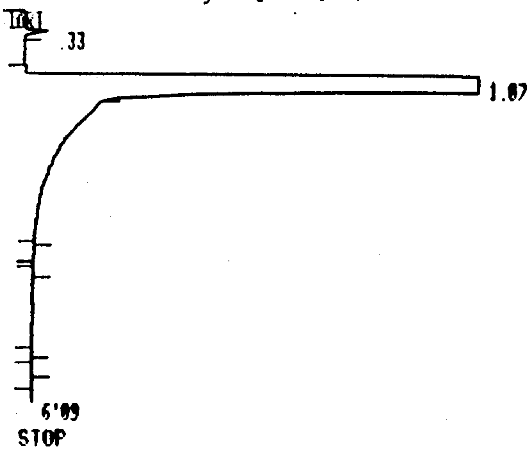
| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 986 | PB | 0.049 | 0.056 |
| 1.07 | 1748900 | PB | 0.124 | 99.944 |

TOTAL AREA= 1749800
MUL FACTOR= 1.0000E+00

FIGURE A-18

Tube CI-11 R-134a/PPG (butyl monoether)

14 Days @ 175°C




RUN # 8 APR/15/92 11:51:51

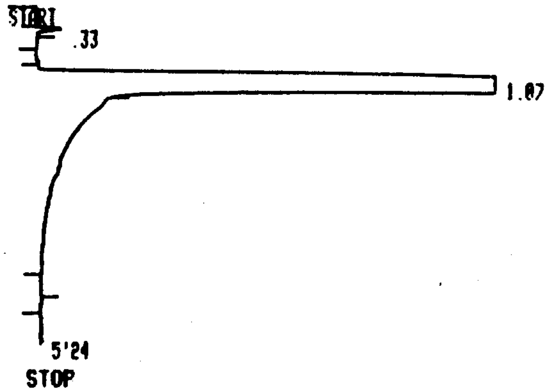
AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 1229 | BB | 0.050 | 0.071 |
| 1.07 | 1731800 | PB | 0.124 | 99.929 |

TOTAL ARE 1733000
MUL FACTO .0000E+00

Tube CI-23

14 Days @ 200°C




RUN # 12 APR/15/92 14:29:46

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 1226 | BB | 0.051 | 0.071 |
| 1.07 | 1732200 | PB | 0.125 | 99.929 |

TOTAL AREA= 1733400
MUL FACTOR= 1.0000E+00

TABLE A-10: STABILITY OF R-134A WITH POLYPROPYLENE GLYCOL DIOL

SEALED TUBE CONTENTS:

VALVE STEEL STRIP
1.0 cc (1.0 G) OF TEST LUBRICANT
1.0 G OF R-134A

AGING SCHEDULE:

14 DAYS AT TEMPERATURES SHOWN

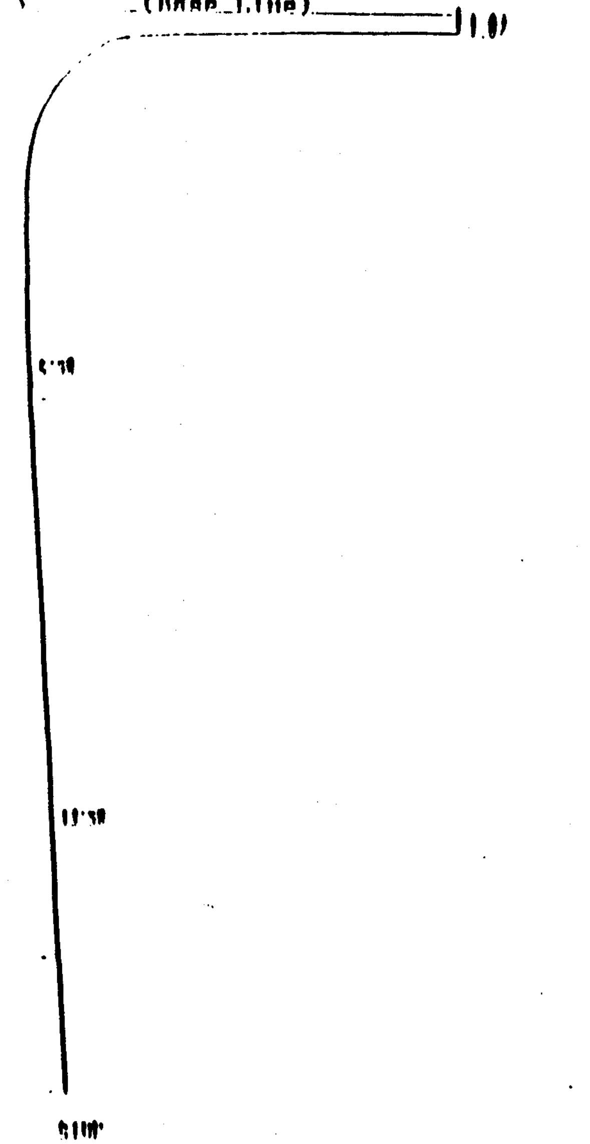
TEST RESULTS:

| | VISUAL APPEARANCE | | CHEMICAL ANALYSIS | | | |
|---|---|---|---|---|---|---|
| AGING TEMP. °C, (°F) | OIL COLOR CHANGE (ASTM) | OTHER VISUAL CHANGES | GC | F ION (UG) | %R-134A REACTED | SIZE EXCLUSION CHROMATOGRAPHY OF OIL |
| 150 (300) | NO CHANGE AT 2.0 | STEEL: NO CHANGE | NO CHANGE | 0.6 | 0.000 | SIGNIFICANT CHANGES IN PAG STRUCTURE |
| 175 (347) | NO CHANGE AT 2.0 | STEEL: NO CHANGE | NO CHANGE | 2.1 | 0.001 | SIGNIFICANT CHANGES IN PAG STRUCTURE |
| 200 (392) | FROM 2.0 TO 2.5 | STEEL: NO CHANGE | NO CHANGE | 0.1 | 0.000 | SIGNIFICANT CHANGES IN PAG STRUCTURE |

(FLUORIDE ANALYSIS BLANK: 8.1)

FIGURE A-19

R-134a "As Received"
(Base Line)



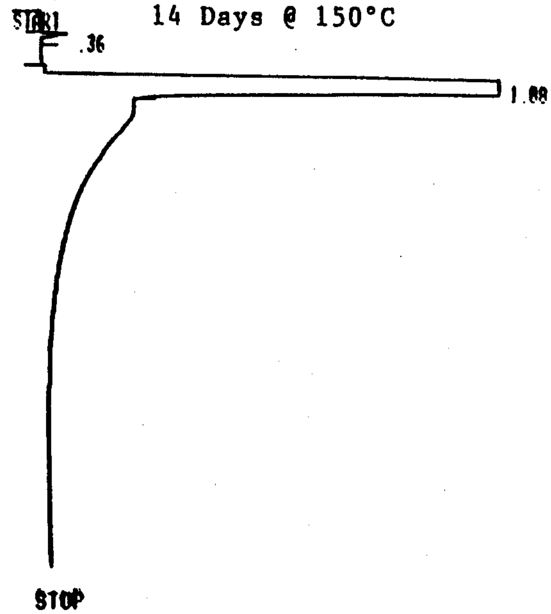
RUN # 11 JAN/16/92 14:02:49

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| [illegible] | [illegible] | PB | 0.140 | 99.939 |

Tube CJ-01 R-134a/Polypropylene-glycol Diol

14 Days @ 150°C




RUN # 4 JUN/24/92 13:35:10

AREA%

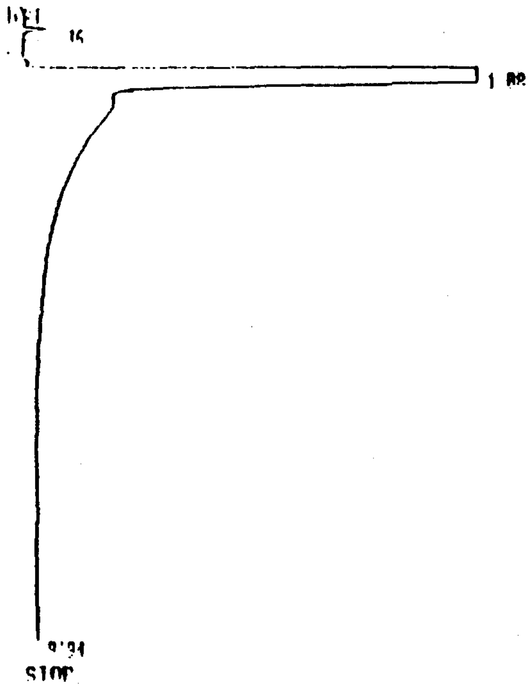
| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.36 | 1456 | PB | 0.055 | 0.076 |
| 1.00 | 1911700 | PB | 0.121 | 99.924 |

TOTAL AREA= 1913200
MUL FACTOR= 1.0000E+00

FIGURE A-20

Gas Chromatograms R-134a/Polypropyleneglycol Diol

Tube CJ-11 (14 Days @175°C)



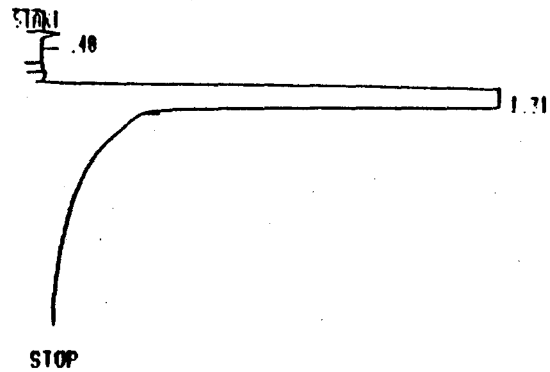

RUN # 5 JUN/24/92 14:04:52

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.36 | 1332 | PB | 0.055 | 0.070 |
| 1.00 | 1915100 | PB | 0.121 | 99.930 |

TOTAL AREA= 1916400
MUL FACTOR= 1.0000E+00

Tube CJ-21 (14days @ 200°C)




RUN # 2 JUL/23/92 14:13:21

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.40 | 1107 | PB | 0.063 | 0.053 |
| 1.31 | 2074700 | PB | 0.146 | 99.947 |

TOTAL AREA= 2075800
MUL FACTOR= 1.0000E+00

TABLE A-11: STABILITY OF R-134A WITH MODIFIED POLYGLYC0L

SEALED TUBE CONTENTS:

VALVE STEEL STRIP
1.0 cc (1.0 G) OF TEST LUBRICANT
1.0 G OF R-134A

AGING SCHEDULE:

14 DAYS AT TEMPERATURES SHOWN

TEST RESULTS:

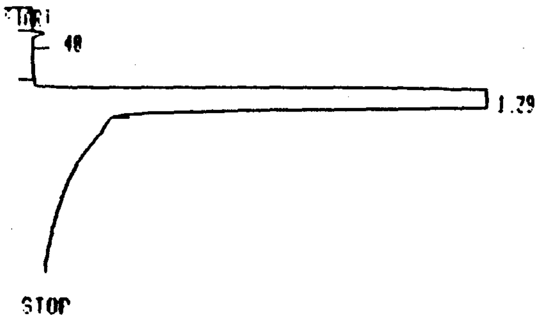
| | VISUAL APPEARANCE | | CHEMICAL ANALYSIS | | | |
|---|---|---|---|---|---|---|
| AGING TEMP. °C, (°F) | OIL COLOR CHANGE (ASTM) | OTHER VISUAL CHANGES | GC | F ION (UG) | %R-134A REACTED | SIZE EXCLUSION CHROMATOGRAPHY OF OIL |
| 150 (300) | FROM 2.5 TO 3.0 | STEEL: NO CHANGE | ELUENT AT 0.08% AT 1.0 MIN. | 1.1 | 0.001 | SIGNIFICANT CHANGES IN PAG STRUCTURE |
| 175 (347) | FROM 2.5 TO 3.5 | STEEL: NO CHANGE | 0.05% AT 1.0 MIN. | 2.3 | 0.001 | SIGNIFICANT CHANGES IN PAG STRUCTURE |
| 200 (392) | FROM 2.5 TO 4.0 | STEEL: NO CHANGE | TRACE OF ELUENT AT 1.03 MIN. | 2.2 | 0.001 | SIGNIFICANT CHANGES IN PAG STRUCTURE |

(FLUORIDE ANALYSIS BLANK: 8.2)

FIGURE A-21

Gas Chromatograms R-134a/Modified Polyglycol

R-134a "As Received"



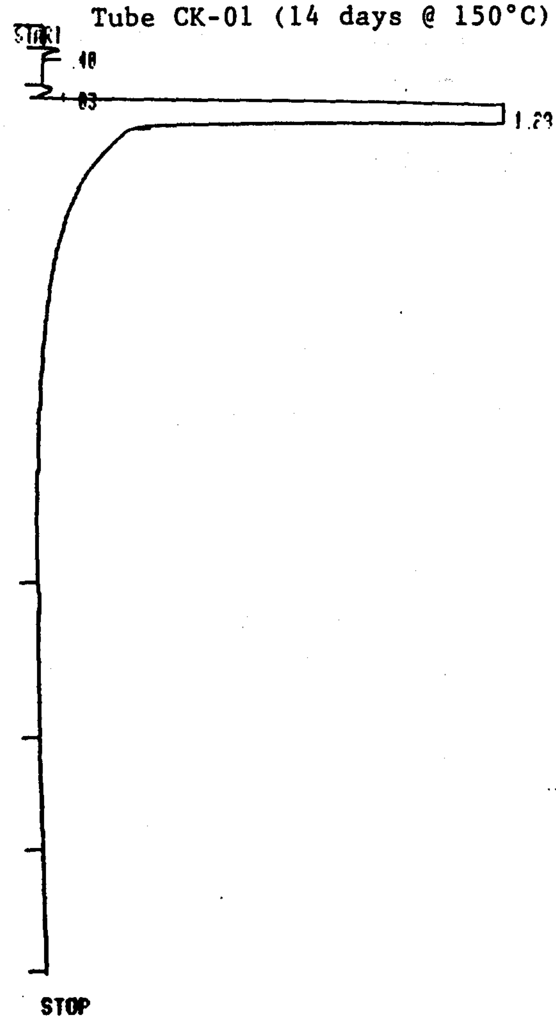

RUN # 2 AUG/26/92 10:30:23

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.40 | 837 | BB | 0.064 | 0.050 |
| 1.39 | 1660100 | PB | 0.148 | 99.950 |

TOTAL AREA= 1661000
MUL FACTOR= 1.0000E+00

Tube CK-01 (14 days @ 150°C)




RUN # 21 JUL/22/92 12:16:37

AREA%

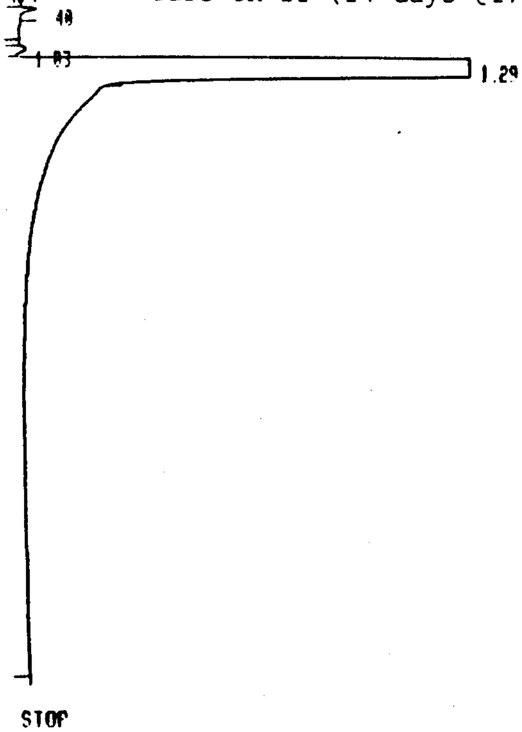
| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.40 | 1000 | BB | 0.063 | 0.048 |
| 1.03 | 1745 | BP | 0.124 | 0.083 |
| 1.28 | 2094200 | PB | 0.144 | 99.869 |

TOTAL AREA= 2097000
MUL FACTOR= 1.0000E+00

FIGURE A-22

Gas Chromatograms R-134a/Modified Polyglycol

Tube CK-11 (14 days @175°C)



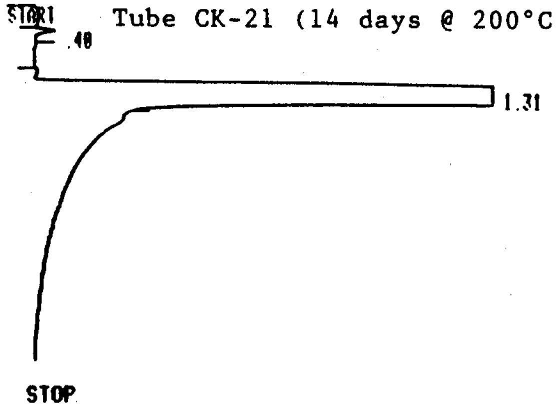

RUN # 22 JUL/22/92 13:55:16

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.40 | 983 | PB | 0.057 | 0.049 |
| 1.03 | 1070 | BP | 0.103 | 0.053 |
| 1.29 | 2011300 | PB | 0.143 | 99.898 |

TOTAL AREA= 2013400
MUL FACTOR= 1.0000E+00

Tube CK-21 (14 days @ 200°C)




RUN # 3 JUL/23/92 15:09:08

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.40 | 1044 | PB | 0.056 | 0.049 |
| 1.31 | 2133200 | PB | 0.146 | 99.951 |

TOTAL AREA= 2134200
MUL FACTOR= 1.0000E+00

TABLE A-12: STABILITY OF R-134A WITH PENTAERYTHRITOL ESTER (MIXED ACID II)

SEALED TUBE CONTENTS

VALVE STEEL STRIP
1.0 cc (1.0 G) OF TEST LUBRICANT
1.0 G OF R-134A

AGING SCHEDULE:

14 DAYS AT TEMPERATURES SHOWN

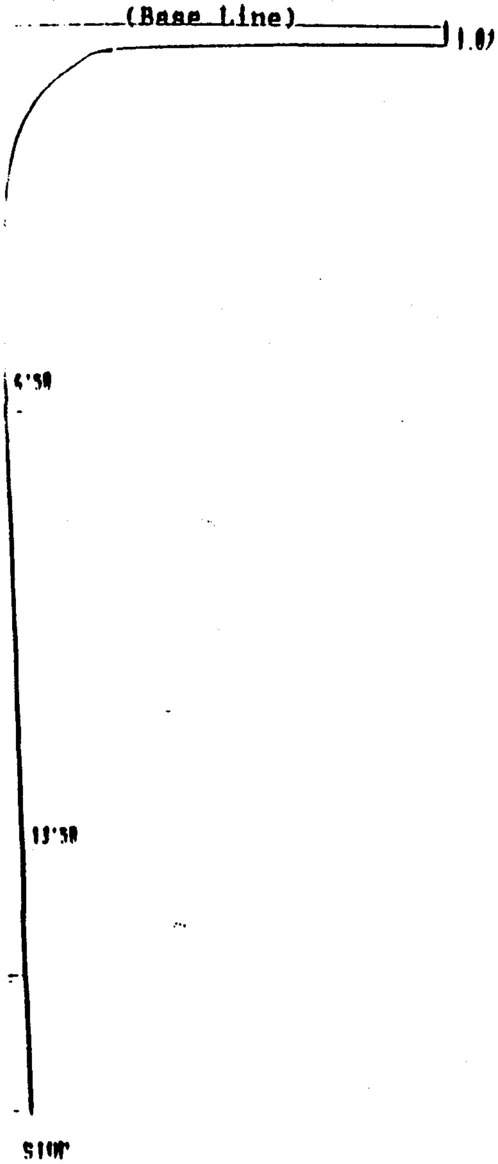
TEST RESULTS:

| | VISUAL APPEARANCE | | CHEMICAL ANALYSIS | | | | |
|---|---|---|---|---|---|---|---|
| AGING TEMP. °C, (°F) | OIL COLOR CHANGE (ASTM) | OTHER VISUAL CHANGES | GC | F ION (UG) | %R-134A REACTED | OIL ACID NO. | OIL IR (DIFFERENTIAL SCAN) |
| 150 (300) | NO CHANGE AT 2.5 | STEEL: NO CHANGE | NO CHANGE | 0.8 | 0.000 | 0.00 | NO CHANGE |
| 175 (347) | NO CHANGE AT 2.5 | STEEL: LIGHT STAIN | NO CHANGE | 0.8 | 0.000 | 0.00 | NO CHANGE |
| 200 (392) | FROM 2.5 TO 3.0 | STEEL: DARKENED | NO CHANGE | 0.7 | 0.000 | 0.67 | NO CHANGE |

(FLUORIDE ANALYSIS BLANK: 3.1)

FIGURE A-23

R-134a "As Received"
(Base Line)




RUN # 11 JAN/16/92 14:02:49

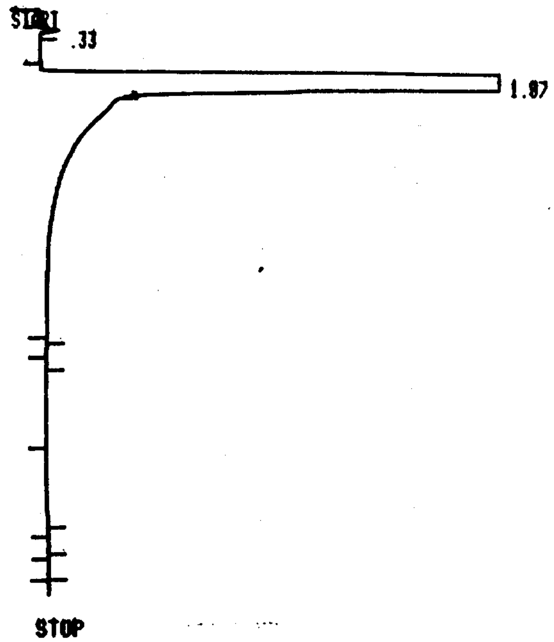
AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 1012 | BB | 0.098 | 0.065 |
| 1.07 | 2000200 | PB | 0.140 | 99.935 |

TOTAL AREA= 2002009
MUL FACTOR= 1.0000E+00

Tube CL-01; R-134a/PE Ester (mixed acid II)

14 Days @ 150°C




RUN # 9 APR/15/92 13:25:09

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 1023 | BB | 0.052 | 0.059 |
| 1.07 | 1749100 | PB | 0.125 | 99.942 |

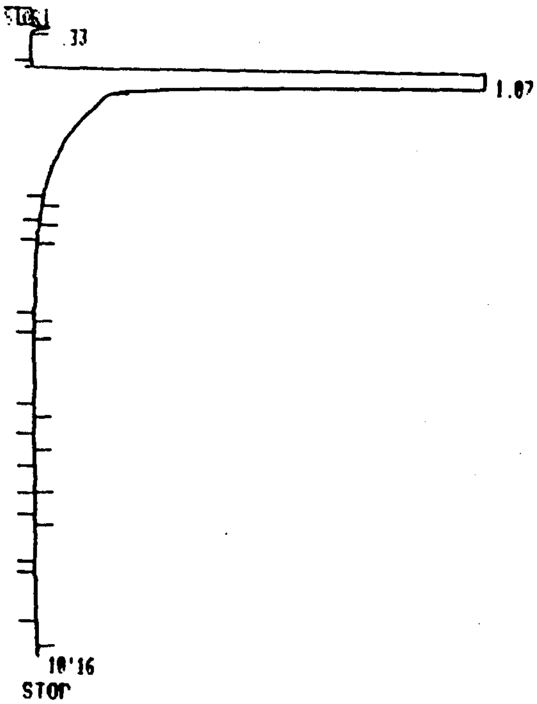
TOTAL AREA= 1750100
MUL FACTOR= 1.0000E+00

FIGURE A-24

R-134a/PE Ester (mixed acid II)

Tube CL-12

14 Days @ 175°C




RUN # 10 APR/15/92 13:44:57

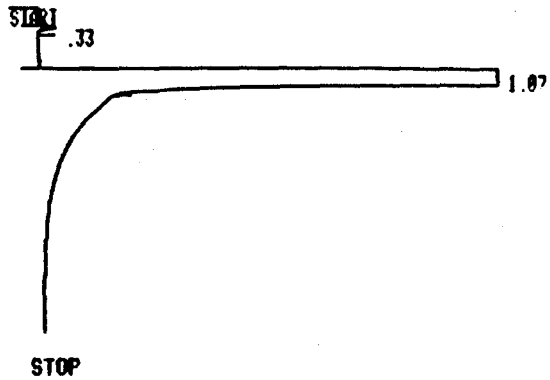
AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 1006 | BB | 0.053 | 0.057 |
| 1.07 | 1779400 | PB | 0.125 | 99.943 |

TOTAL AREA= 1780400
MUL FACTOR= 1.0000E+00

Tube CL-21

14 Days @ 200°C




RUN # 11 APR/15/92 14:06:08

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 970 | BB | 0.051 | 0.055 |
| 1.07 | 1772500 | PB | 0.125 | 99.945 |

TOTAL AREA= 1773400
MUL FACTOR= 1.0000E+00

TABLE A-13: STABILITY OF R-134A WITH PENTAERYTHRITOL ESTER (MIXED ACID I)

SEALED TUBE CONTENTS:

VALVE STEEL STRIP
1.0 cc (1.0 G) OF TEST LUBRICANT
1.0 G OF R-134A

AGING SCHEDULE:

14 DAYS AT TEMPERATURES SHOWN

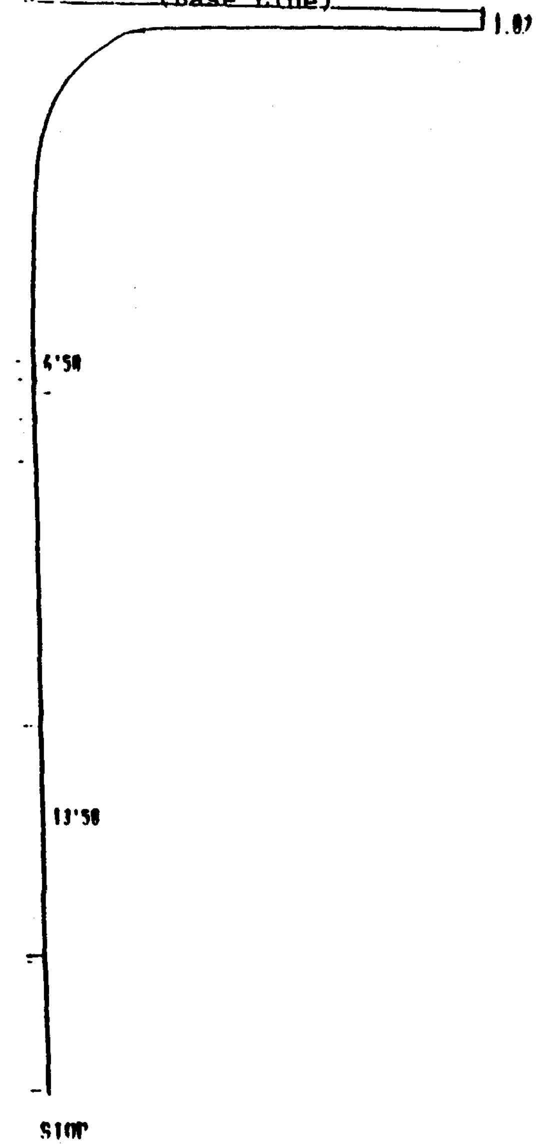
TEST RESULTS:

| | VISUAL APPEARANCE | | CHEMICAL ANALYSIS | | | | |
|---|---|---|---|---|---|---|---|
| AGING TEMP. °C, (°F) | OIL COLOR CHANGE (ASTM) | OTHER VISUAL CHANGES | GC | F ION (UG) | %R-134A REACTED | OIL ACID NO. | OIL IR (DIFFERENTIAL SCAN) |
| 150 (300) | NO CHANGE AT 2.5 | STEEL: NO CHANGE | NO CHANGE | 2.5 | 0.001 | 0.00 | NO CHANGE |
| 175 (347) | NO CHANGE AT 2.5 | STEEL: NO CHANGE | NO CHANGE | 1.4 | 0.001 | 0.00 | NO CHANGE |
| 200 (392) | FROM 2.5 TO 3.0 | STEEL: NO CHANGE | NO CHANGE | 1.8 | 0.001 | 0.74 | NO CHANGE |

(FLUORIDE ANALYSIS BLANK: 3.1)

FIGURE A-25

R-134a "As Received"
(Base Line)




RUN # 11 JAN/16/92 14:02:49

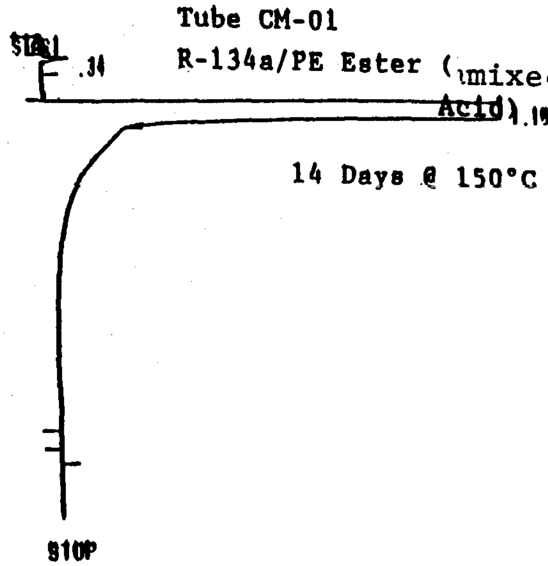
AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 1812 | BB | 0.052 | 0.065 |
| 1.07 | 2800200 | PB | 0.140 | 99.935 |

TOTAL AREA= 2802009
MUL FACTOR= 1.0000E+00

Tube CM-01
R-134a/PE Ester (mixed acid I)
~~Acid)~~

14 Days @ 150°C




RUN # 13 MAR/04/92 11:10:47

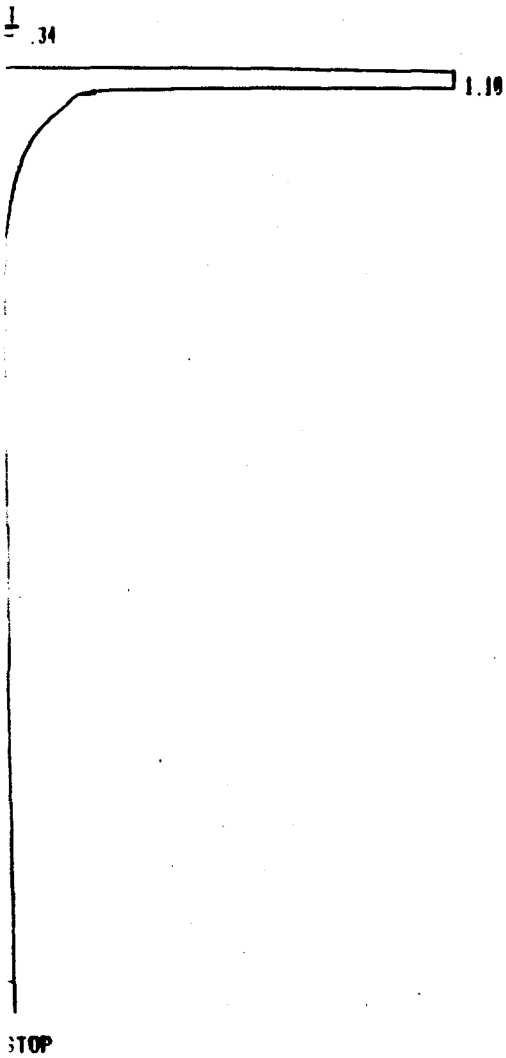
AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 1303 | BB | 0.052 | 0.073 |
| 1.10 | 1807700 | PB | 0.120 | 99.927 |

TOTAL AREA= 1809100
MUL FACTOR= 1.0000E+00

FIGURE A-26

Tube CM-11
R-134a/PE Ester (mixed acid I)
14 Days @ 175°C

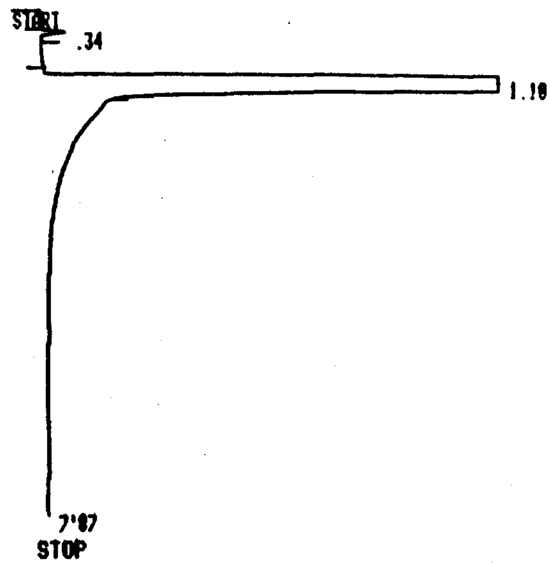



RUN # 14 MAR/04/92 12:07:46

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 1221 | BB | 0.054 | 0.066 |
| 1.10 | 1853200 | PB | 0.128 | 99.934 |

Tube CM-21
R-134a/PE Ester (mixed acid I)
14 Days @ 200°C




RUN # 7 APR/01/92 11:11:11

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 1273 | BB | 0.052 | 0.081 |
| 1.10 | 1562200 | PB | 0.125 | 99.919 |

TOTAL AREA= 1563500
MUL FACTOR= 1.0000E+00

TABLE A-14: STABILITY OF R-134A WITH PENTAERYTHRITOL ESTER (100 cSt)

SEALED TUBE CONTENTS:

VALVE STEEL STRIP
1.0 cc (1.0 G) OF TEST LUBRICANT
1.0 G OF R-134A

AGING SCHEDULE:

14 DAYS AT TEMPERATURES SHOWN

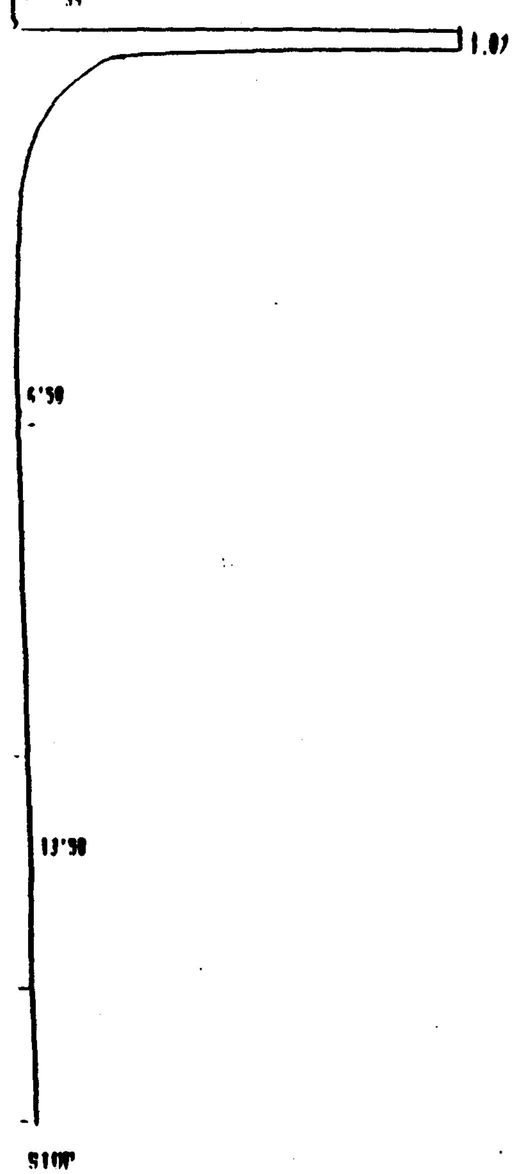
TEST RESULTS:

| | VISUAL APPEARANCE | | CHEMICAL ANALYSIS | | | | |
|---|---|---|---|---|---|---|---|
| AGING TEMP. °C (°F) | OIL COLOR CHANGE (ASTM) | OTHER VISUAL CHANGES | GC | F ION (UG) | %R-134A REACTED | OIL ACID NO. | OIL IR (DIFFERENTIAL SCAN) |
| 150 (300) | NO CHANGE AT 3.0 | STEEL: NO CHANGE | NO CHANGE | 1.6 | 0.001 | 0.94 | SPLIT C=O AT 1700 To 1750 $CM^{-1}$ |
| 175 (347) | FROM 3.0 TO 3.5 | STEEL: DARKENED | NO CHANGE | 1.0 | 0.001 | 0.63 | SPLIT C=O 1700 To 1750 $CM^{-1}$ |
| 200 (392) | FROM 3.0 TO 4.5 | STEEL: BLACKENED | 0.56% OF $CO_2$ FORMED | 6.8 | 0.004 | 0.99 | AS ABOVE, PLUS C-O ABSORPTION AROUND 1200 $CM^{-1}$ |

COMMENTS ON IR ANALYSIS: SPLIT CARBONYL BANDS INDICATE FORMATION OF EITHER NEW ACID MOLECULES (TRANSESTERIFICATION), OR ALDEHYDES AND KETONES, I.E. DESTRUCTION OF ESTER LINKAGES. AT 200 C ALCOHOLS ARE FORMED (C-O ABSORPTION) TOGETHER WITH THE APPEARANCE OF $CO_2$ (FROM GC ANALYSIS). THIS INDICATES THE BREAKAGE OF ESTER LINKAGES BY A DECARBOXYLATION MECHANISM.

FIGURE A-27

R-134a "As Received"




RUN # 11 JAN/16/92 14:02:49

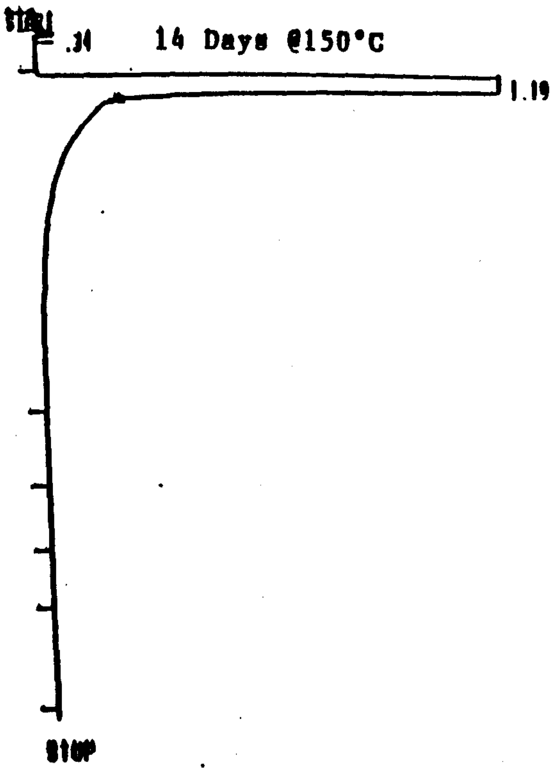
AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 1012 | BB | 0.092 | 0.063 |
| 1.07 | 2000200 | PB | 0.140 | 99.935 |

TOTAL AREA= 2092009
MUL FACTOR= 1.0000E+00

Tube CN-01 R134a/PE (100 cSt)

14 Days @150°C




RUN # 19 MAR/04/92 13:32:23

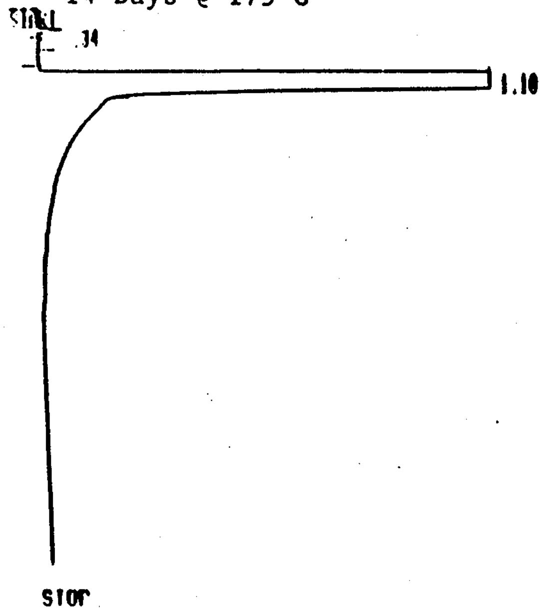
AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 1014 | BB | 0.051 | 0.055 |
| 1.10 | 1049000 | PB | 0.120 | 99.945 |

TOTAL AREA= 1050000
MUL FACTOR= 1.0000E+00

FIGURE A-28

Tube CN-11
R-134a/PE Ester (100 cSt)
14 Days @ 175°C



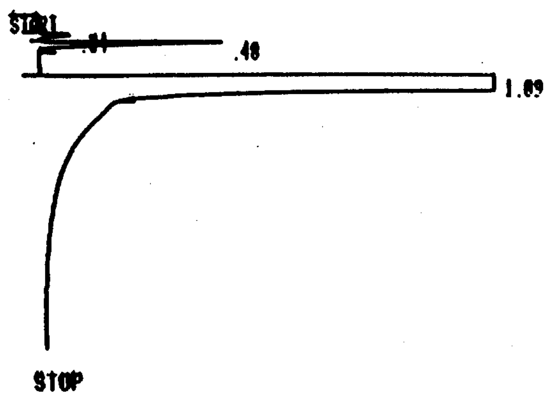

RUN # 16 MAR/04/92 14:47:44

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 1363 | BP | 0.053 | 0.072 |
| 1.10 | 1896000 | PB | 0.129 | 99.928 |

TOTAL AREA= 1897400
MUL FACTOR= 1.0000E+00

Tube CN-22
R134a/PE Ester (100 cSt)
14 Days @200°C




RUN # 8 APR/01/92 11:38:40

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 1627 | PP | 0.050 | 0.089 |
| 0.48 | 10123 | PB | 0.052 | 0.555 |
| 1.09 | 1811800 | PB | 0.127 | 99.356 |

TOTAL AREA= 1823500
MUL FACTOR= 1.0000E+00

TABLE A-15: STABILITY OF R-142B WITH ALKYLBENZENE OIL

SEALED TUBE CONTENTS:

VALVE STEEL STRIP
1.0 CC (0.9 G) OF TEST LUBRICANT
0.9 G OF R-142B

AGING SCHEDULE:

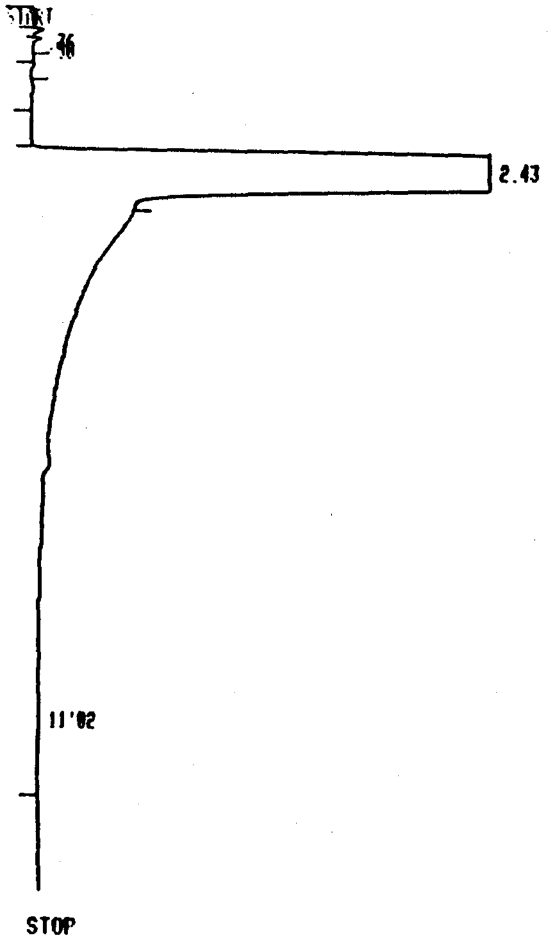
14 DAYS AT TEMPERATURES SHOWN

TEST RESULTS:

| | VISUAL APPEARANCE OF TUBES | | CHEMICAL ANALYSIS | | |
|---|---|---|---|---|---|
| AGING TEMP, °C (°F) | OIL COLOR CHANGE (ASTM) | OTHER VISUAL OBSERVATIONS | GC | CL ION (UG) | %R-142B REACTED |
| 150 (300) | NO CHANGE AT 2.0 | STEEL: NO CHANGE | NO CHANGE | 34.8 | 0.011 |
| 175 (347) | FROM 2.0 TO 2.5 | STEEL: SLIGHT DARKENING | NO CHANGE | 45.0 | 0.014 |
| 200 (392) | FROM 2.0 TO 3.0 | STEEL: ETCHED AND STAINED | NO MEASURABLE CHANGE | 525 | 0.169 |

FIGURE A-29

R-142b "As Received"




RUN # 8 JUN/11/92 08:42:05

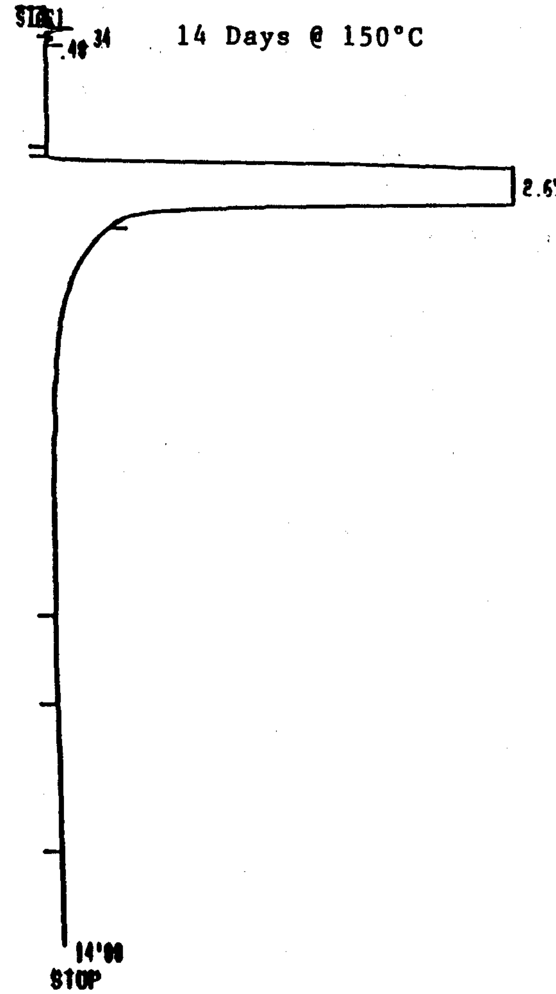
AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.36 | 635 | BP | 0.050 | 0.025 |
| 0.50 | 555 | PB | 0.054 | 0.022 |
| 2.43 | 2519200 | PB | 0.286 | 99.953 |

TOTAL AREA= 2520400
MUL FACTOR= 1.0000E+00

Tube CO-02 R-142b/Alkylbenzene

14 Days @ 150°C




RUN # 9 MAR/10/92 14:45:31

AREA%

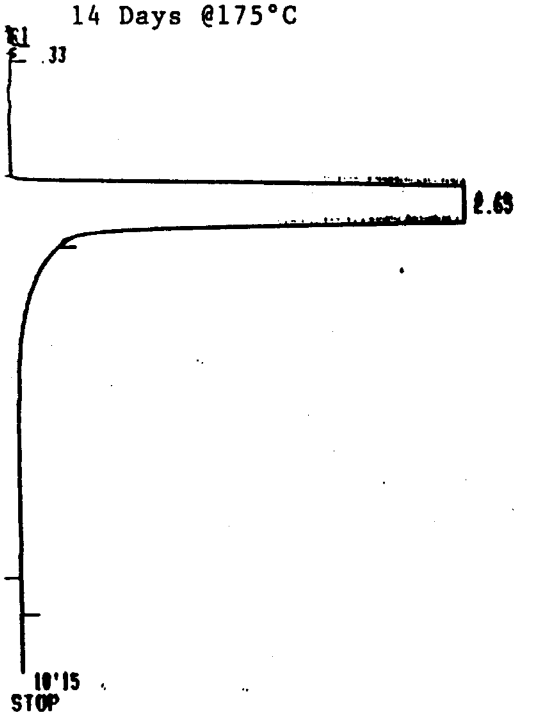
| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 1507 | BP | 0.052 | 0.082 |
| 0.48 | 403 | PB | 0.049 | 0.022 |
| 2.65 | 1029700 | PB | 0.313 | 99.896 |

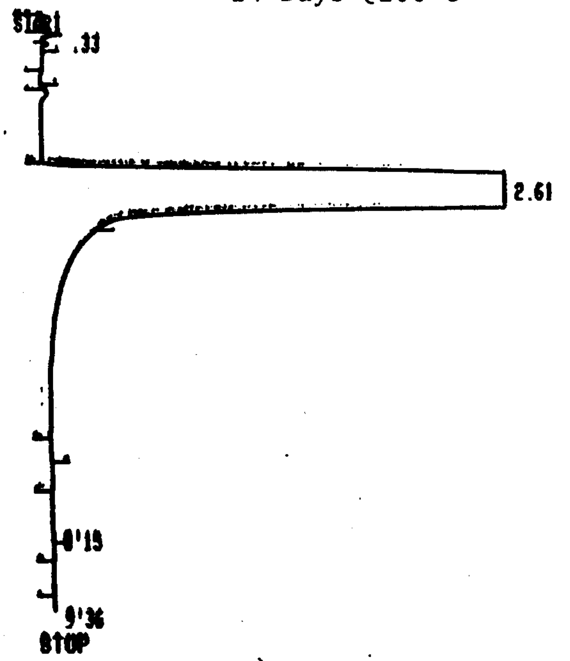
TOTAL AREA= 1031600
MUL FACTOR= 1.0000E+00

FIGURE A-30

Tube CO-11 | R-142b/Alkylbenzene | Tube CO-21

14 Days @175°C | | 14 Days @200°C




RUN # 10 MAR/10/92 15:34:04

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 1116 | BP | 0.053 | 0.062 |
| 2.65 | 1799500 | PB | 0.314 | 99.938 |

TOTAL AREA= 1800600
MUL FACTOR= 1.0000E+00

RUN # 17 APR/15/92 16:29:43

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 1034 | BP | 0.052 | 0.059 |
| 2.61 | 1753000 | PB | 0.309 | 99.941 |

TOTAL AREA= 1754000
MUL FACTOR= 1.0000E+00

TABLE A-16: STABILITY of R-143A WITH PENTAERYTHRITOL ESTER (MIXED ACID I)

SEALED TUBE CONTENTS:

VALVE STEEL STRIP
1.0 cc (1.0 G) OF TEST LUBRICANT
1.0 G OF R-143A

AGING SCHEDULE:

14 DAYS AT TEMPERATURES SHOWN

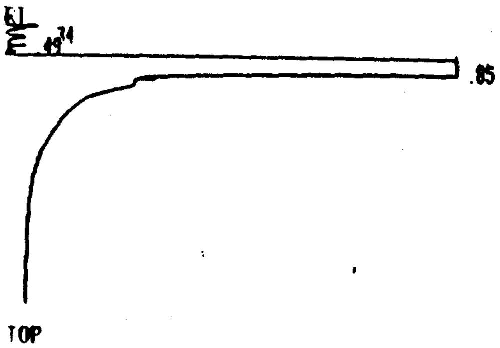
TEST RESULTS:

| | VISUAL APPEARANCE | | CHEMICAL ANALYSIS | | | | |
|---|---|---|---|---|---|---|---|
| AGING TEMP. °C, (°F) | OIL COLOR CHANGE (ASTM) | OTHER VISUAL CHANGES | GC | F ION (UG) | %R-143A REACTED | OIL ACID NO. | OIL IR (DIFFEREN-TIAL SCAN) |
| 150 (300) | FROM 2.5 TO 3.0 | STEEL: NO CHANGE | NO CHANGE | 2.6 | 0.001 | 0.51 | NO CHANGE |
| 175 (347) | FROM 2.5 TO 3.0 | STEEL: NO CHANGE | NO CHANGE | 4.1 | 0.002 | 1.07 | NO CHANGE |
| 200 (392) | FROM 2.5 TO 3.0 | STEEL: BLACKENED | NO CHANGE | 1.1 | 0.001 | 0.99 | NO CHANGE |

(FLUORIDE ANALYSIS BLANK: 3.6)

FIGURE A-31

R-143a "As Received"




JN # 1 FEB/19/92 08:44:18

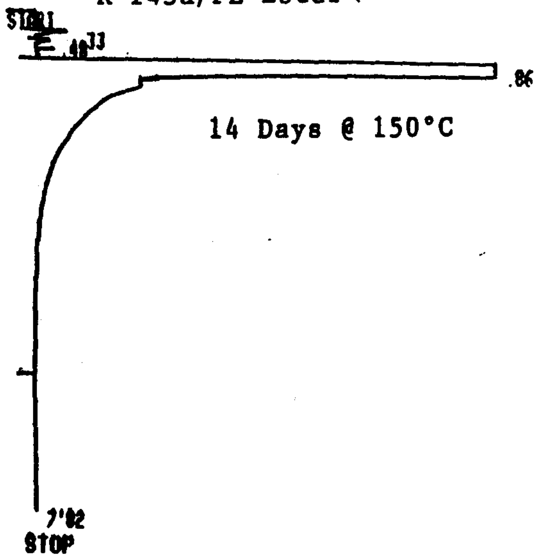
REA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 1611 | BP | 0.049 | 0.052 |
| 0.49 | 1064 | PB | 0.057 | 0.035 |
| 0.85 | 3070100 | PB | 0.114 | 99.913 |

TAL AREA= 3072800
L FACTOR= 1.0000E+00

Note: 0.49 min peak is probably $CO_2$.

Tube CP-02
R-143a/PE Ester (mixed acid I)




RUN # 7 MAR/10/92 13:46:21

AREA%

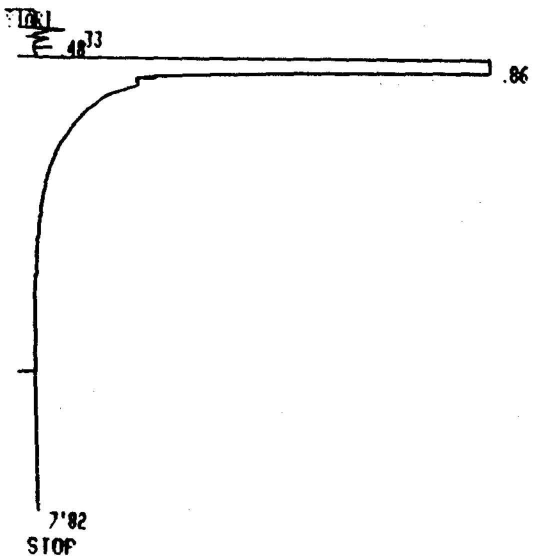
| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 1674 | BP | 0.051 | 0.082 |
| 0.48 | 757 | PB | 0.049 | 0.037 |
| 0.86 | 2050000 | PB | 0.103 | 99.882 |

TOTAL AREA= 2052400
MUL FACTOR= 1.0000E+00

FIGURE A-32

Tube CP-11 R-143a/PE Ester (mixed acid I) Tube CP-21

14 Days @ 175°C



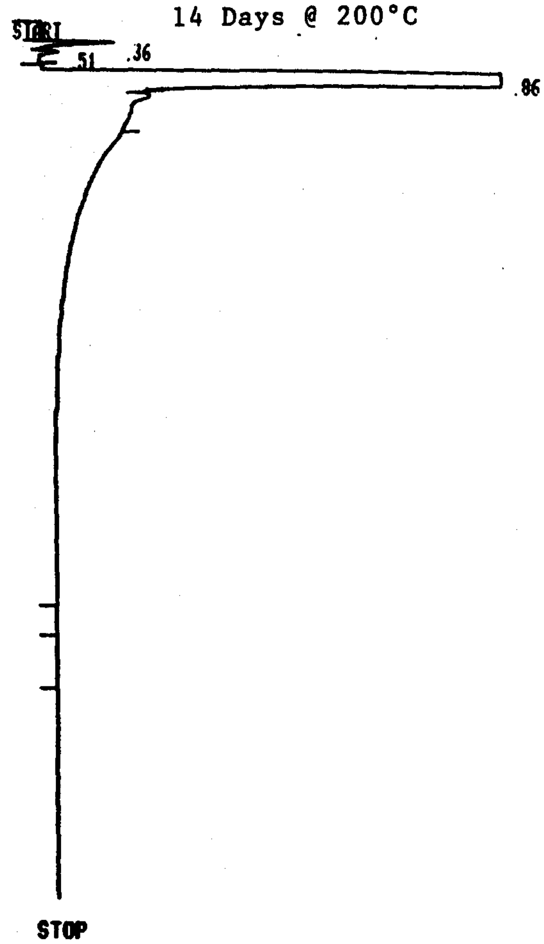

RUN # 7 MAR/18/92 13:46:21

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 1674 | BP | 0.051 | 0.082 |
| 0.48 | 757 | PB | 0.049 | 0.037 |
| 0.86 | 205[illegible] | PB | 0.103 | 99.882 |

TOTAL AREA= 2052400
MUL FACTOR= 1.0000E+00

14 Days @ 200°C




RUN # 13 JUN/11/92 11:09:11

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.36 | 4126 | PV | 0.053 | 0.189 |
| 0.51 | 1024 | VB | 0.053 | 0.047 |
| 0.86 | 2182000 | PB | 0.101 | 99.765 |

TOTAL AREA= 2187100
MUL FACTOR= 1.0000E+00

TABLE A-17: STABILITY OF R-152A WITH ALKYLBENZENE

SEALED TUBE CONTENTS:

VALVE STEEL STRIP
1.0 CC (0.9 G) OF TEST LUBRICANT
0.9 G OF R-152A

AGING SCHEDULE:

14 DAYS AT TEMPERATURES SHOWN

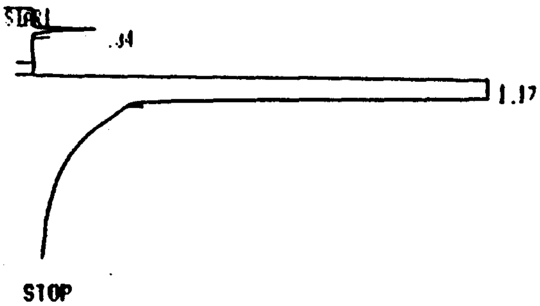
TEST RESULTS:

| | VISUAL APPEARANCE OF TUBES | | CHEMICAL ANALYSIS | | |
|---|---|---|---|---|---|
| AGING TEMP, °C (°F) | COLOR CHANGE OF OIL (ASTM) | OTHER VISUAL OBSERVATIONS | GC | F ION (UG) | %R-152A REACTED |
| 150 (300) | FROM 2.0 TO 2.5 | STEEL: NO CHANGE | NO CHANGE | 0.4 | 0.000 |
| 175 (347) | FROM 2.0 TO 2.5 | STEEL: NO CHANGE | NO CHANGE | 2.0 | 0.001 |
| 200 (392) | FROM 2.0 TO 3.0 | STEEL: DARKENED | NO CHANGE | 1.1 | 0.000 |
| | | (FLUORIDE ANALYSIS BLANK: | | 3.1) | |

NOTE: R-152A AND ALKYLBENZENE LUBRICANT WERE NOT MISCIBLE AT ROOM TEMPERATURE. ALL TUBES EXHIBITED TWO DISTINCT LIQUID PHASES.

FIGURE A-33

R-152a "As Received"




RUN # 10 MAR/04/92 10:22:50

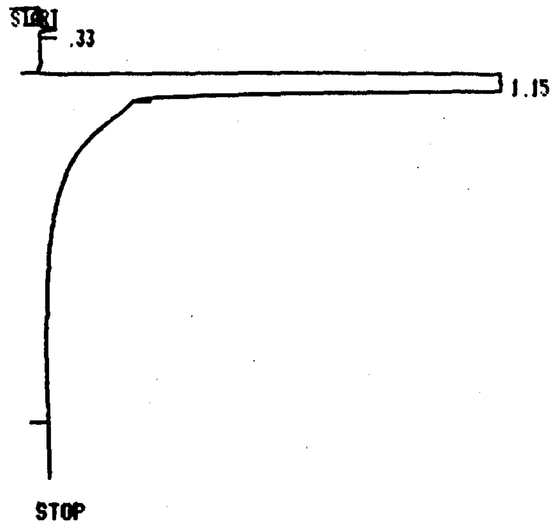
AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 3322 | BB | 0.050 | 0.134 |
| 1.17 | 2468900 | PB | 0.141 | 99.866 |

TOTAL AREA= 2472300
MUL FACTOR= 1.0000E+00

Tube CQ-01; R-152a/Alkylbenzene

14 Days @ 150°C




RUN # 13 APR/15/92 14:48:36

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 938 | BB | 0.052 | 0.044 |
| 1.15 | 2146500 | PB | 0.135 | 99.956 |

TOTAL AREA= 2147500
MUL FACTOR= 1.0000E+00

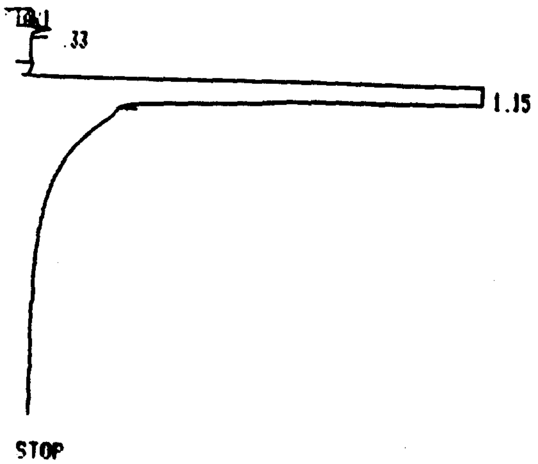
<u>FIGURE A-34</u>

Tube CQ-12; R-152a/Alkylbenzene

14 Days @ 175°C

Tube CQ-21

14 Days @ 200°C

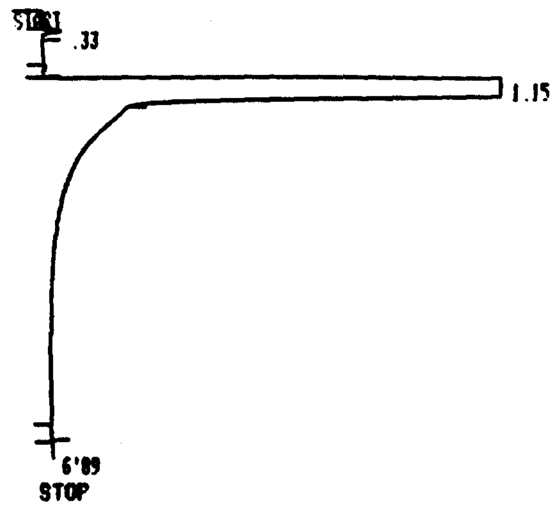



RUN # 14 APR/15/92 15:12:25

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 1066 | BB | 0.052 | 0.051 |
| 1.15 | 2004600 | PB | 0.134 | 99.949 |

TOTAL AREA= 2005600
MUL FACTOR= 1.0000E+00




RUN # 15 APR/15/92 15:35:40

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 952 | BB | 0.051 | 0.044 |
| 1.15 | 2170400 | PB | 0.135 | 99.956 |

TOTAL AREA= 2171400
MUL FACTOR= 1.0000E+00

TABLE A-18: STABILITY OF R-134 WITH PENTAERYTHRITOL ESTER (MIXED ACID I)

SEALED TUBE CONTENTS:

VALVE STEEL STRIP
1.0 cc (1.0 G) OF TEST LUBRICANT
1.0 G OF R-134

AGING SCHEDULE:

14 DAYS AT TEMPERATURES SHOWN

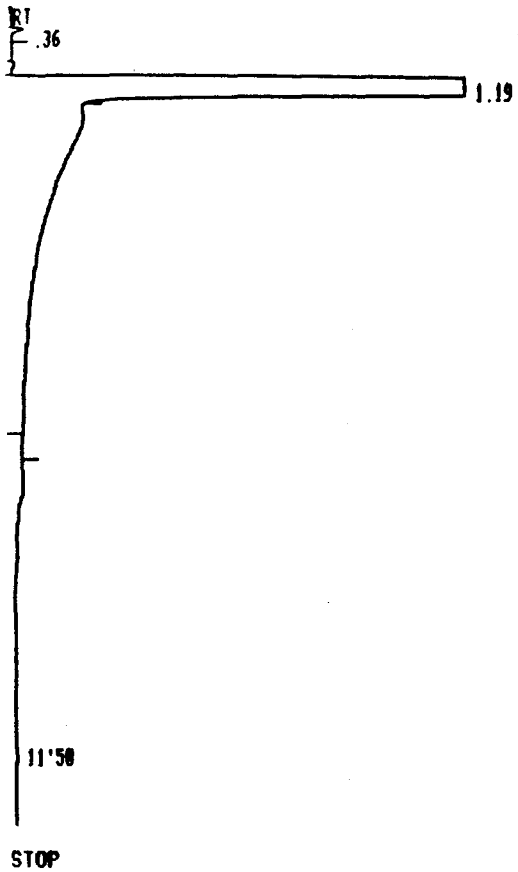
TEST RESULTS:

| | VISUAL APPEARANCE | | CHEMICAL ANALYSIS | | | | |
|---|---|---|---|---|---|---|---|
| AGING TEMP. °C, (°F) | OIL COLOR CHANGE (ASTM) | OTHER VISUAL CHANGES | GC | F ION (UG) | %R-134 REACTED | OIL ACID No. | OIL IR (DIFFEREN-TIAL SCAN) |
| 150 (300) | NO CHANGE AT 2.5 | STEEL: NO CHANGE | NO CHANGE | 1.2 | 0.001 | 0.59 | NO SIGNIF. CHANGES |
| 175 (347) | FROM 2.5 TO 3.0 | STEEL: NO CHANGE | NO CHANGE | 0.1 | 0.000 | 0.83 | NO SIGNIF. CHANGES |
| 200 (392) | FROM 2.5 TO 3.0 | STEEL: DARKENED | NO CHANGE | 0.7 | 0.000 | 1.69 | NO SIGNIF. CHANGES |

(FLUORIDE ANALYSIS BLANK: 3.6)

FIGURE A-35

R-134 "As Received"



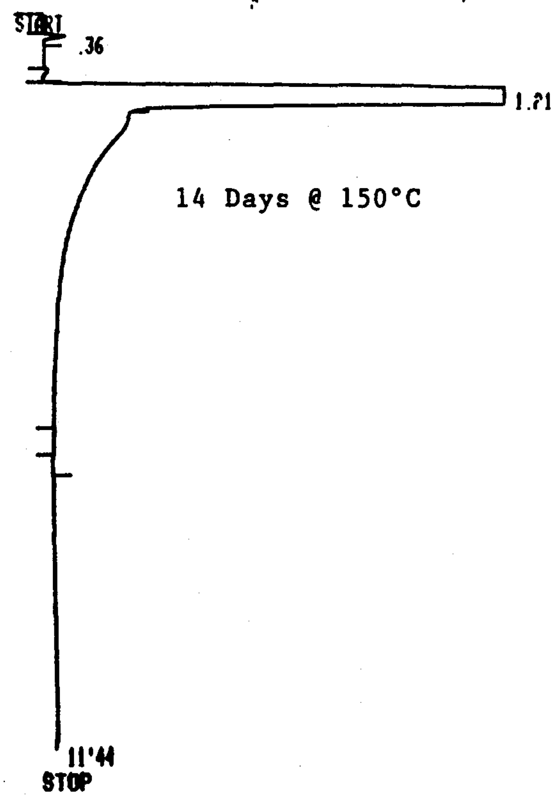

RUN # 7 JUN/11/92 08:26:36

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.36 | 666 | PB | 0.056 | 0.035 |
| 1.19 | 1904200 | PB | 0.133 | 99.965 |

TOTAL AREA= 1904900
MUL FACTOR= 1.0000E+00

Tube CR-2 R-134/PE Ester (mixed acid)




RUN # 2 JUN/24/92 11:34:29

AREA%

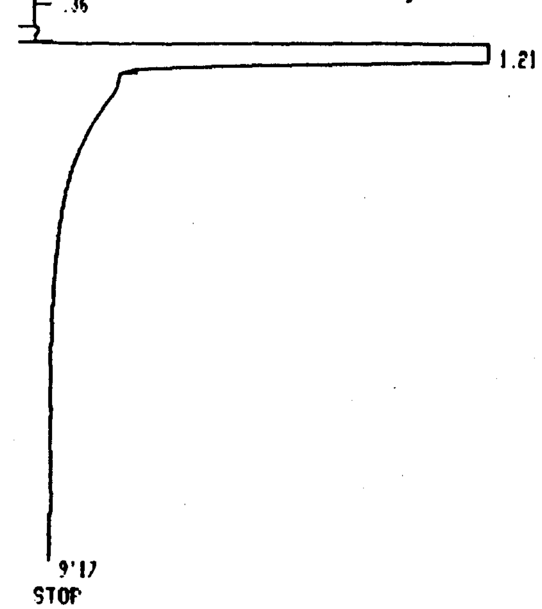
| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.36 | 1210 | PB | 0.054 | 0.065 |
| 1.21 | 1867600 | PB | 0.135 | 99.935 |

TOTAL AREA= 1868800
MUL FACTOR= 1.0000E+00

FIGURE A-36

Gas Chromatograms of R-134/Pentaerythritol Ester (mixed acid I)

Tube CR-11 14 Days at 175°C



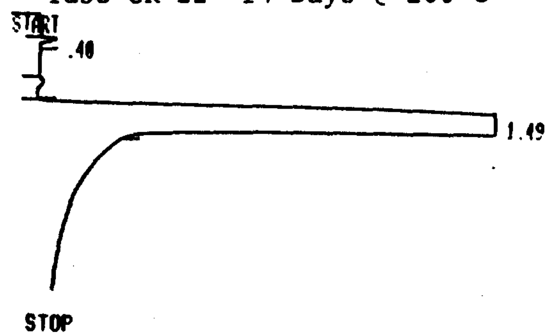

RUN # 3 JUN/24/92 12:14:46

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.36 | 916 | BB | 0.052 | 0.050 |
| 1.21 | 1834700 | PB | 0.135 | 99.950 |

TOTAL AREA= 1835600
MUL FACTOR= 1.0000E+00

Tube CR-22 14 Days @ 200°C




RUN # 5 JUL/23/92 16:10:13

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.40 | 1070 | BB | 0.062 | 0.054 |
| 1.49 | 1967900 | PB | 0.163 | 99.946 |

TOTAL AREA= 1969000
MUL FACTOR= 1.0000E+00

TABLE A-19: STABILITY OF R-11 WITH MINERAL OIL (ISO VG32)

SEALED TUBE CONTENTS:

VALVE STEEL STRIP
1.0 cc (1.0 G) OF TEST LUBRICANT
0.9 G OF R-11

AGING SCHEDULE:

14 DAYS AT TEMPERATURES SHOWN

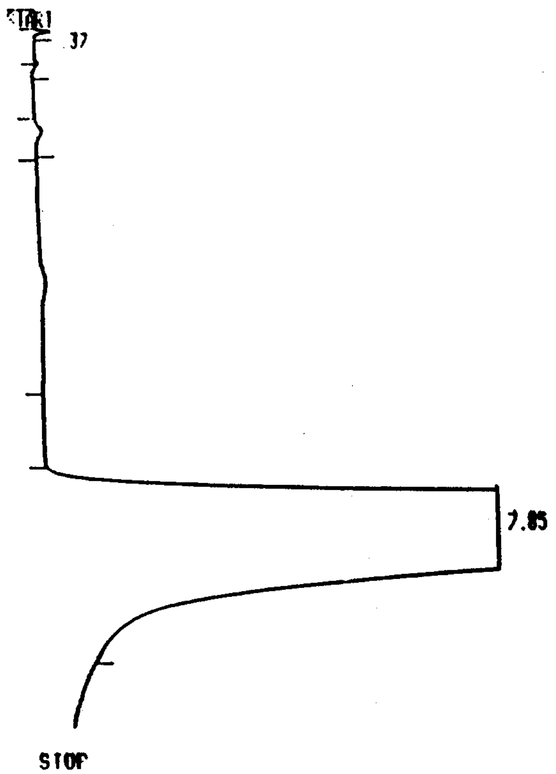
TEST RESULTS:

| | VISUAL APPEARANCE OF TUBES | | CHEMICAL ANALYSIS | | |
|---|---|---|---|---|---|
| AGING TEMP. °C (°F) | OIL COLOR CHANGE (ASTM) | OTHER VISUAL OBSERVATIONS | GC -% OF R-11 REACTED (UNCORRECTED), PRODUCTS FOUND | CL ION (UG) | %R-11 REACTED (FROM CL ION) |
| 105 (220) | FROM 2.5 TO 4.5 | STEEL: LIGHT STAINS | NO MEASURABLE CHANGES | 1,063 | 0.46 |
| 150 (300) | FROM 2.5 TO >8.0 (BLACK) | OIL: GELLED, STEEL: COVERED WITH SLUDGE | 72.0 R-21, <0.5 R-31, 1.0 $CO_2$ | 224,600 | 72.0 |
| 180 (320) . | FROM 2.5 TO >8.0 (BLACK) | OIL: GELLED, STEEL: COVERED WITH SLUDGE | R-11 HAS DISAPPEARED, 47.8 R-21, 17.2 R-31, 31.4 $CO_2$ | 270,200 | 87.0 |

NOTE: SEE PHOTOGRAPHIC RECORD OF VISUAL OBSERVATIONS IN APPENDIX D

FIGURE A-37

R-11 "As Received"




RUN # 10 JUN/24/92 17:18:40

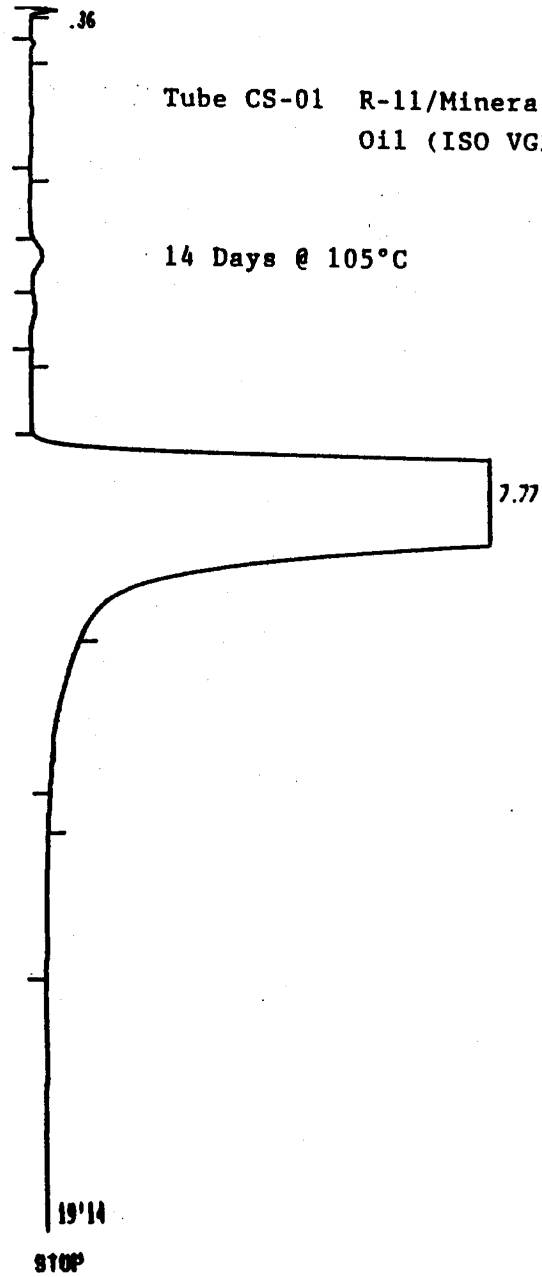
AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.37 | 900 | BB | 0.057 | 0.053 |
| 7.85 | 1698000 | PB | 0.920 | 99.947 |

TOTAL AREA= 1698900
MUL FACTOR= 1.0000E+00

Tube CS-01 R-11/Mineral Oil (ISO VG32)

14 Days @ 105°C




RUN # 8 JUN/24/92 16:02:29

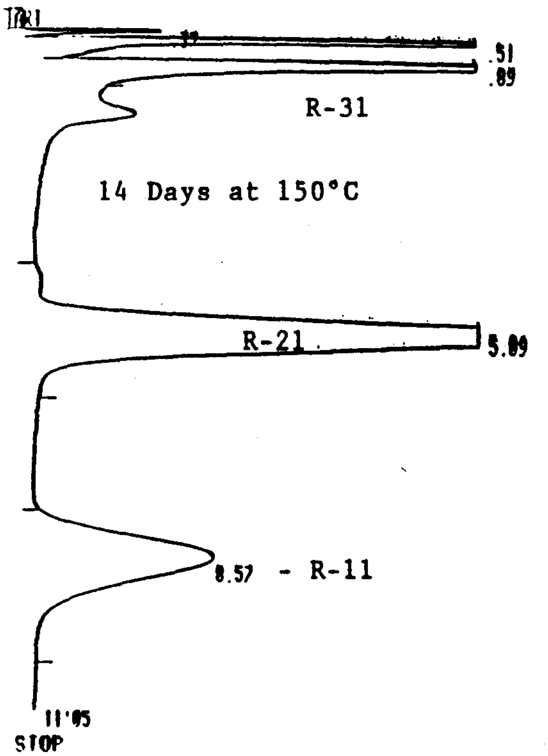
AREA%

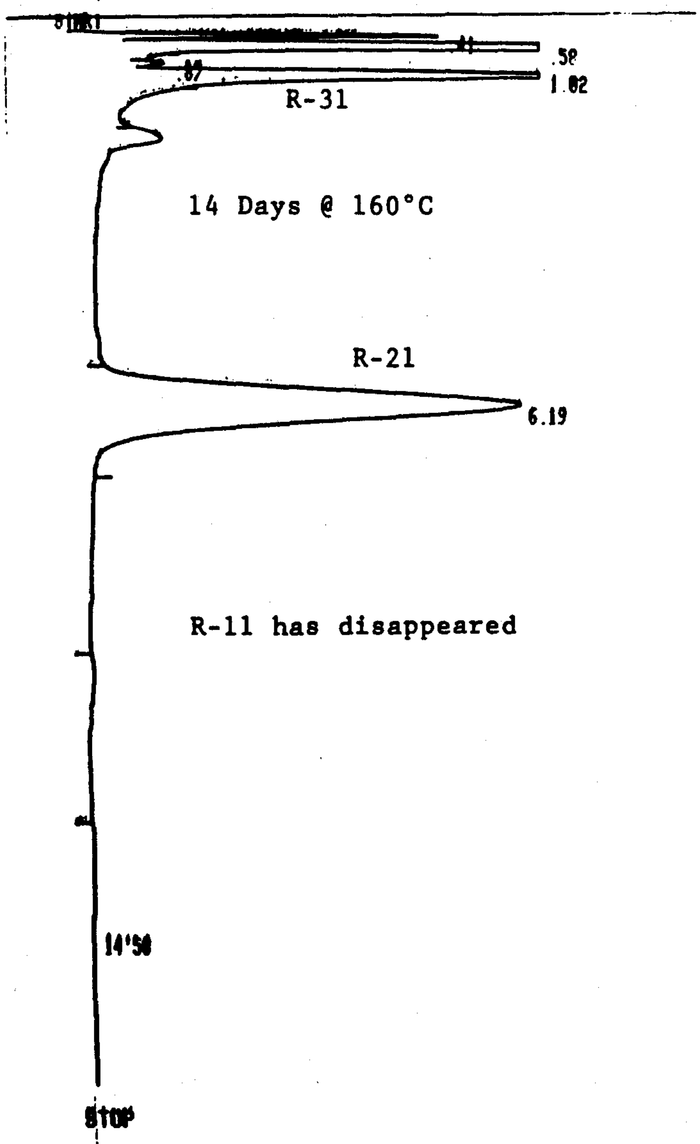
| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.36 | 1544 | PB | 0.056 | 0.080 |
| 7.77 | 1939000 | PB | 0.945 | 99.920 |

TOTAL AREA= 1940500
MUL FACTOR= 1.0000E+00

FIGURE A-38

Tube CS-14 R-11/Mineral Oil (ISO VG32) Tube CS-21




RUN # 9 JUN/24/92 16:45:52

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.37 | 7231 | PP | 0.051 | 1.044 |
| 0.51 | 94343 | PB | 0.055 | 13.624 |
| 0.89 | 115240 | BB | 0.145 | 16.642 |
| 5.09 | 315350 | PB | 0.463 | 45.539 |
| 8.57 | 160310 | BB | 0.833 | 23.151 |

TOTAL AREA= 692480
MUL FACTOR= 1.0000E+00

RUN # 4 JUL/22/92 16:32:23

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.41 | 18121 | PV | 0.052 | 3.425 |
| 0.58 | 166110 | VB | 0.074 | 31.398 |
| 0.87 | 1172 | BP | 0.061 | 0.222 |
| 1.02 | 90938 | PB | 0.106 | 17.189 |
| 6.19 | 252720 | BB | 0.563 | 47.767 |

TABLE A-20: STABILITY OF R-11 WITH WHITE NAPHTHENIC MINERAL OIL (ISO VG46)

SEALED TUBE CONTENTS:

VALVE STEEL STRIP
1.0 cc (1.0 G) OF TEST LUBRICANT
0.9 G OF R-11

AGING SCHEDULE:

14 DAYS AT TEMPERATURES SHOWN

TEST RESULTS:

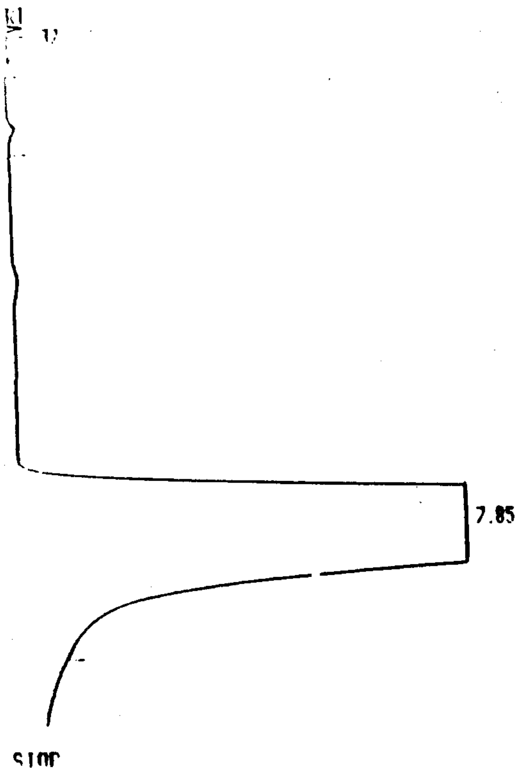
| | VISUAL APPEARANCE OF TUBES | | CHEMICAL ANALYSIS | | |
|---|---|---|---|---|---|
| AGING TEMP. °C, (°F) | OIL COLOR CHANGE (ASTM) | OTHER VISUAL OBSERVATIONS | GC-% OF R-11 REACTED (UNCORRECTED), PRODUCTS FOUND | CL ION (UG) | %R-11 REACTED (FROM CL ION) |
| 105 (220) | NO CHANGE FROM 2.0 | STEEL: LIGHT STAINS | NO MEASURABLE CHANGE | 33 | 0.014 |
| 150 (300) | FROM 2.0 TO >8.0 (BLACK) | STEEL: ETCHED, LIGHT SLUDGE | 17.8 R-21<br>1.37 R-31<br>$CO_2$ | 94,500 | 45.2 |
| 160 (320) | FROM 2. 0 TO >8.0 (BLACK) | STEEL: ETCHED, OIL: GELLED | R-11: ALMOST DISAPPEARED,<br>75.0 R-21<br>10.4 R-31<br>13.7 $CO_2$ | 202,500 | 97. 0 |

NOTE: SEE PHOTOGRAPHIC RECORD OF VISUAL OBSERVATIONS IN APPENDIX D

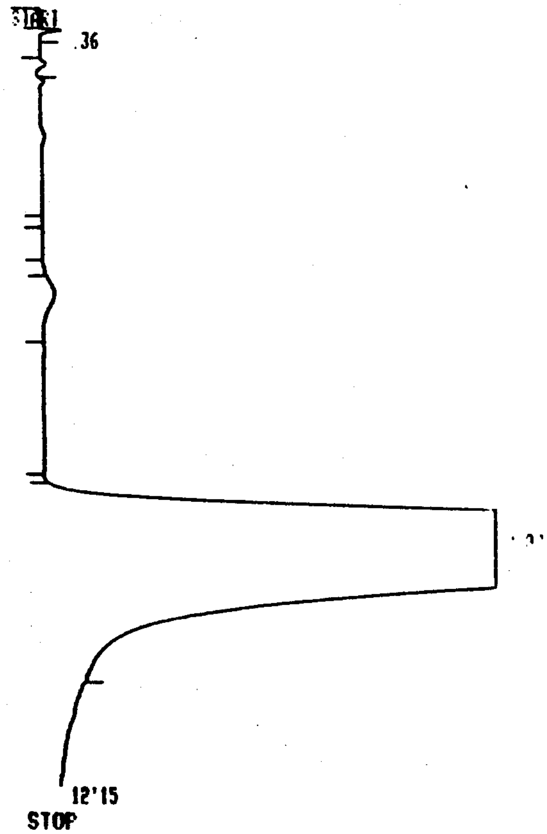
FIGURE A-39

Gas Chromatograms of R-11/White Naphthenic Mineral Oil (ISO VG46)

R-11 "As Received"




Tube CT-01 (14 days @105°C)




RUN # 10 JUN/24/92 17:18:40

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.37 | 900 | BB | 0.057 | 0.053 |
| 7.85 | 1698000 | PB | 0.928 | 99.947 |

TOTAL AREA= 1698900
MUL FACTOR= 1.0000E+00

RUN # 2 JUL/08/92 11:18:02

AREA%

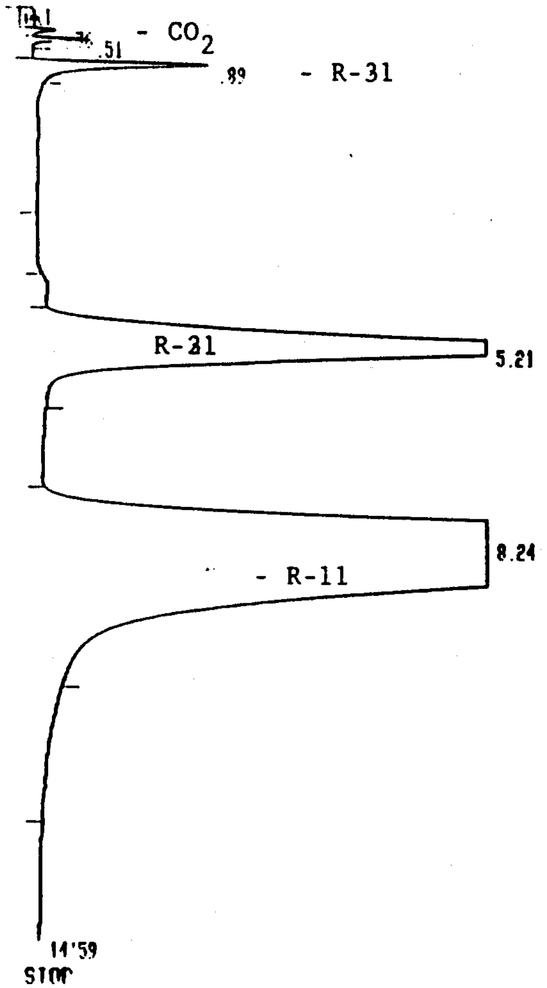
| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.36 | 1158 | BB | 0.054 | 0.071 |
| 8.07 | 1622600 | BB | 9.958 | 99.929 |

TOTAL AREA= 1623790
MUL FACTOR= 1.0000E+00

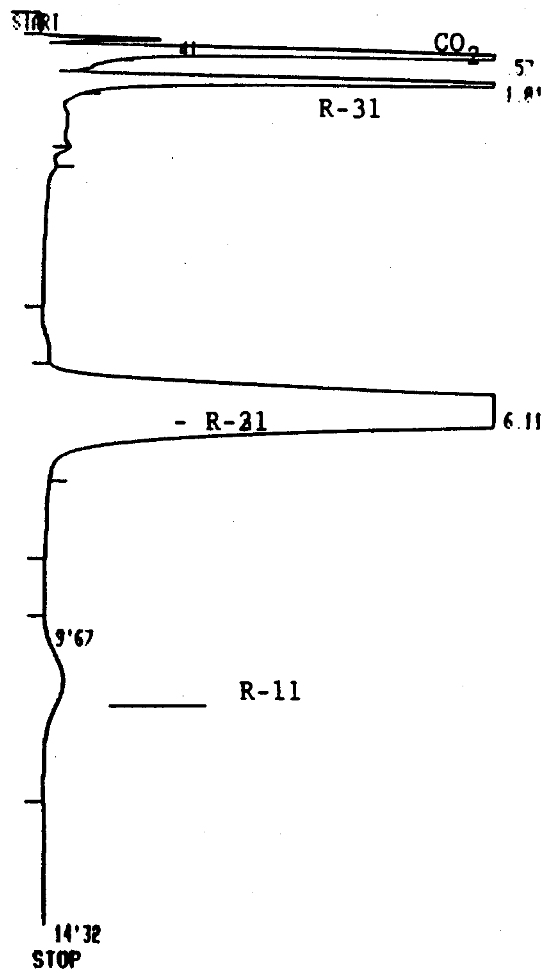
FIGURE A-40

Gas Chromatograms of R-11/White Naphthenic Mineral Oil (ISO VG 46)

Tube CT-13 (14 days @ 150°C)




Tube CT-21 (14 days @ 160°C)




RUN #    JUL/08/92   13:15:43

AREA%

| RT | A | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.36 | .11 | BP | 0.054 | 0.081 |
| 0.51 | ·08 | PB | 0.054 | 0.199 |
| 0.8? | .570 | PB | 0.113 | 1.367 |
| 5.2 | ·510 | BB | 0.464 | 17.828 |
| 8.? | ?300 | PB | 0.940 | 80.524 |

TOT   1511780
MU   ..0000E+00

RUN #   5   JUL/22/92   17:26:15

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.41 | 5906 | PP | 0.051 | 0.837 |
| 0.57 | 96454 | PB | 0.080 | 13.675 |
| 1.01 | 73669 | PB | 0.109 | 10.445 |
| 6.11 | 529300 | BB | 0.564 | 75.043 |

TOTAL AREA=   705330
MUL FACTOR= 1.0000E+00

TABLE A-21: STABILITY OF R-123 WITH WHITE NAPHTHENIC MINERAL OIL (ISO VG46)

SEALED TUBE CONTENTS:

VALVE STEEL STRIP
1.0 cc (1.0 G) OF TEST LUBRICANT
0.9 G OF R-123

AGING SCHEDULE:

14 DAYS AT TEMPERATURES SHOWN

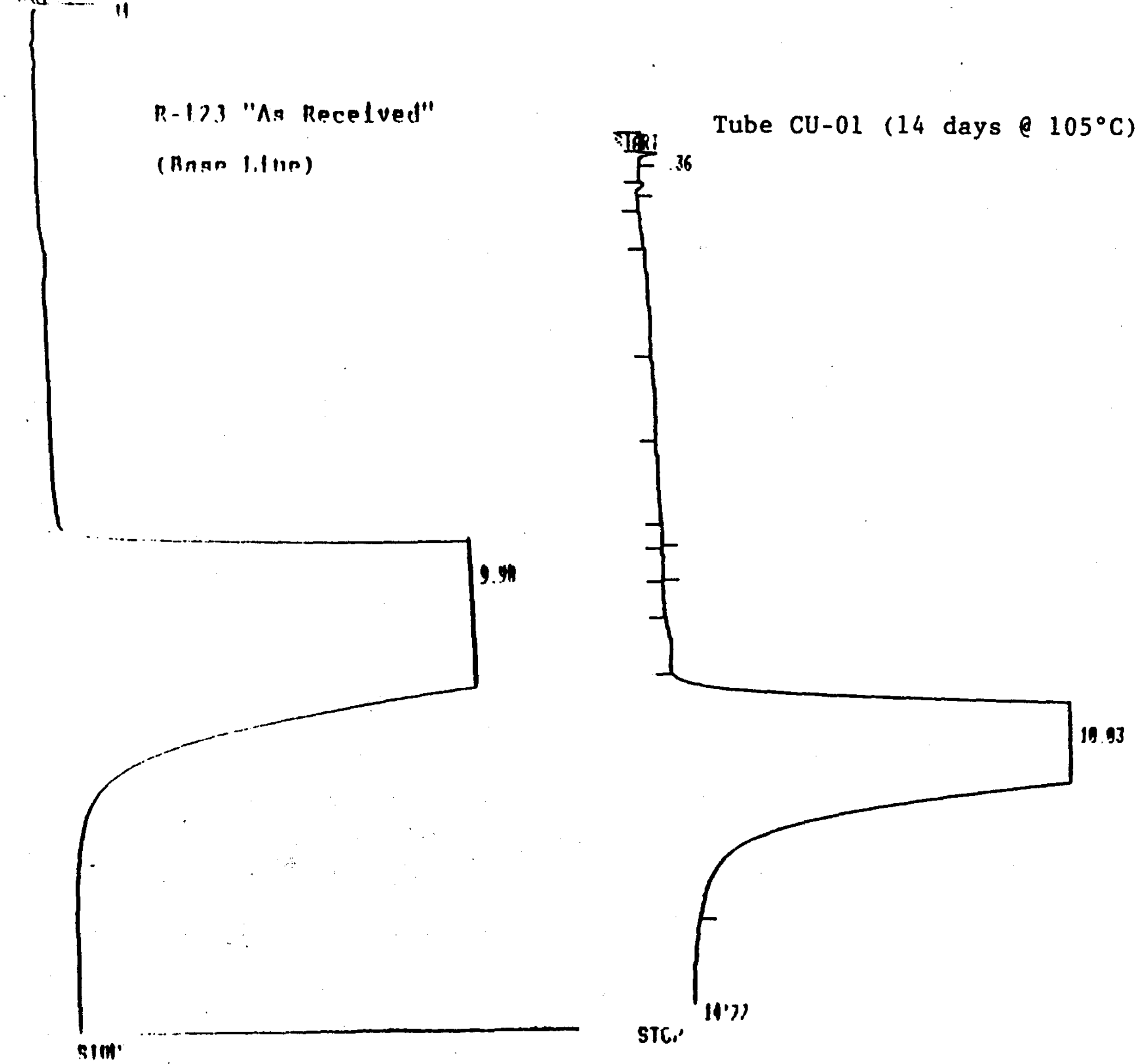
TEST RESULTS:

| | VISUAL APPEARANCE OF TUBES | | CHEMICAL ANALYSIS | | |
|---|---|---|---|---|---|
| AGING TEMP. °C (°F) | OIL COLOR CHANGE (ASTM) | OTHER VISUAL OBSERVATIONS | GC-% OF R-123 REACTED (UNCORRECTED), PRODUCTS FOUND | CL ION (UG) | %R-123 REACTED (FROM CL ION) |
| 105 (220) | NO CHANGE FROM 2.0 | STEEL: NO CHANGE | NO CHANGE | 48 | 0.002 |
| 150 (300) | FROM 2.0 TO 7.5 | STEEL: LIGHT DEPOSITS (OIL SLUDGE) | 3.9 R-133A<br>0.08 R-143A | 12,200 | 5.84 |
| 160 (320) | FROM 2.5 TO >8.0 (BLACK) | STEEL: ETCHED, SLUDGE DEPOSITS | 10.9 R-133A<br>0.38 R-133A<br>$CO_2$ | 37,000 | 8.9 |

NOTE: SEE PHOTOGRAPHIC RECORD OF VISUAL OBSERVATIONS IN APPENDIX D

FIGURE A-41

Gas Chromatograms R-123/White Naphthenic Mineral Oil (ISO VG 46)




RUN # 14 JAN/16/92 15:25:30

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 4060 | BB | 0.054 | 0.105 |
| 9.90 | 3851000 | BB | 1.711 | 99.895 |

TOTAL AREA= 3855000
MUL FACTOR= 1.0000E+00

RUN # 1 JUL/08/92 10:37:20

AREA%

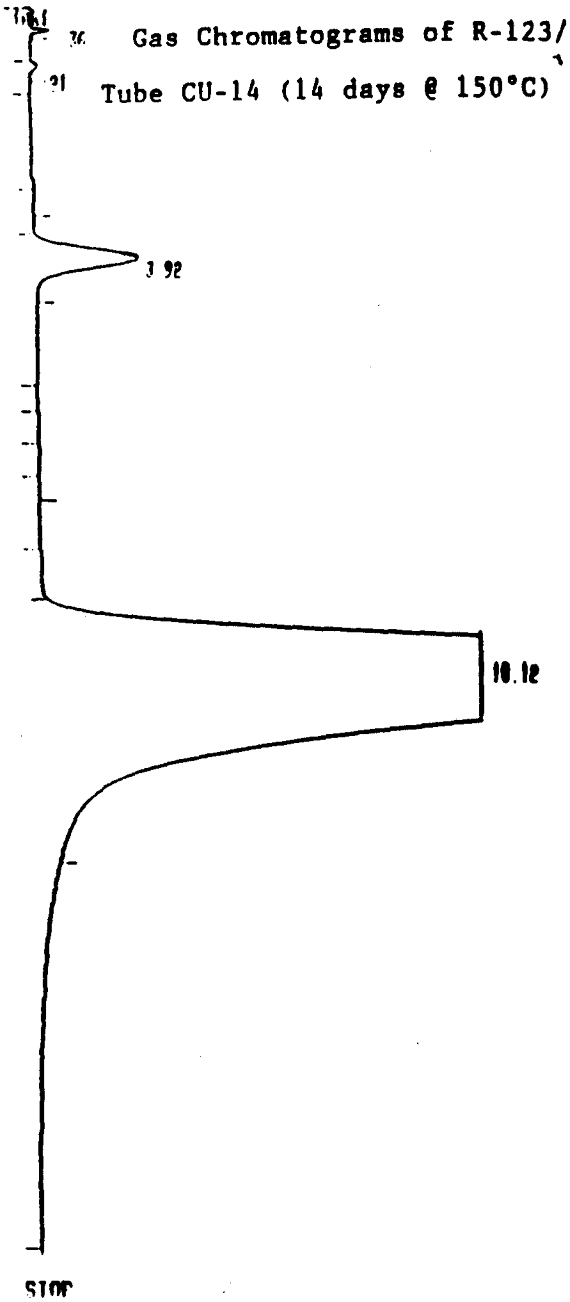
| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.36 | 1089 | BB | 0.053 | 0.073 |
| 10.03 | 1486300 | BB | 1.270 | 99.927 |

TOTAL AREA= 1487400
MUL FACTOR= 1.0000E+00

FIGURE A-42

Gas Chromatograms of R-123/White Naphthenic Mineral Oil (ISO VG46)

Tube CU-14 (14 days @ 150°C)

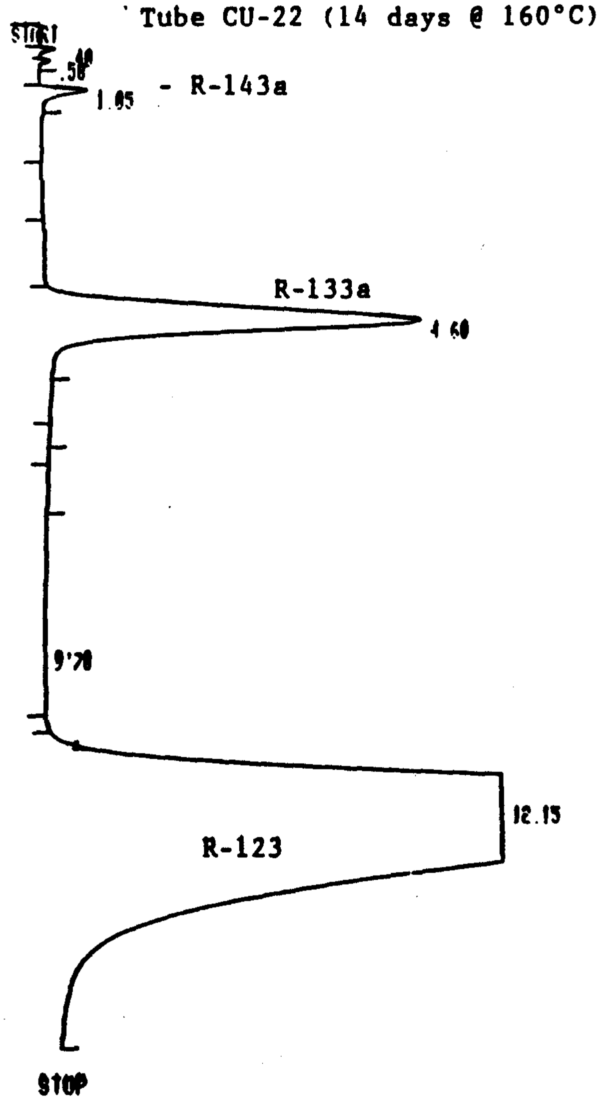



RUN 0 3 JUL/08/92 11:45:43

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.36 | 1083 | BB | 0.054 | 0.074 |
| 0.91 | 1170 | BB | 0.130 | 0.080 |
| 3.92 | 40572 | PB | 0.362 | 2.777 |
| 10.12 | 1418200 | BB | 1.273 | 97.069 |

Tube CU-22 (14 days @ 160°C)




RUN 0 23 JUL/22/92 14:36:24

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.40 | 1056 | BP | 0.065 | 0.067 |
| 0.58 | 734 | PB | 0.062 | 0.047 |
| 1.05 | 6011 | PB | 0.122 | 0.380 |
| 4.60 | 171710 | BB | 0.434 | 10.866 |
| 12.15 | 1400700 | BB | 1.452 | 88.640 |

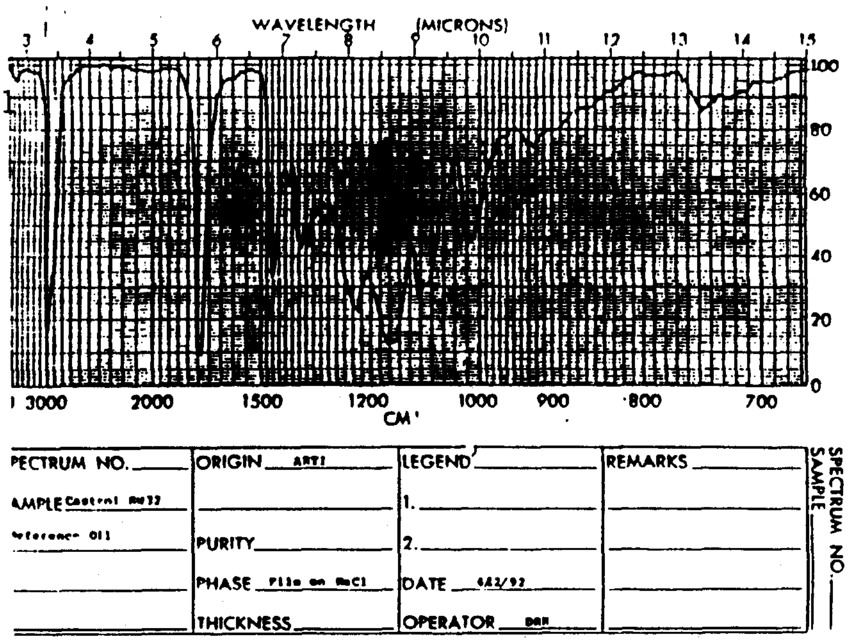
TOTAL AREA= 1580200
MUL FACTOR= 1.0000E+00

APPENDIX B

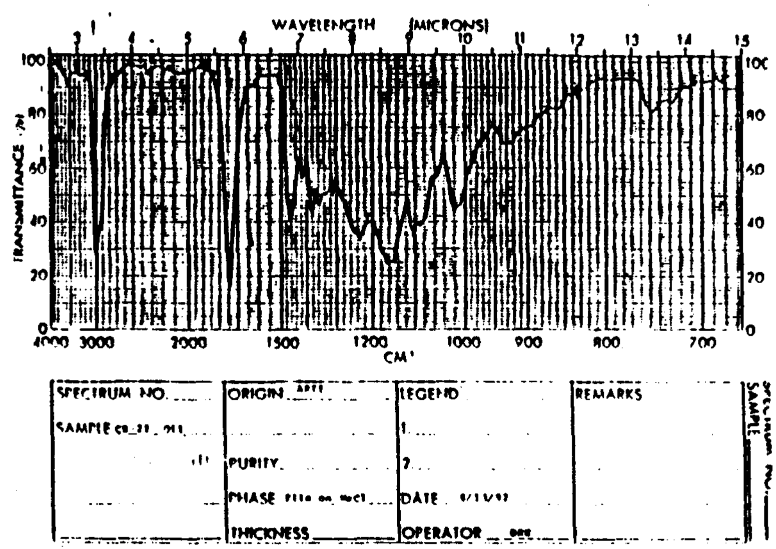
FIGURE B-01

IR Spectra of Pentaerythritol Ester (mixed acid I)aged with R-32

- - - Unused Oil - - -




- - Oil aged for 14 Days at 200°C with R-32




- - Differential Scan: Aged Oil vs. Unused Oil

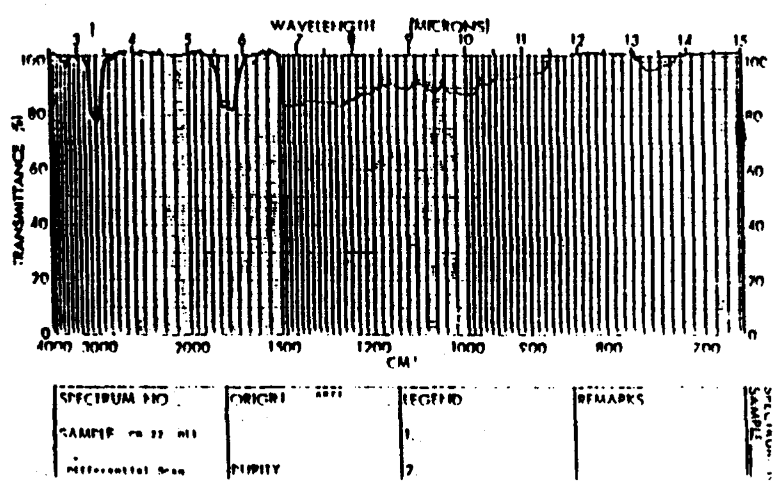
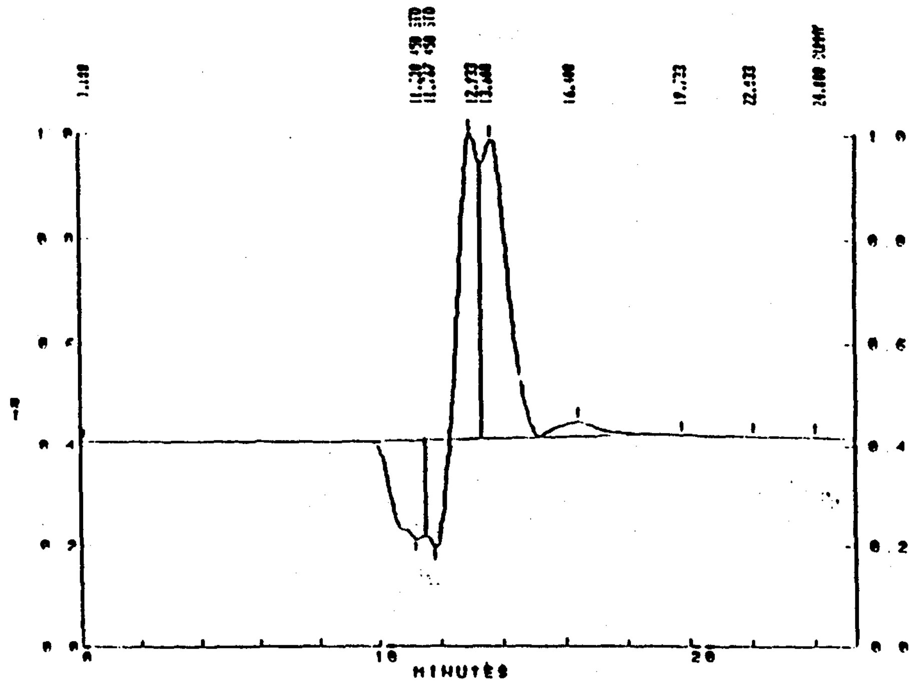
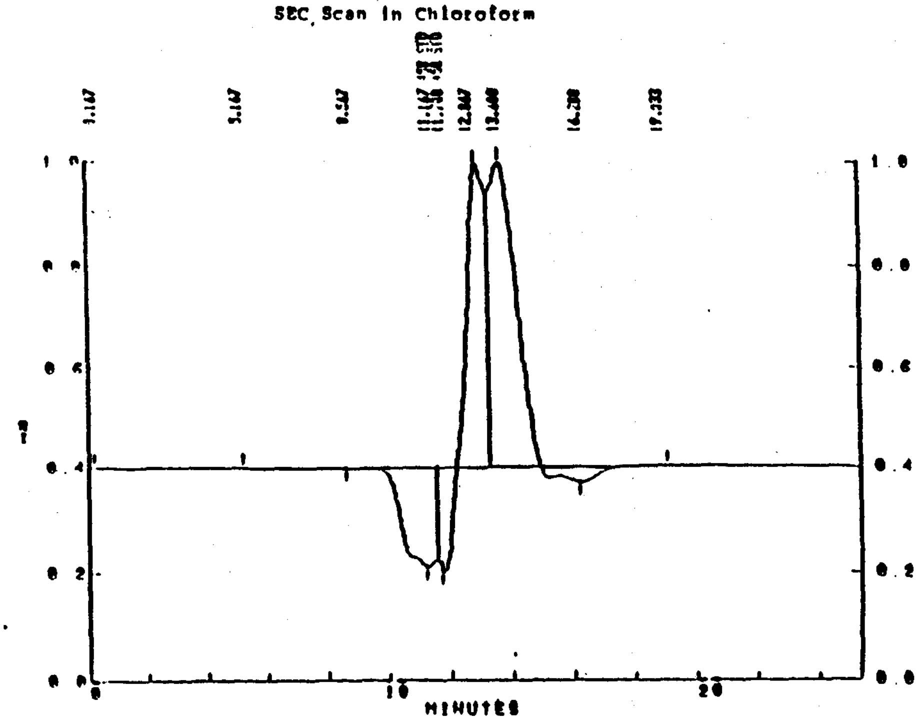


FIGURE B-02

Size Exclusion Chromatogram: PPG butyl monoether/R-32, 14 days @ 150°C



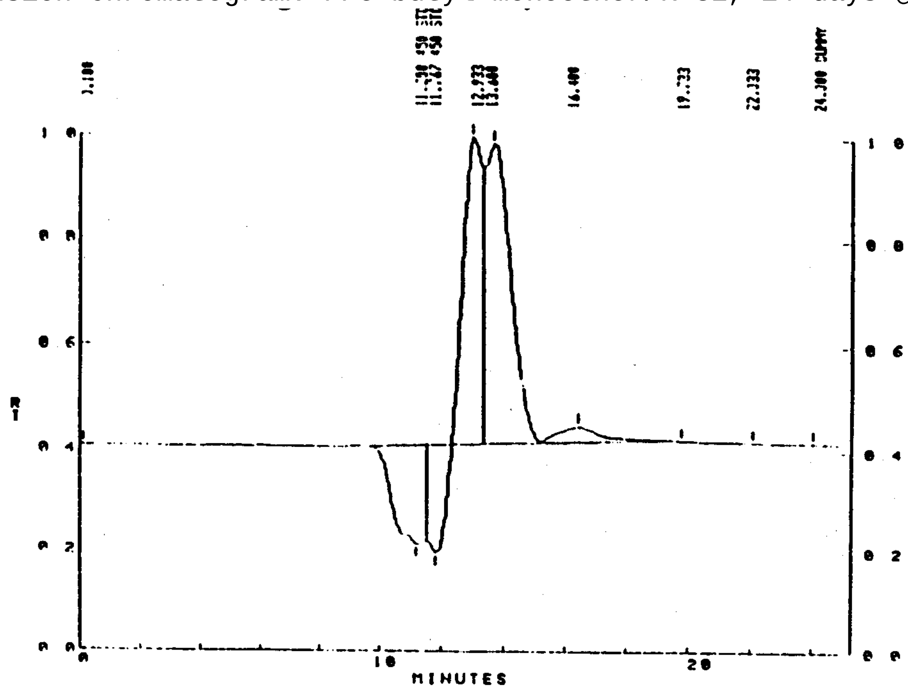

Polypropylene Glycol (butyl monoether) - Unused
SEC Scan in Chloroform



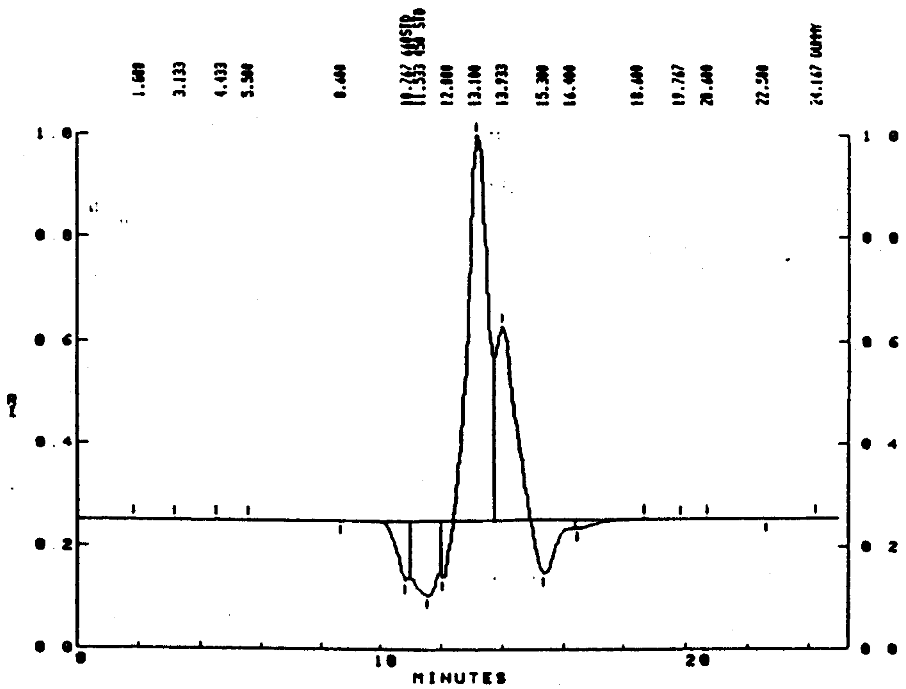

R-12/PPG (butyl monoether) Aged Oil 150°C (Tube #CC-02)

## FIGURE B-03

Size Exclusion Chromatogram: PPG butyl monoether/R-32, 14 days @ 175°C



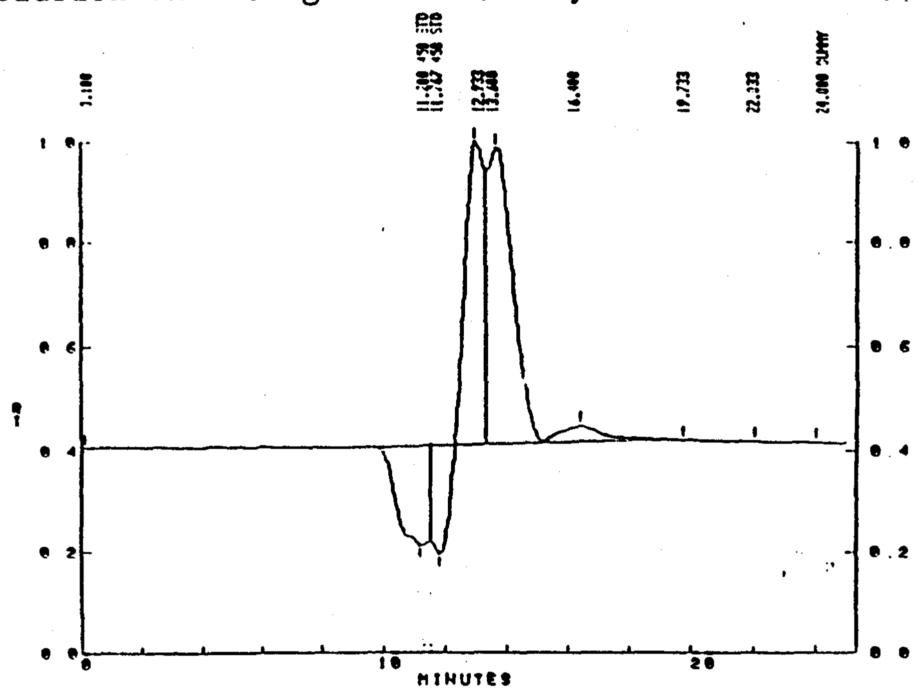

Polypropylene Glycol (butyl monoether) - Unused
SEC Scan in Chloroform



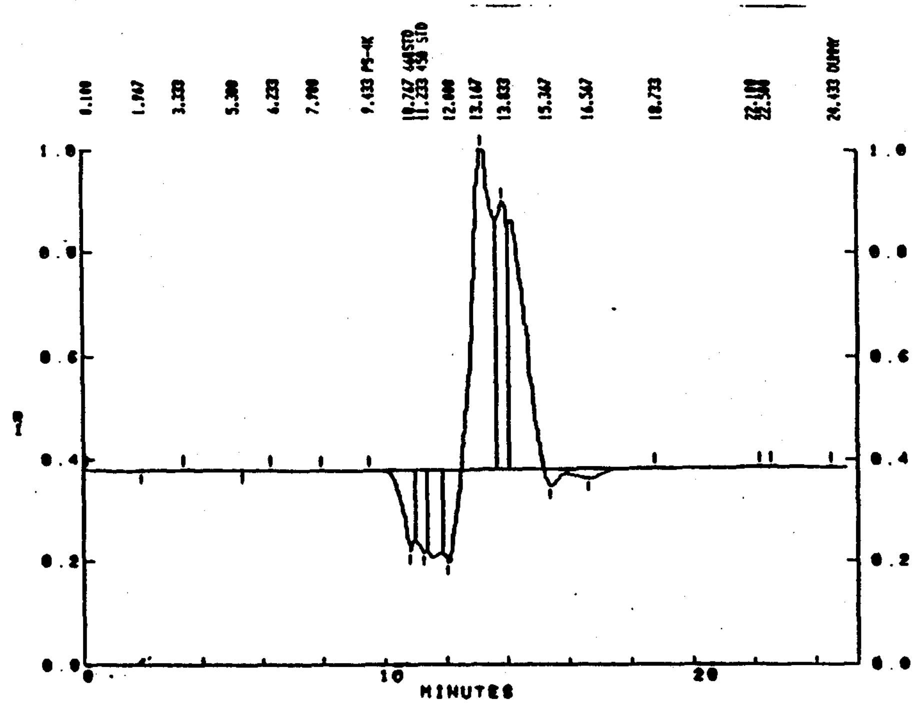

R-32/PPG (butyl monoether) Aged Oil 175°C (Tube CC-16)

FIGURE B-04

Size Exclusion Chromatograms: PPG butyl monoether/R-32, 14 days @ 200°C




Polypropylene Glycol (butyl monoether) - Unused
SEC Scan in Chloroform

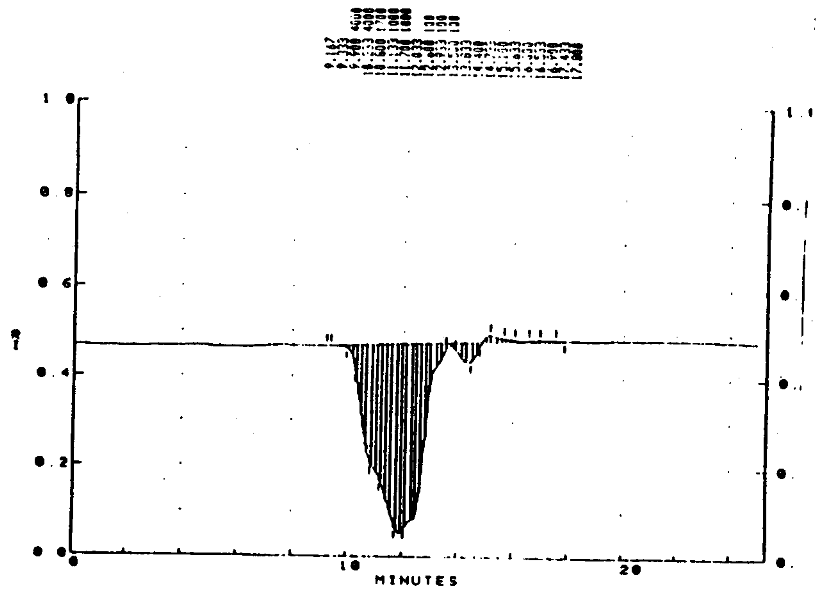



R-32/PPG (butyl monoether) Aged Oil 200°C (Tube CC-24)

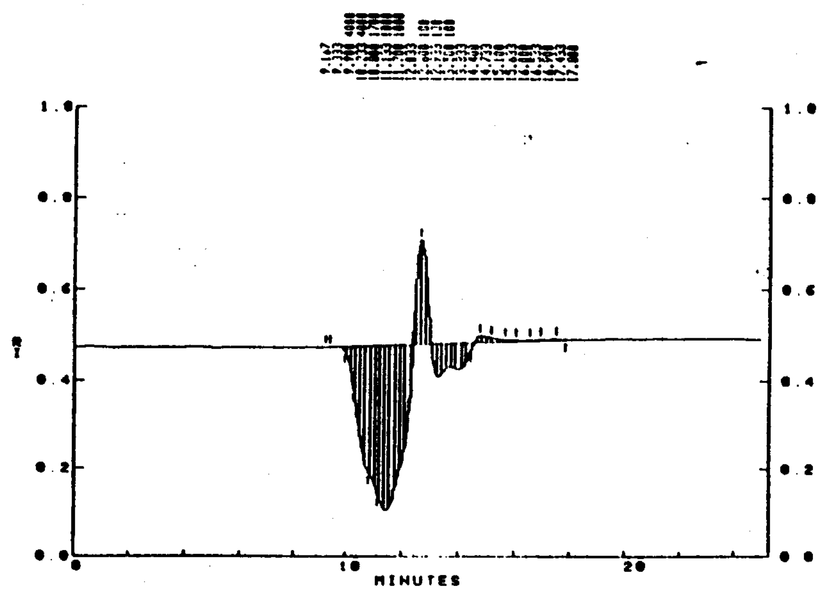
FIGURE B-05

Size Exclusion Chromatograms of PPG butyl monoether samples aged with R-125.

--- 14 days at 150°C




--- 14 days at 175°C




--- 14 days at 200°C

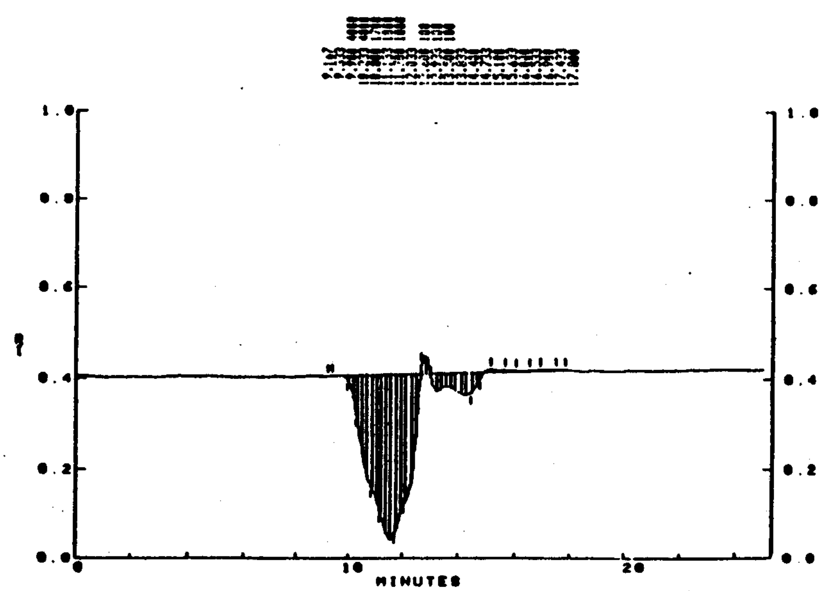
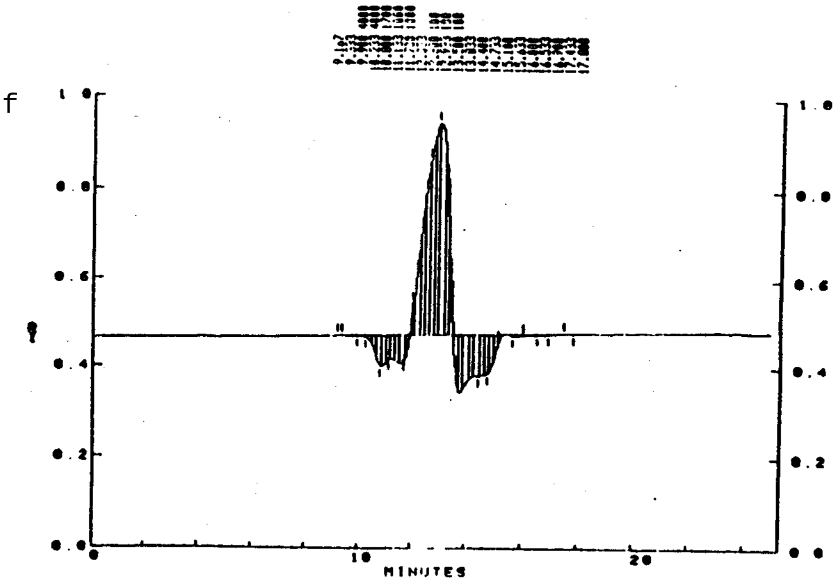


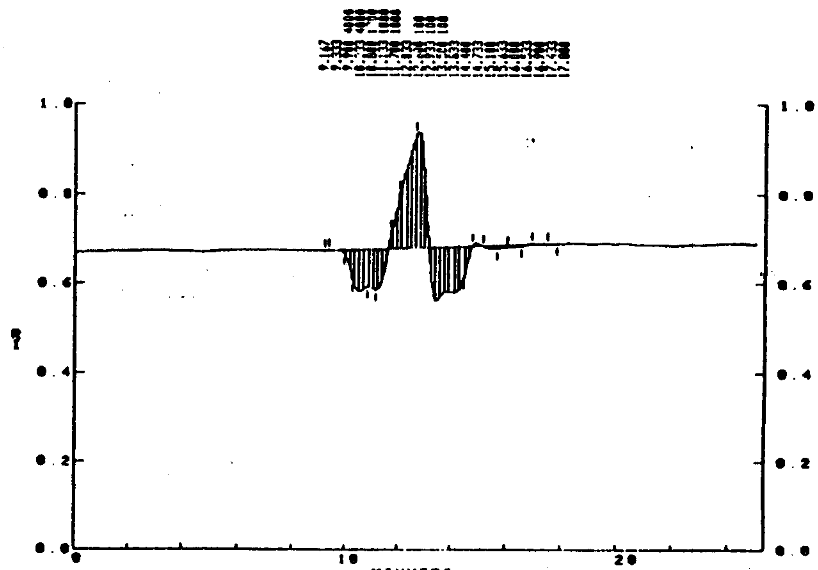
FIGURE B-06

Size Exclusion Chromatograms of Modified Polyglycol aged with R-125.

--- 14 days at 150°C




--- 14 days at 175°C



--- 14 days at 200°C

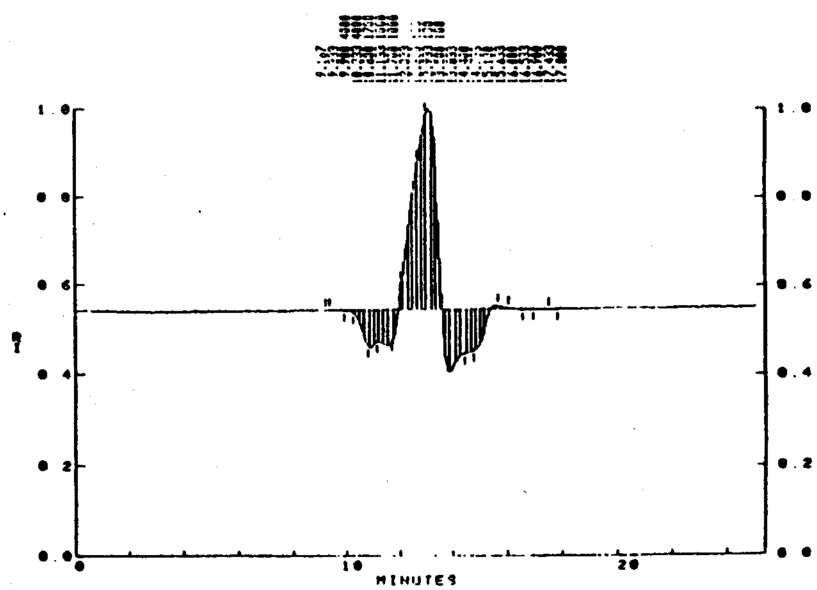
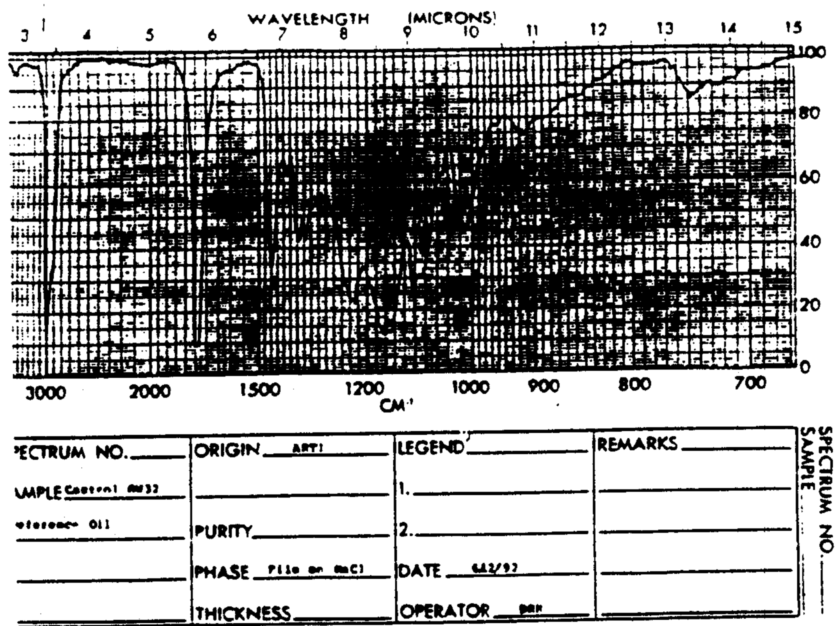


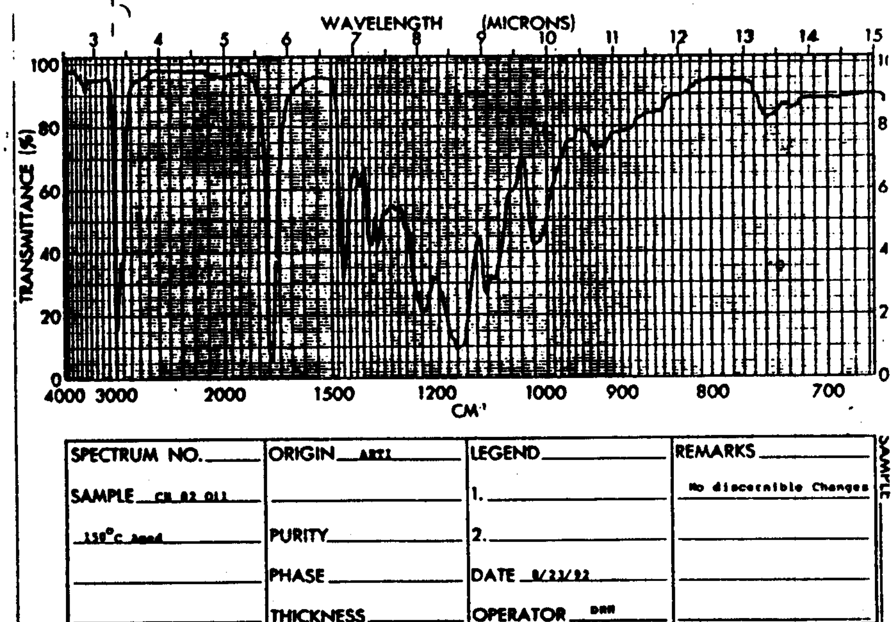
FIGURE B-07

IR Spectra Pentaerythritol Ester (mixed acid I) after Aging at 150°C with R-125.

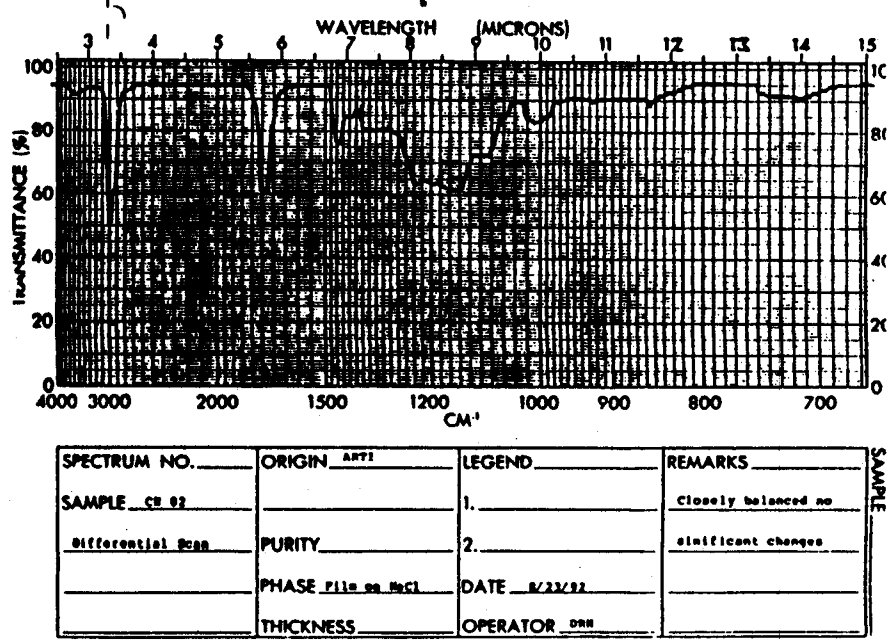
---Unused PE ester, acids




--- Aged PE ester, acids



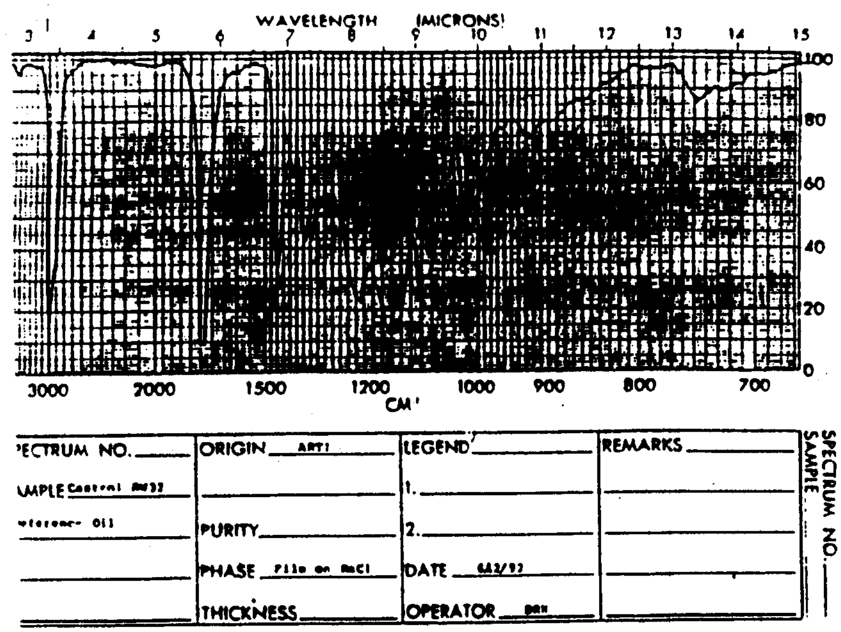

--- Differential Scan: Aged 0i1 vs. New Oil



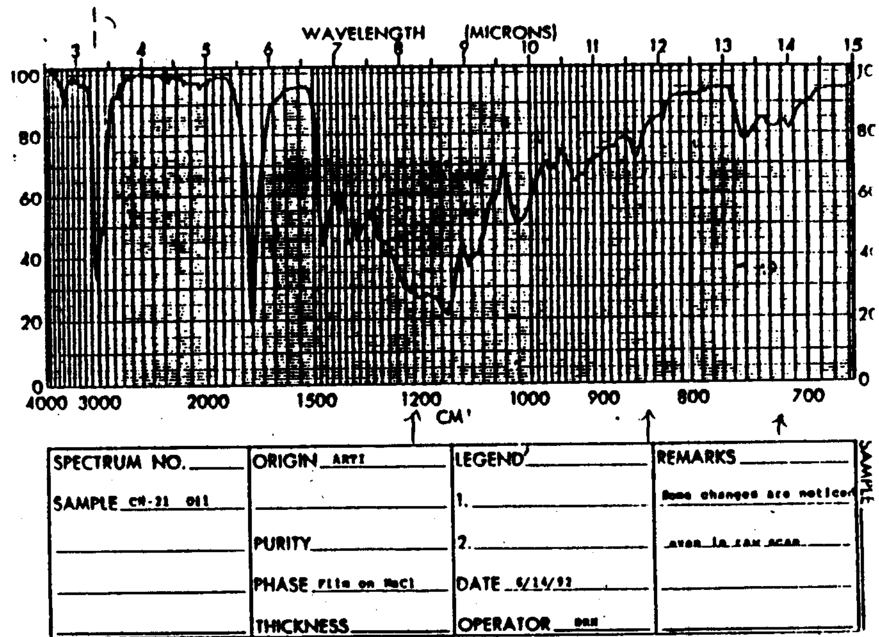
## FIGURE B-08

IR Spectra of Pentaerythritol Ester after Aging at 200°C with R-125.

--- Unused PE ester, mixed acids I




--- Aged PE ester, mixed acids I




--- Differential Scan: Aged Oil vs. Unused Oil

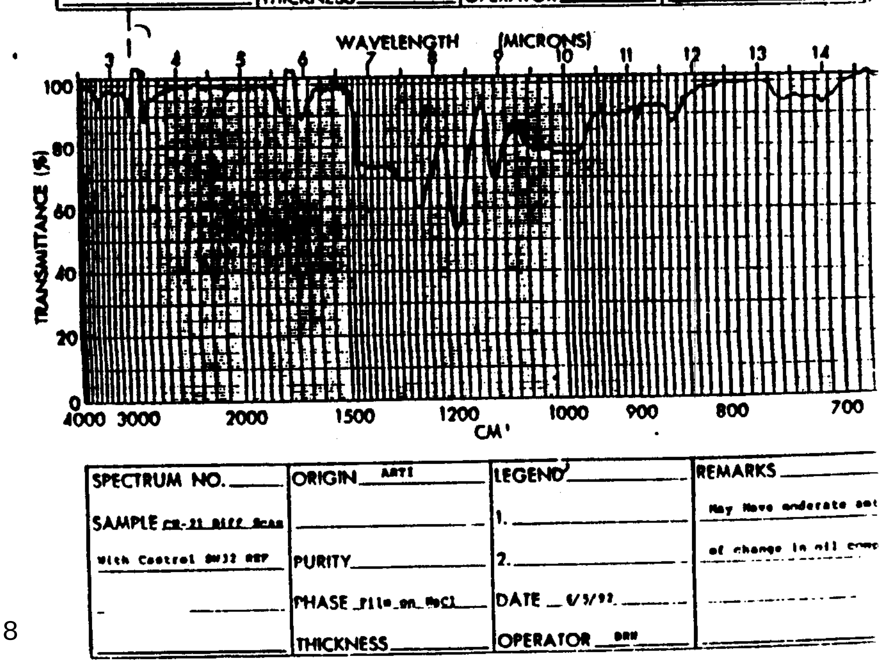
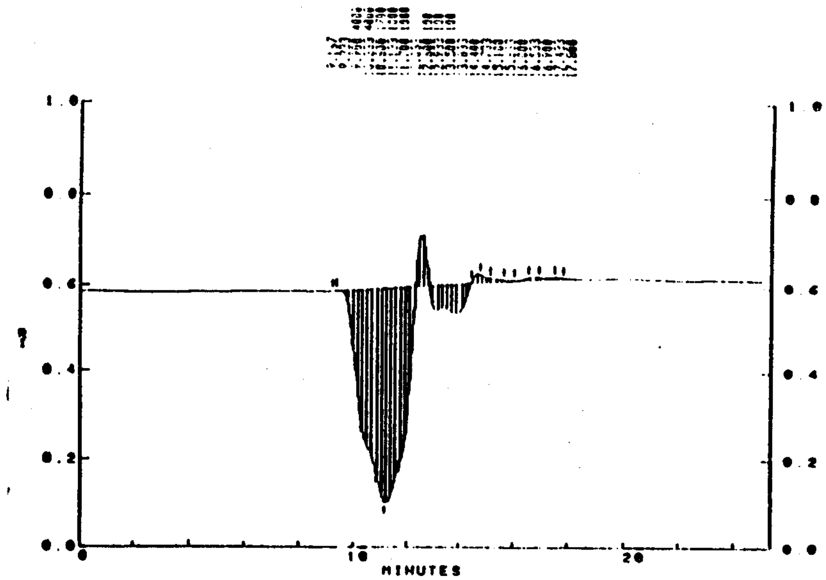


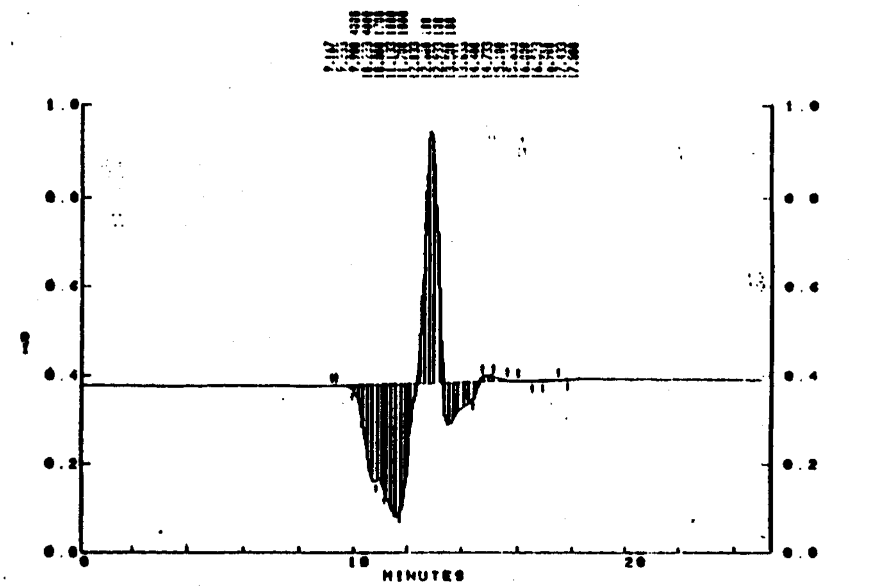
FIGURE B-09

Size Exclusion Chromatograms:
PPG butyl monoether/R-134a

--- 14 days at 150°C




--- 14 days at 175°C




--- 14 days at 200°C

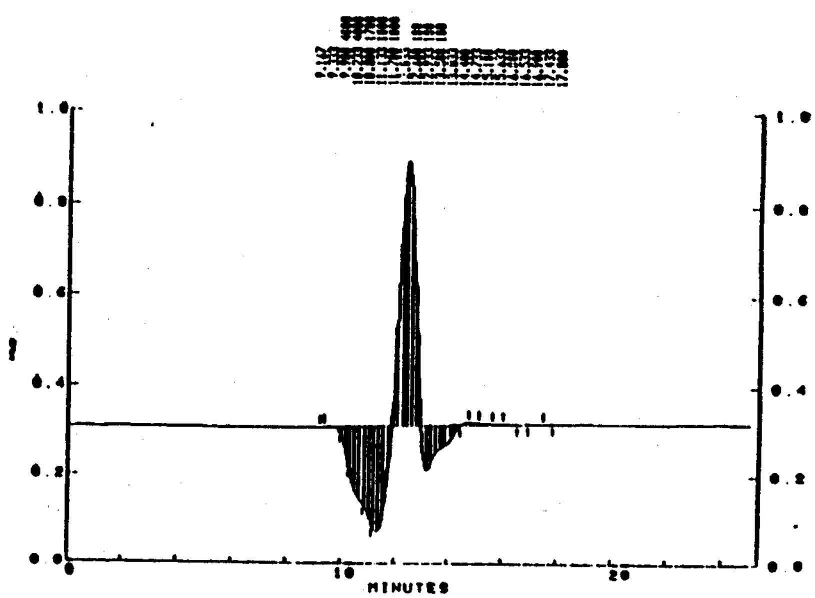
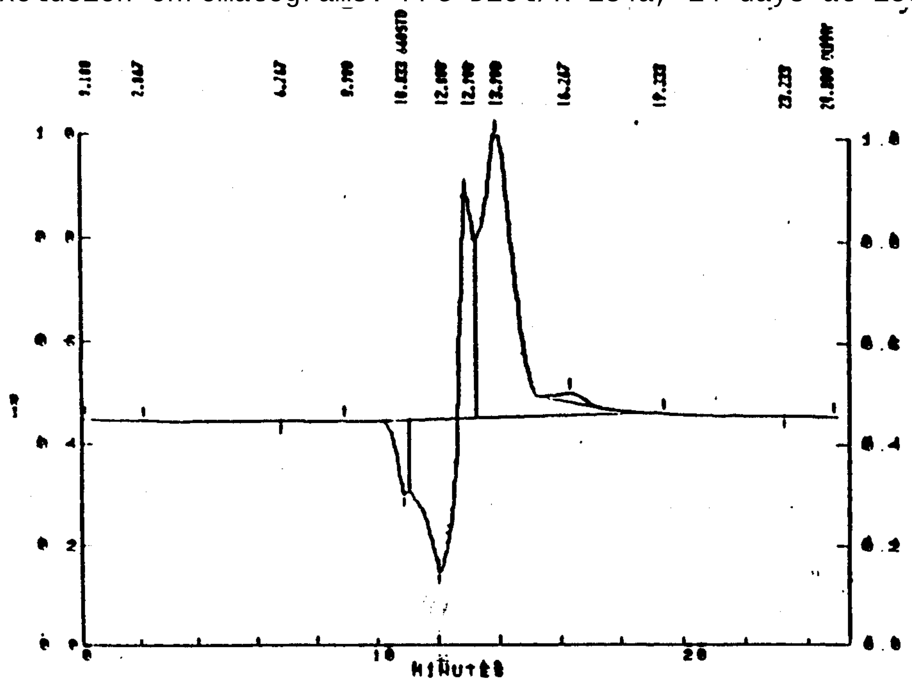
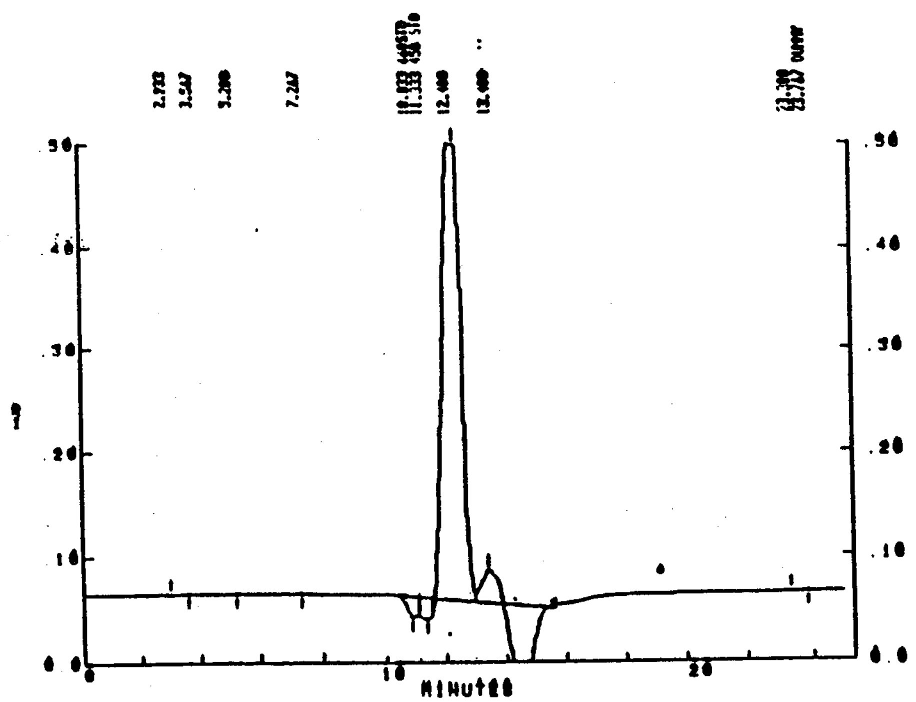


FIGURE B-10

Size Exclusion Chromatograms: PPG Diol/R-134a, 14 days at 150°C



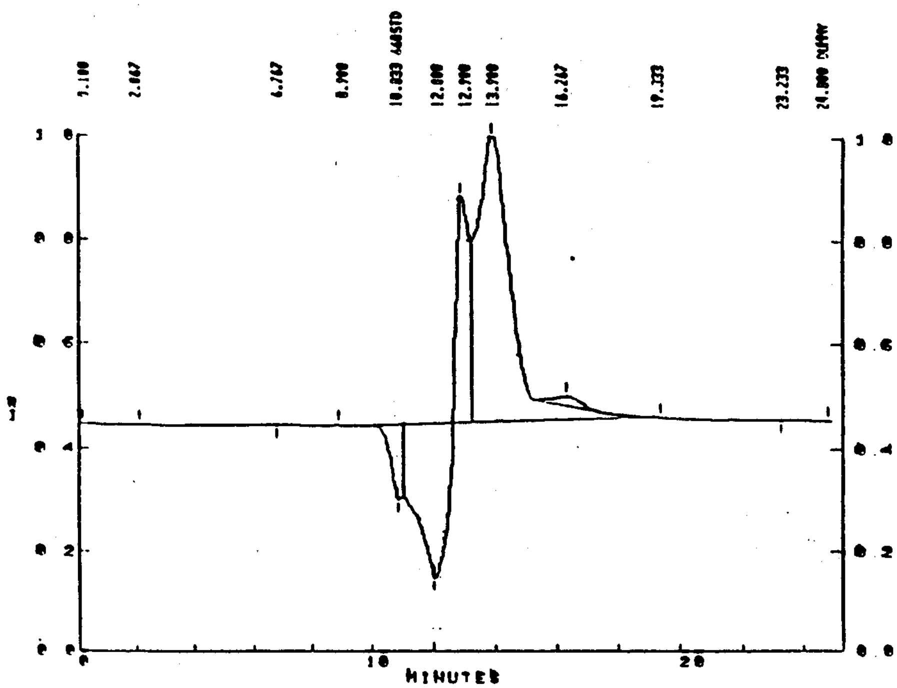
Polypropyleneglycol Diol - Unused Oil
SEC Scan in Chloroform



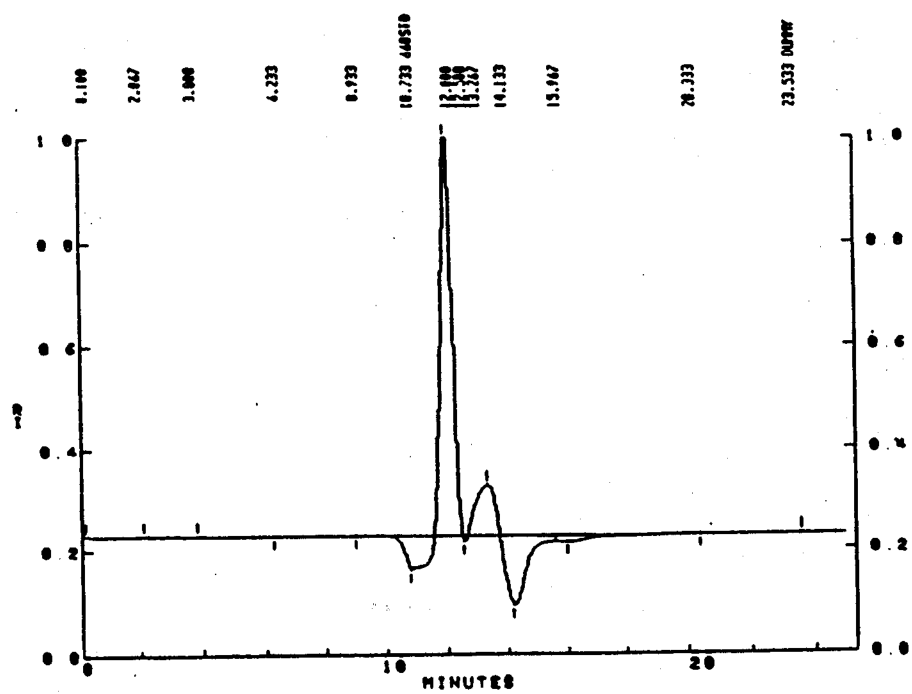
Polypropyleneglycol Diol - Aged Oil, 150°C (Tube CJ-02)

## FIGURE B-11

Size Exclusion Chromatogram: PPG Diol/R-134a, 14 days at 175°C



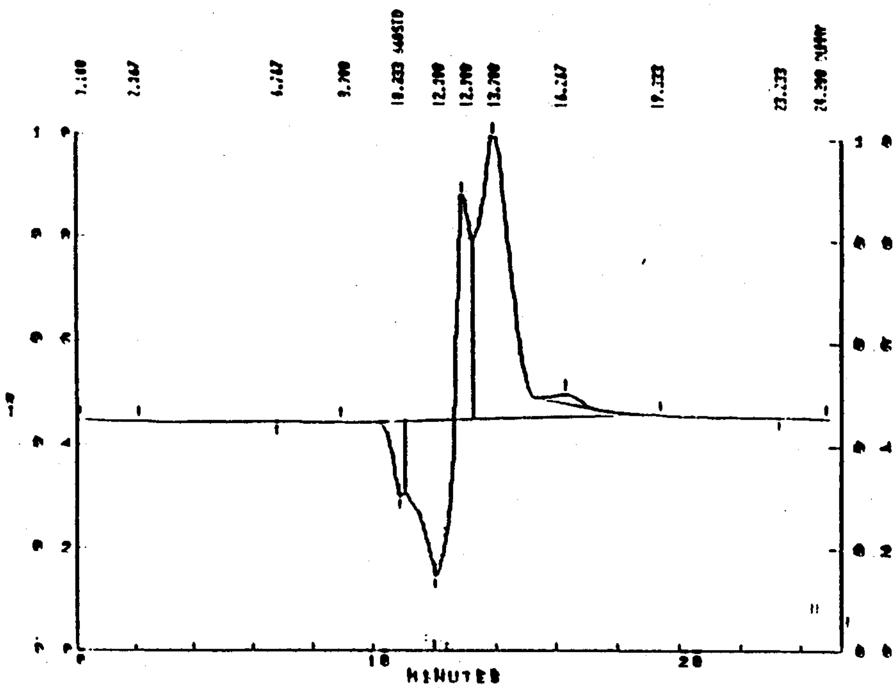

Polypropyleneglycol Diol - Unused Oil
SEC Scan in Chloroform



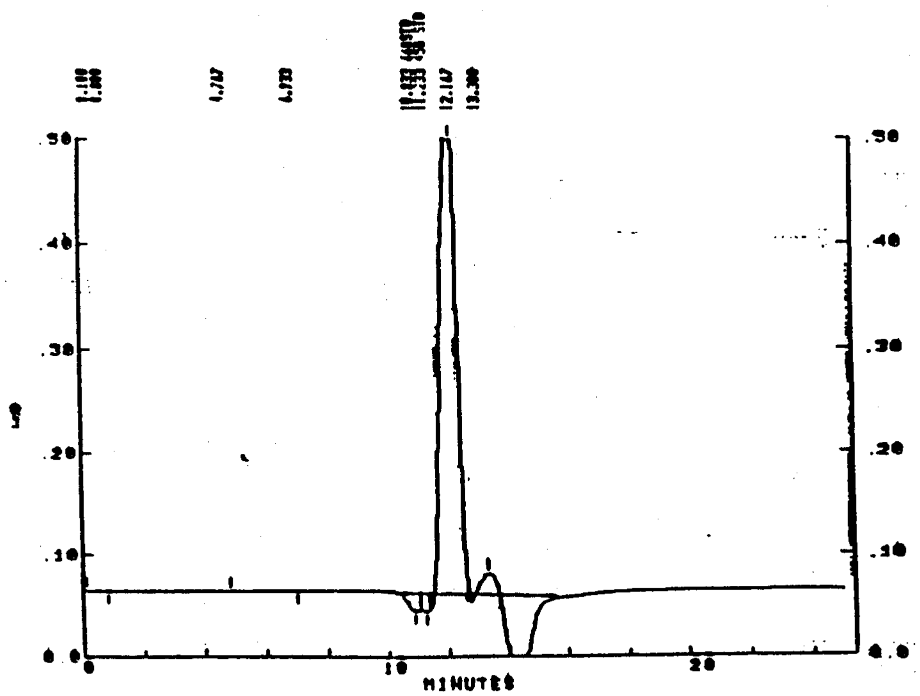

Polypropyleneglycol Diol - Aged Oil, 175°C (Tube CJ-12)

FIGURE B-12

Size Exclusion Chromatograms: PPG Diol/R-134a, 14 days at 200°C



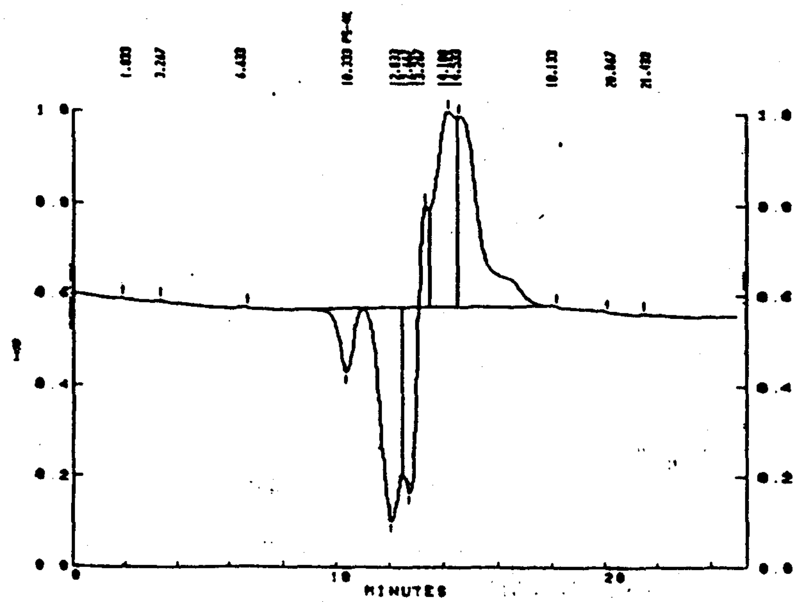

Polypropyleneglycol Diol - Unused Oil
SEC Scan in Chloroform



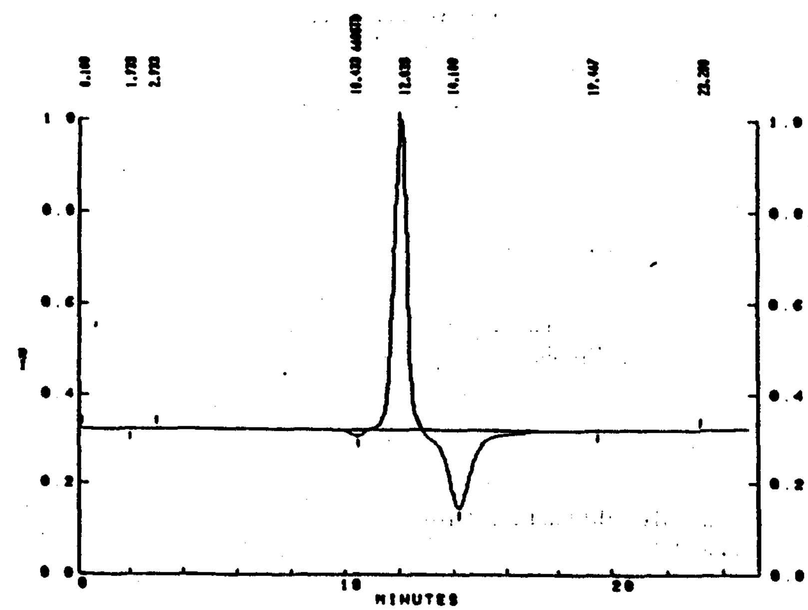

Polypropyleneglycol Diol - Aged Oil, 200°C (Tube CJ-22)

<u>FIGURE B-13</u>

Size Exclusion Chromatograms: Modified Polyglycol/R-134a, 14 days at 150°C



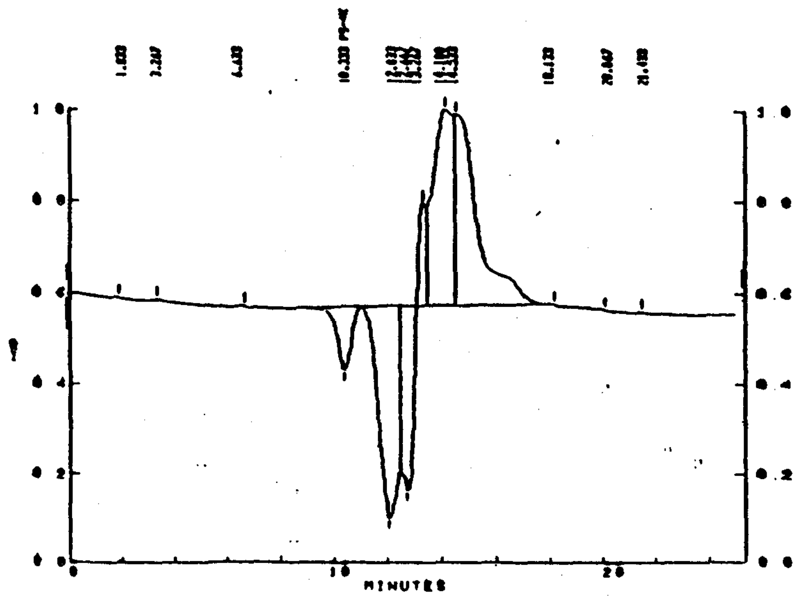

**Modified Polyglycol - Unused Oil**
**SEC Scan in Chloroform**



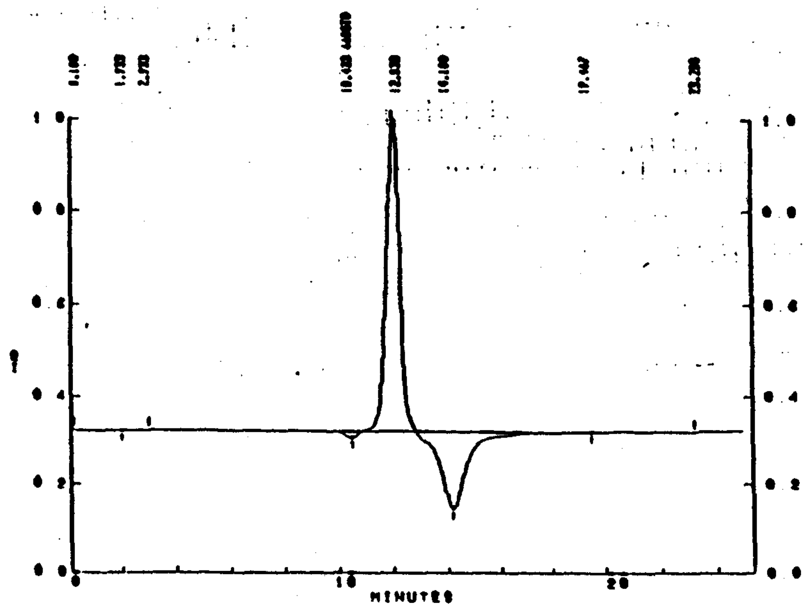

**Modified Polyglycol - Aged Oil, 150°C (Tube CK-02)**

FIGURE B-14

Size Exclusion Chromatograms: Modified Polyglycol/R-134a, 14 days at 175°C



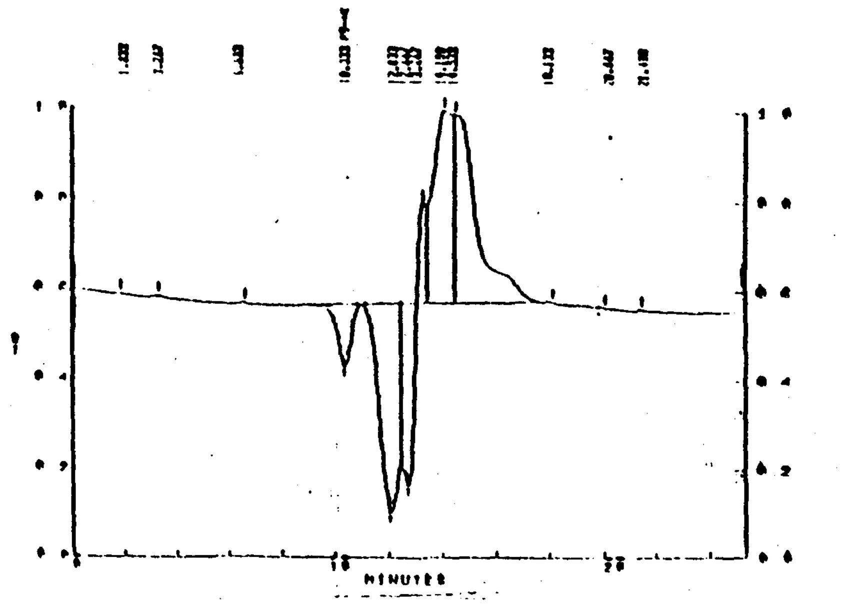

Modified Polyglycol - Unused Oil
SEC Scan in Chloroform



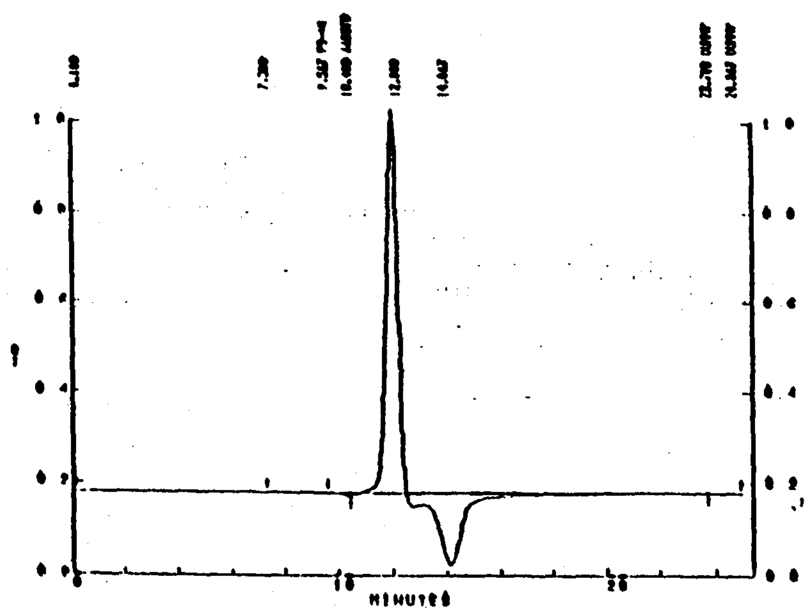

Modified Polyglycol - Aged Oil, 150°C (Tube CR-02)

## FIGURE B-15

Size Exclusion Chromatograms: Modified Polyglycol/R-134a, 14 days at 200°C




Modified Polyglycol - Unused-Oil
SEC Scan in Chloroform

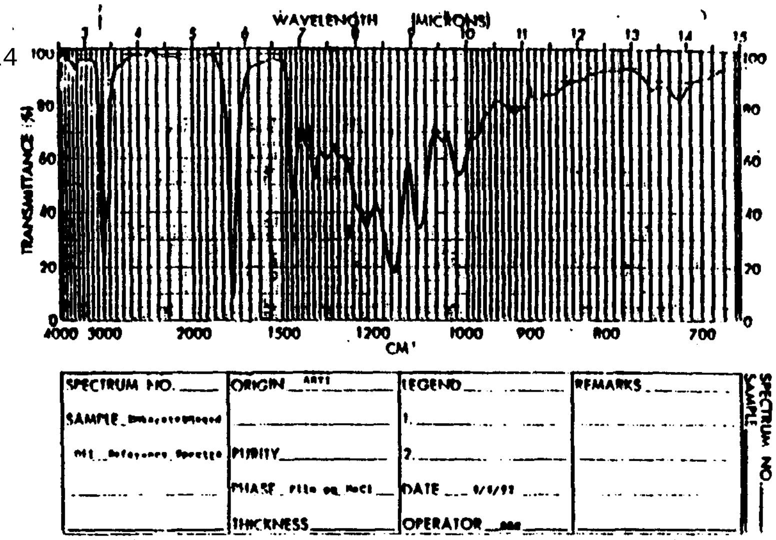



Modified Polyglycol - Aged Oil, 200°C (Tube CR-22)

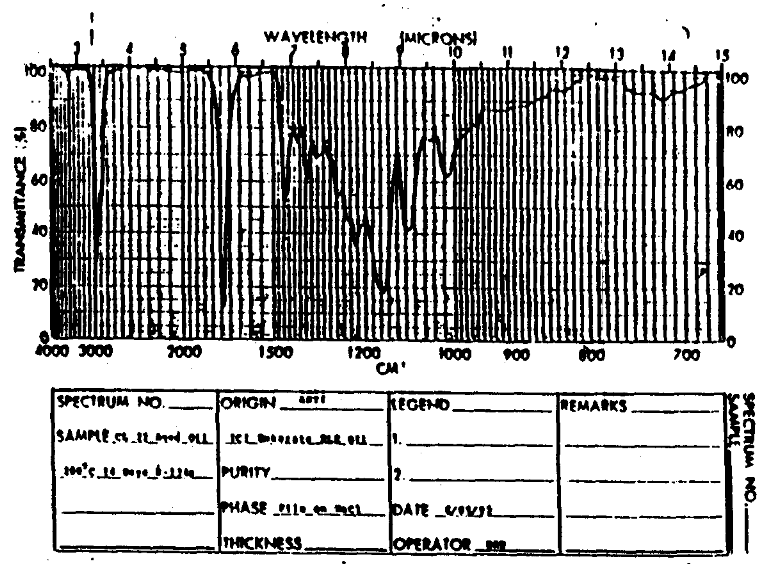
## FIGURE B-16

IR Spectra: Pentaerythritol Ester - mixed acid II/R-134a, 14 days at 200°C

- - Unused Oil




- - Aged Oil, 14 days at 200°C




- - Differential Scan: Aged oil vs. new oil

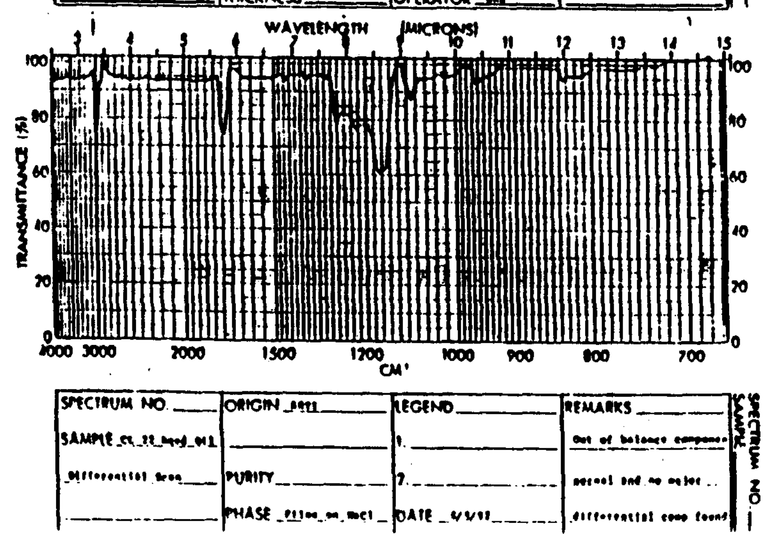
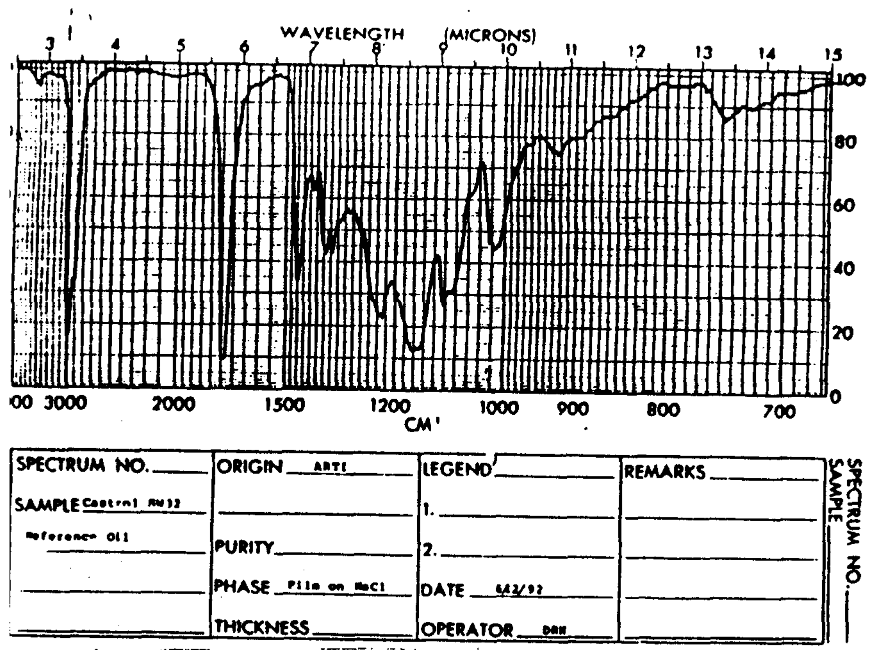


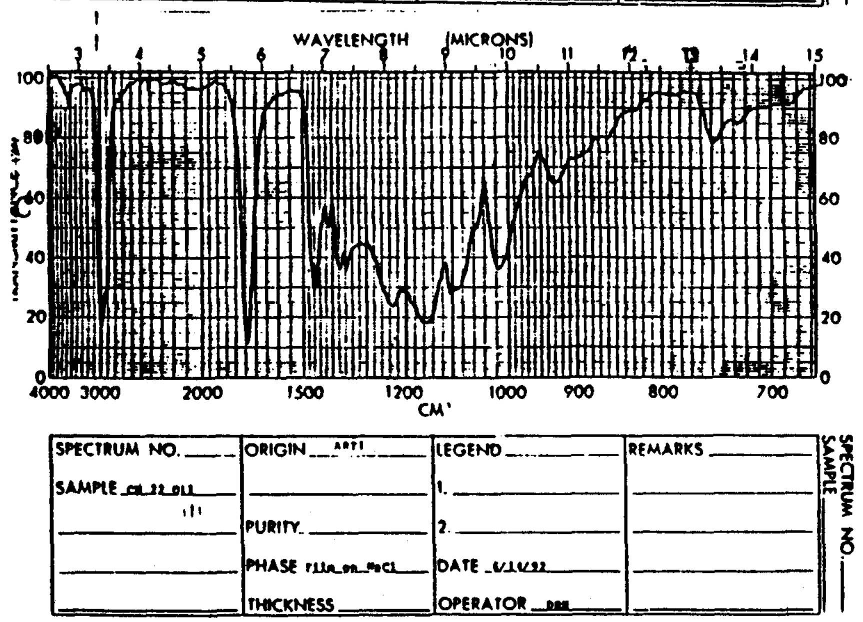
FIGURE B-17

IR Spectra: Pentaerythritol Ester -(mixed acid I)/R-134a 14 days at 200°C

--- Unused Oil




--- Aged Oil, 14 days at 200°C




--- Differential Scan: Aged Oil vs. Unused Oil

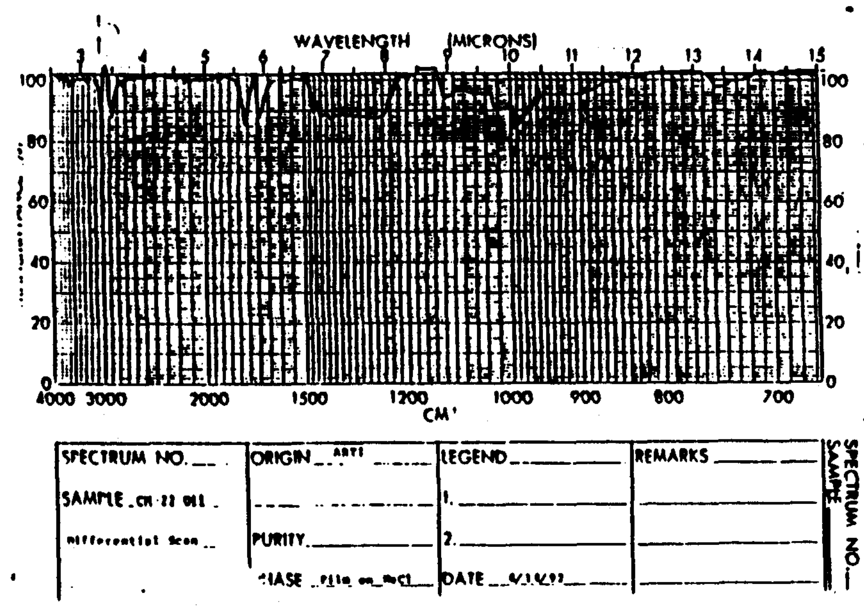
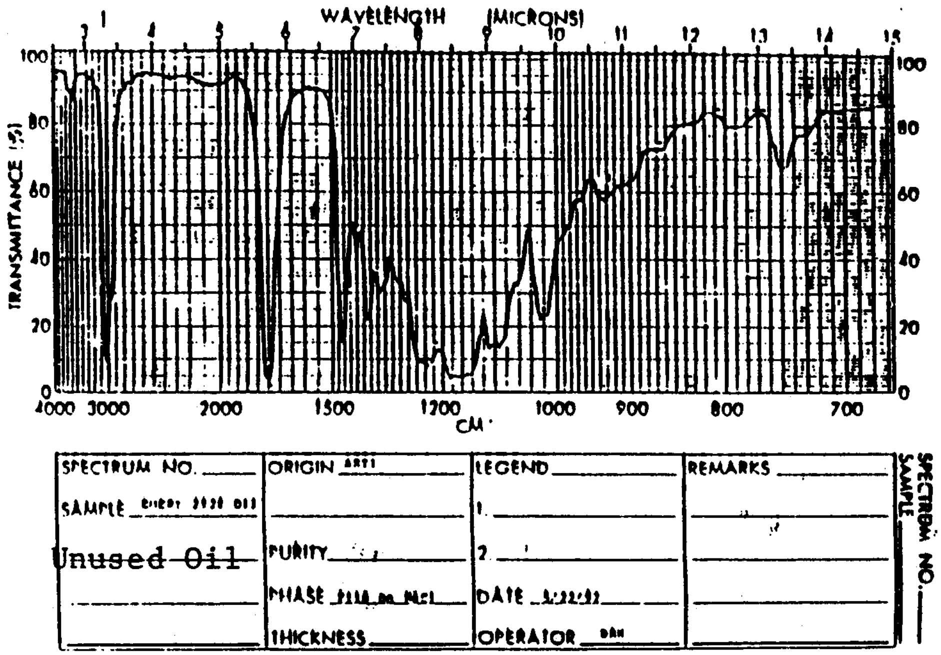


FIGURE B-18

IR Spectra: Pentaerythritol Ester (100 cSt)/R-134a, 14 days at 150°C



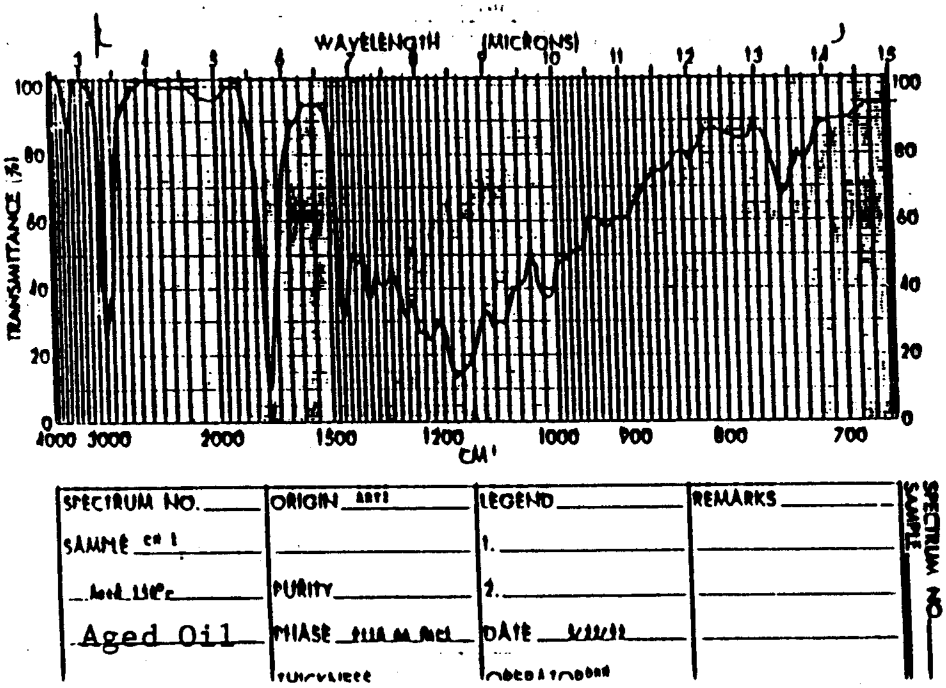



FIGURE B-19

IR Spectra: Pentaerythritol Ester (100 cSt)/R-134a, 14 days at 200°C

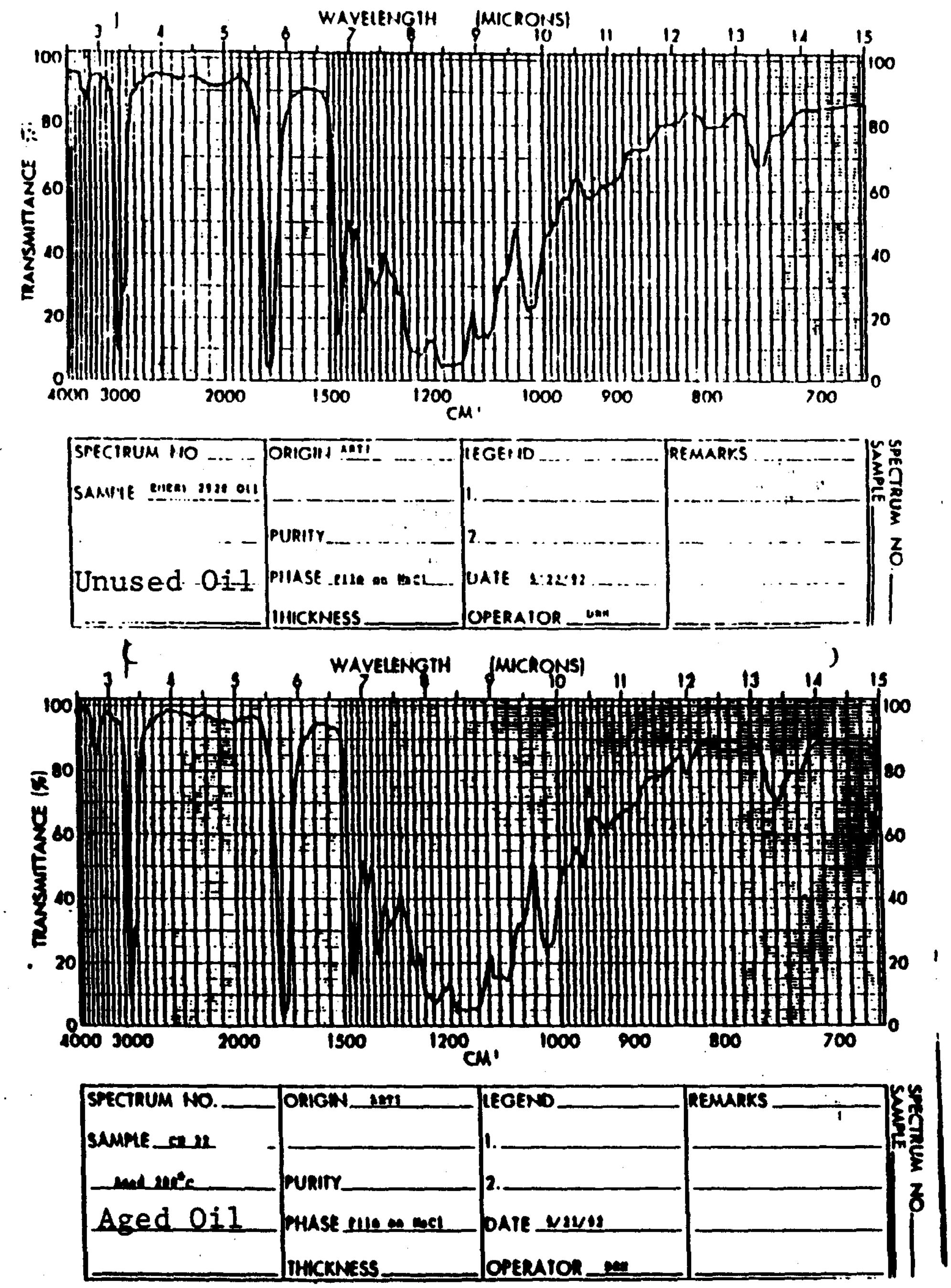
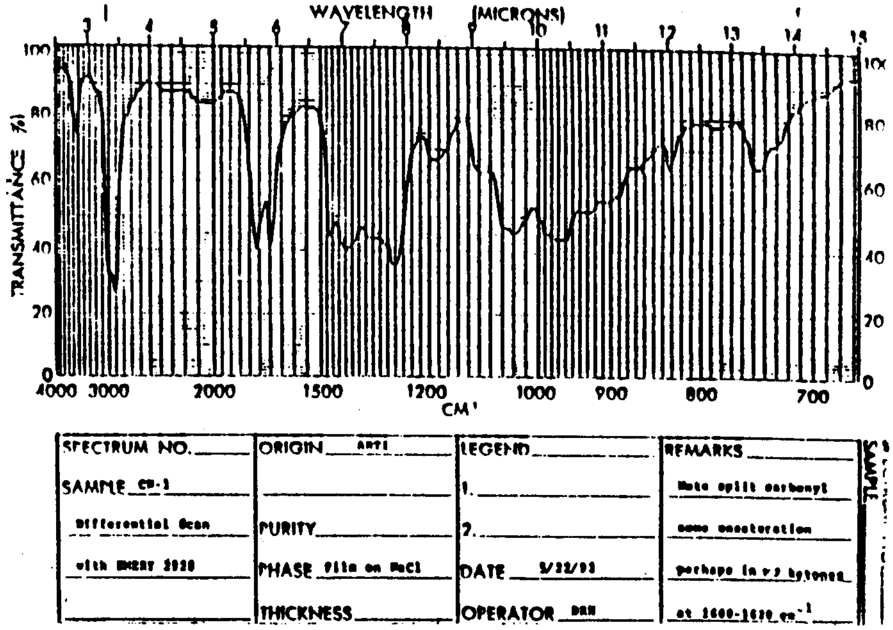


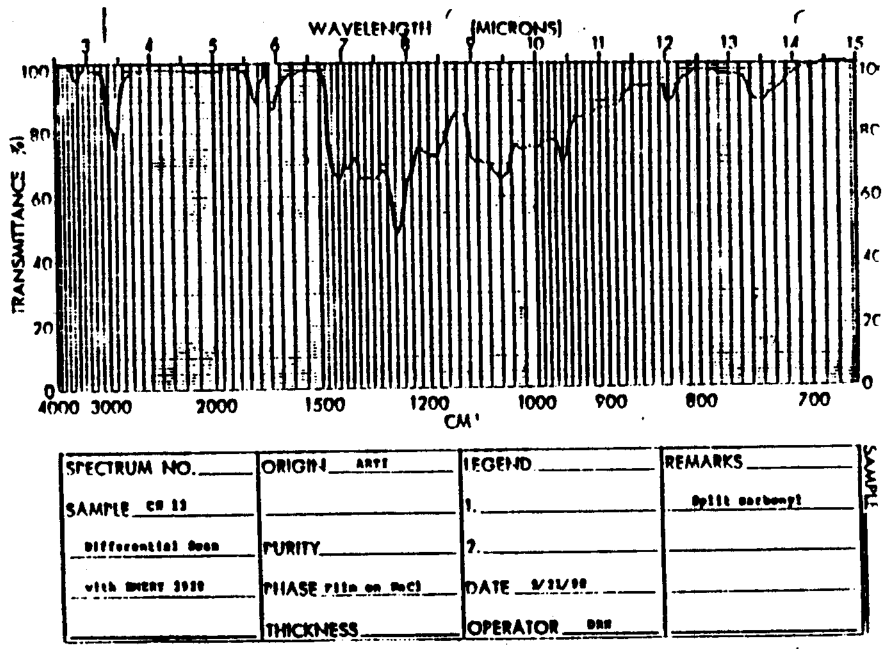
FIGURE B-20

IR Spectra: Differential Scans Pentaerythritol Ester (100 cSt)/R-134a - Aged Oils (150°C, 175°C, 200°C) vs. Unused Oil

- - - 150°C




- - - 175°C




- - - 200°C

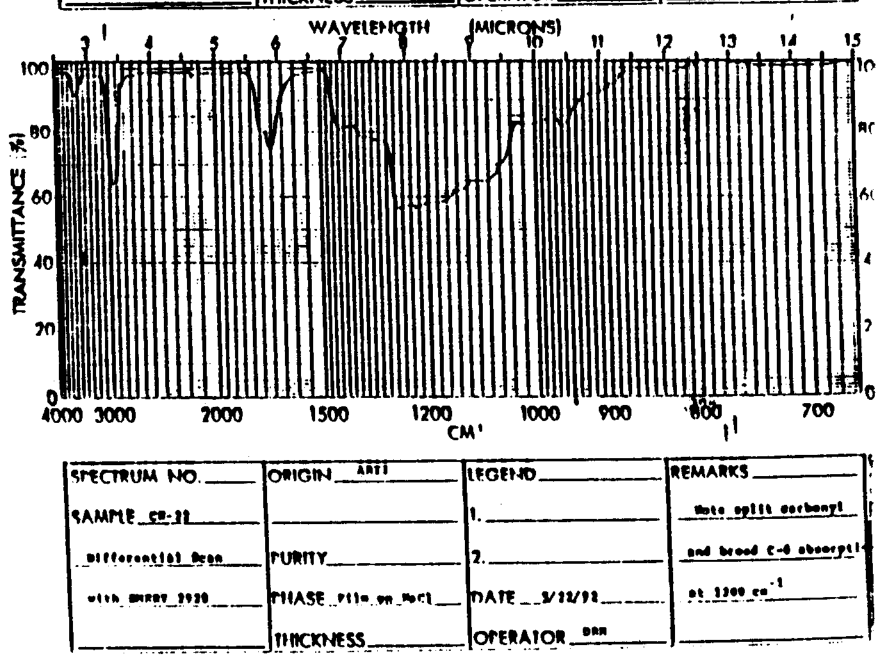
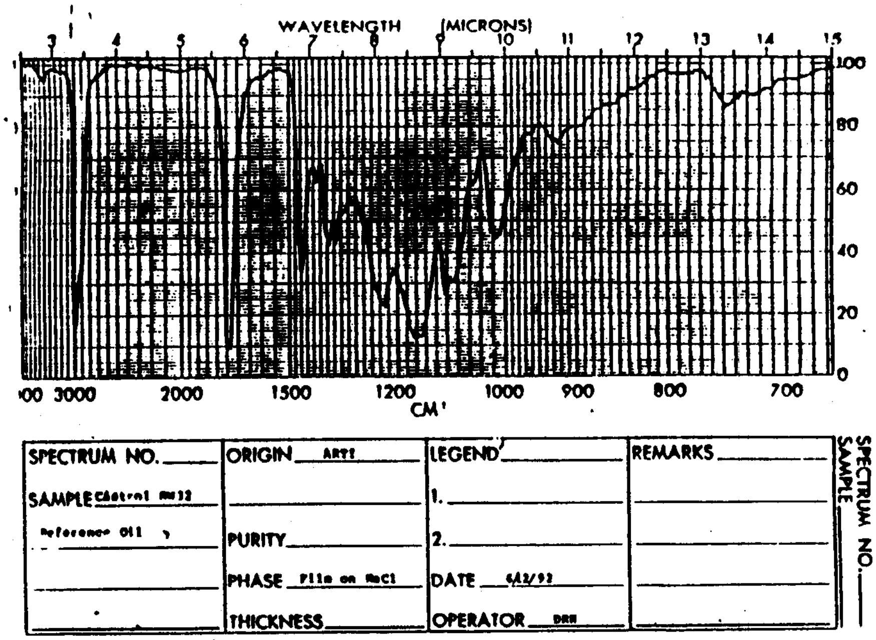


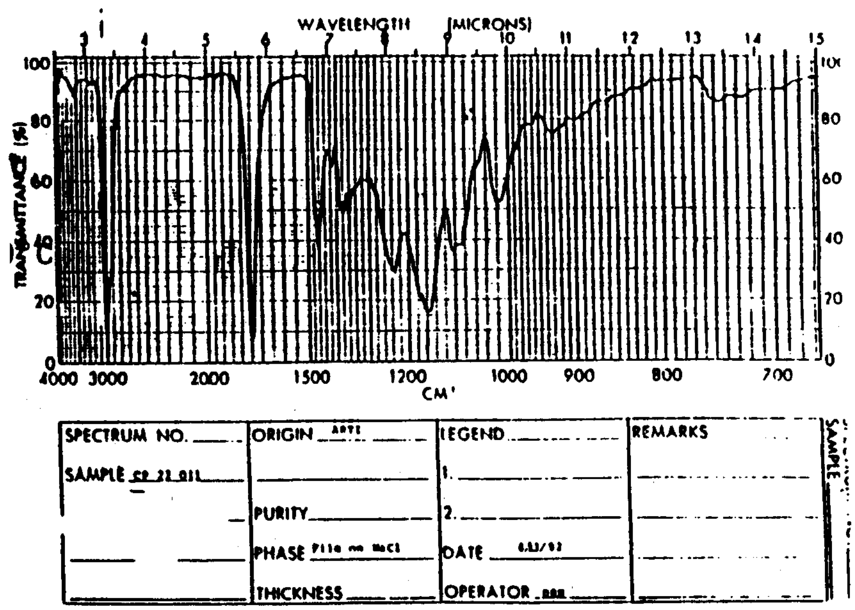
FIGURE B-21

IR Spectra: Pentaerythritol Ester (mixed acid I)/R-143a 14 days at 200°C

- - Unused Oil




- - Aged Oil, 14 days at 200°C




- - Differential Scan: Aged Oil vs. Unused Oil

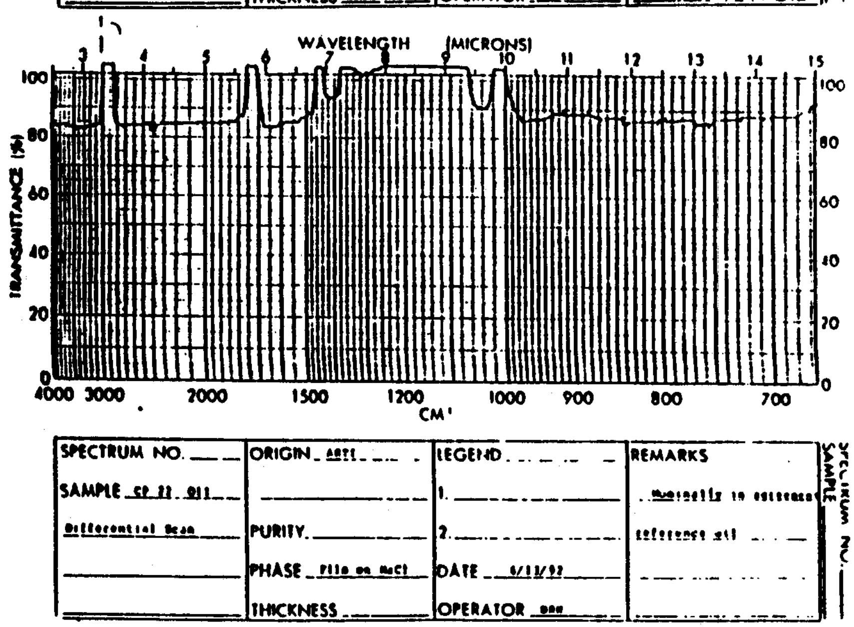
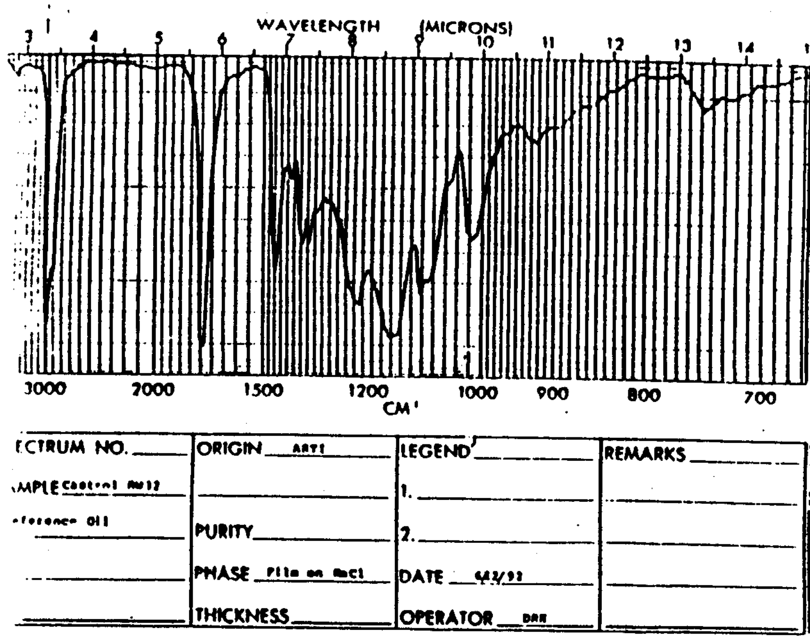


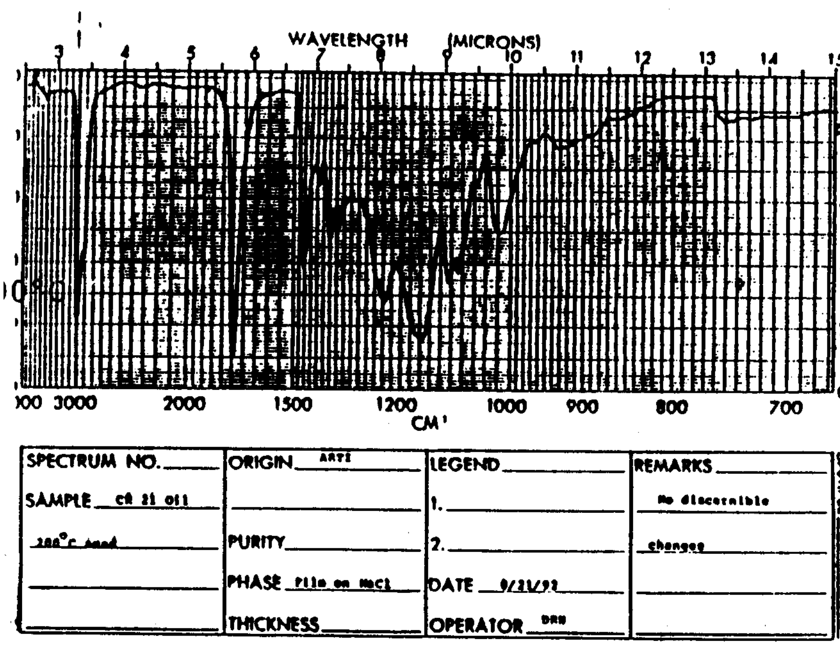
FIGURE B-22

IR Spectra: Pentaerythritol Ester- (mixed acid I)/R-134, 14 days at 200°C

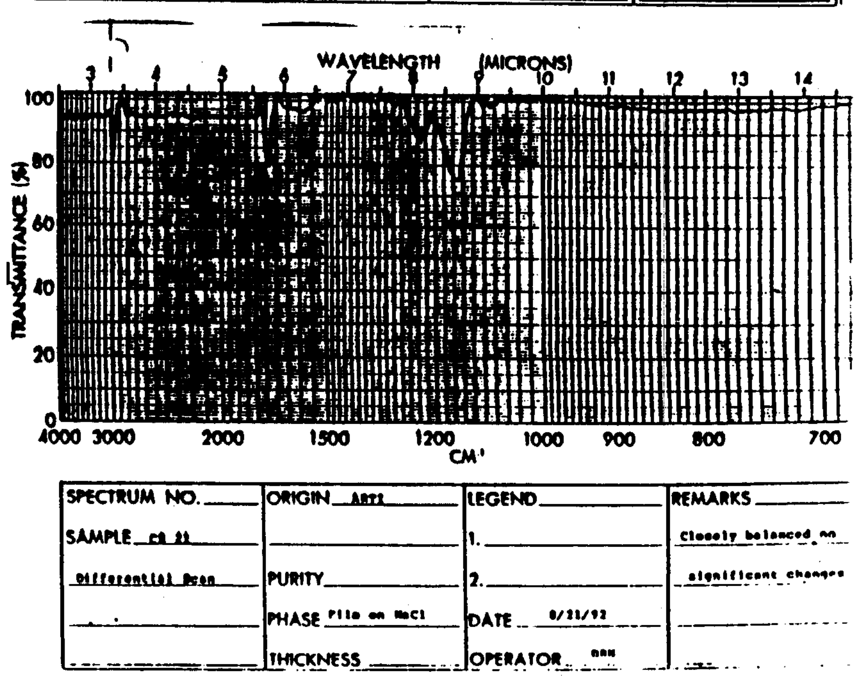
- - Unused Oil




- - Aged Oil, 14 days at 200°C




- - Differential Scan: Aged vs. Unused Oil



# APPENDIX C

APPENDIX C LISTS ALL TEST REFRIGERANTS AND LUBRICANTS USED IN THE COURSE OF THIS PROJECT. GENERIC DESIGNATIONS AND THEIR COMMERCIAL EQUIVALENTS ARE CROSS REFERENCED, WHERE APPLICABLE. ALSO DOCUMENTED IN APPENDIX C IS ALL INFORMATION DEVELOPED ON THE PURITY LEVEL OF THE MATERIALS USED.

TABLE C-1: TEST REFRIGERANTS

THE PURITY OF ALL REFRIGERANTS USED WAS DETERMINED BY GC ANALYSIS OF LIQUID PHASE REFRIGERANT SAMPLES. ABSENCE OF ANY MEASURABLE QUANTITIES OF CONTAMINANT GASES, EXCEPT FOR THE SMALL AMOUNTS OF AIR FROM INADVERTENT LEAKS IN THE GAS HANDLING SYSTEM IS INDICATED IN THE TABLE BY THE NOTATION "PURE MATERIAL".

| REFRIGERANT | GC RESULTS | REFERENCE CHROMATOGRAM |
|---|---|---|
| R-11 | PURE MATERIAL | FIG. C-O1 |
| R-22 | PURE MATERIAL | FIG. C-02 |
| R-32 | PURE MATERIAL | FIG. C-03 |
| R-123 | PURE MATERIAL | FIG. C-04 |
| R-124 | PURE MATERIAL | FIG. C-05 |
| R-125 | UNIDENTIFIED IMPURITY AT <0.5% | FIG. C-06 |
| R-134A | PURE MATERIAL | FIG. C-07 |
| R-134 | POSSIBLE IMPURITY ELUTING AFTER R-134 | FIG. C-08 |
| R-142B | PURE MATERIAL | FIG. C-09 |
| R-143A | $CO_2$ (?) IMPURITY | FIG. C-10 |
| R-152A | PURE MATERIAL | FIG. C-11 |

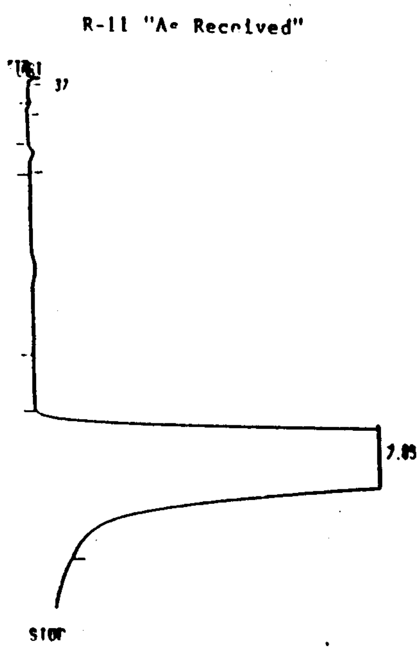
TABLE C-2: TEST LUBRICANTS

| GENERIC IDENTIFICATION | COMMERCIAL SOURCE | COMMERCIAL DESIGNATION | TOTAL ACID NO. (MG KOH/G) | WATER CONTENT (PPM) | METAL CONTENT (PPM) | |
|---|---|---|---|---|---|---|
| | | | | | CU | FE |
| MINERAL OIL (ISO VG32) - NAPHTHENIC | WITCO CHEMICAL | SUNISO 3GS | 0.001 | 18 | 1 | 0 |
| MINERAL OIL (ISO VG46) - NAPHTHENIC WHITE OIL | WITCO CHEMICAL | FREEZENE-NAPHTHENIC HEAVY | 0.11 | 15 | 2 | <1 |
| ALKYLBENZENE | SHRIEVE CHEMICAL | ZEROL 150 | <0.001 | 17 | 1 | 0 |
| POLYPROPYLENE GLYCOL BUTYL-MONOETHER | ICI | EMKAROX (ISO VG32 | 0.05 | 23# | <1 | <1 |
| MODIFIED POLY-GLYCOL (PROPRIET. COMPOSITION) | ALLIED-SIGNAL | BRL-150 | 0.13 | 21# | <1 | <1 |
| POLYPROPYLENE GLYCOL DIOL | DOW CHEMICAL | P 425 (ISO VG22) | 0.07 | 16# | <1 | <1 |
| PENTAERYTHRITOL ESTER (MIXED ACID I) | CASTROL | ICEMATIC SW32 (ISO VG32) | 0.02 | 24# | <1 | <1 |
| PENTAERYTHRITOL ESTER (MIXED ACID II) | ICI | EMKARATE RLE (ISO VG22) | 0.08 | 20# | <1 | <1 |
| PENTAERYTHRITOL ESTER (100 cSt) | HENKEL-EMERY | EMERY 4078X (2928 ISO VG 100) | 0.10 | 18# | <1 | <1 |

# = AFTER VACUUM DEHYDRATION

FIGURE C-01

Gas Chromatogram of
R-11 as Received




RUN # 10 JUN/24/92 17:10:40

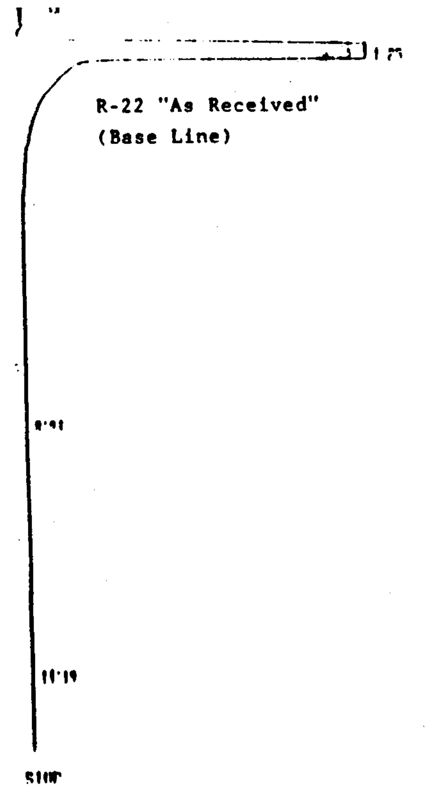
AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.37 | 900 | BB | 0.052 | 0.053 |
| 7.05 | 1698000 | PB | 0.920 | 99.947 |

TOTAL AREA= 1698900
MUL FACTOR= 1.0000E+00

FIGURE C-02

Gas Chromatogram of
R-22 as Received




RUN # 11 JAN/16/92 14:57:36

AREA%

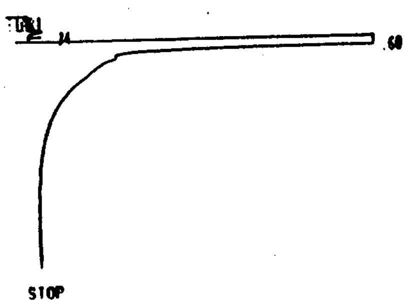
| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.33 | 943 | BB | 0.053 | 0.035 |
| 1.[illegible] | 2726000 | PB | 0.155 | 99.965 |

TOTAL AREA= [illegible]
MUL FACTOR= 1.0000E+00

## FIGURE C-03

Gas Chromatogram of
R-32 as received

R-32 "As Received"




RUN # 2    FEB/19/92 08:56:24

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 1063 | BB | 0.053 | 0.045 |
| 9.60 | 2367700 | PB | 0.080 | 99.955 |

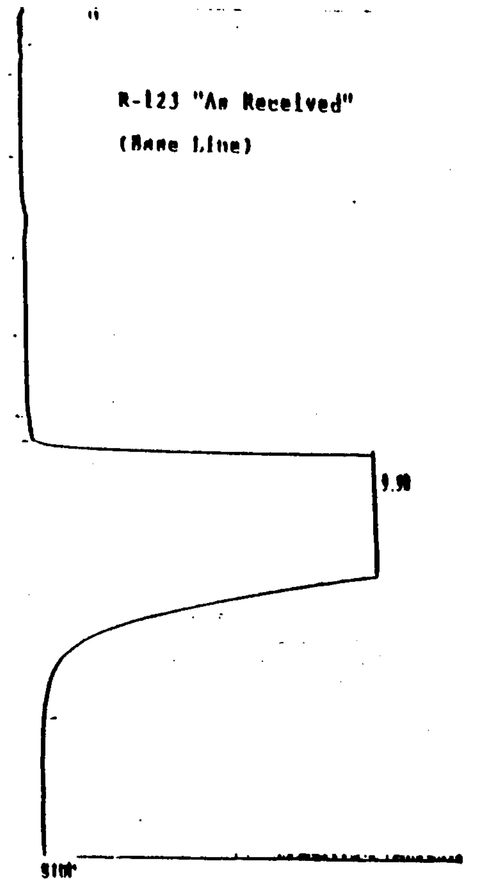
TOTAL AREA= 2368890
MUL FACTOR= 1.0000E+00

## FIGURE C-04

Gas Chromatogram of
R-123 as received

R-123 "As Received"

(Base Line)




RUN # 14    JAN/16/92 15:29:30

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 4060 | BB | [illegible] | [illegible] |
| 9.90 | 3051000 | BB | 1.711 | [illegible] |

TOTAL AREA= 3055090
MUL FACTOR= 1.0000E+00

FIGURE C-05

Gas Chromatogram of
R-124 as received

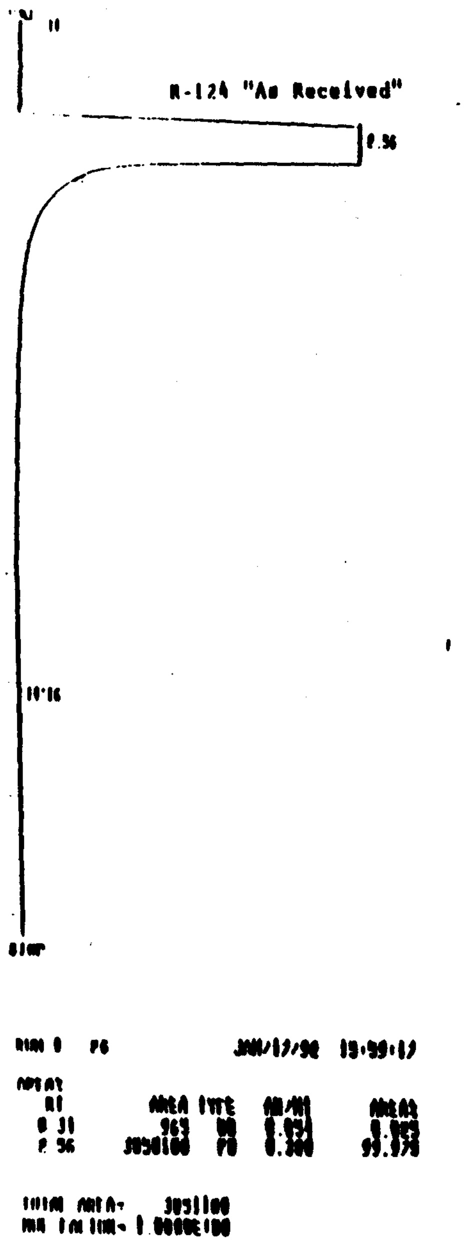



FIGURE C-06

Gas Chromatogram of
R-125 as Received

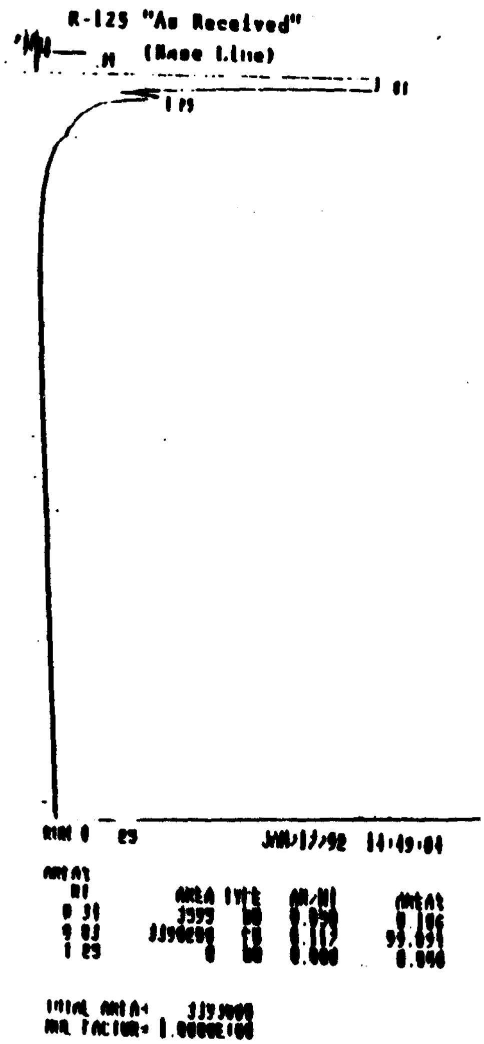
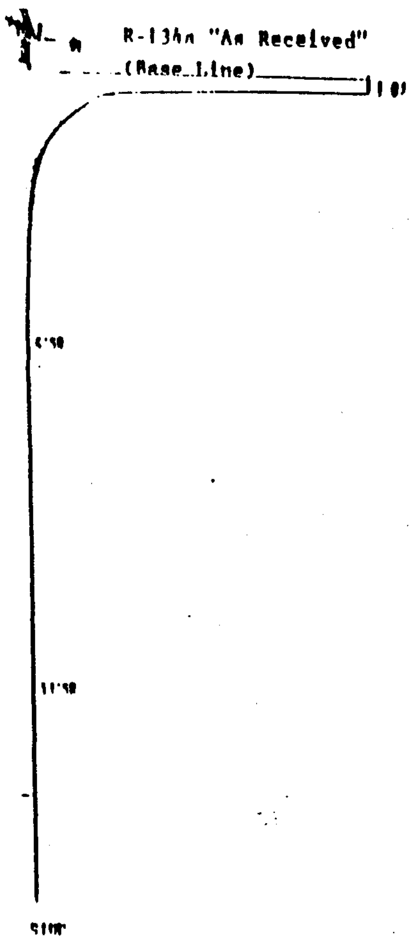


FIGURE C-07

Gas Chromatogram of
R-134a as Received




RUN # 11 JAN/10/92 14:02:49

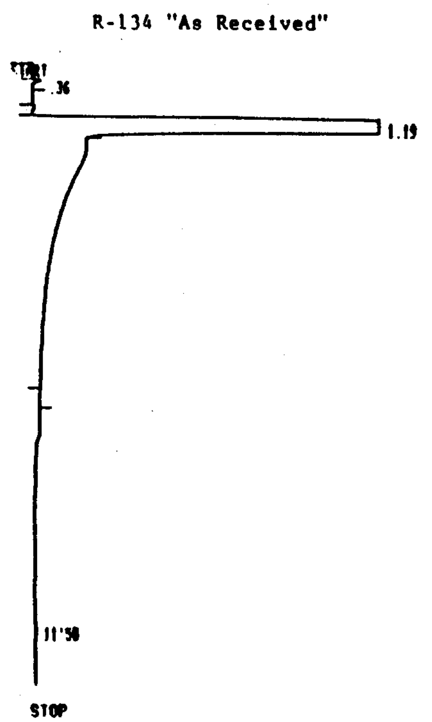
AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.31 | 1012 | BB | 0.052 | 0.053 |
| 1.07 | [illegible] | PB | 0.140 | 99.935 |

TOTAL AREA= [illegible]
MUL FACTOR= 1.0000E+00

FIGURE C-08

Gas Chromatogram of
R-134 as Received




RUN # 7 JUN/11/92 08:26:36

AREA%

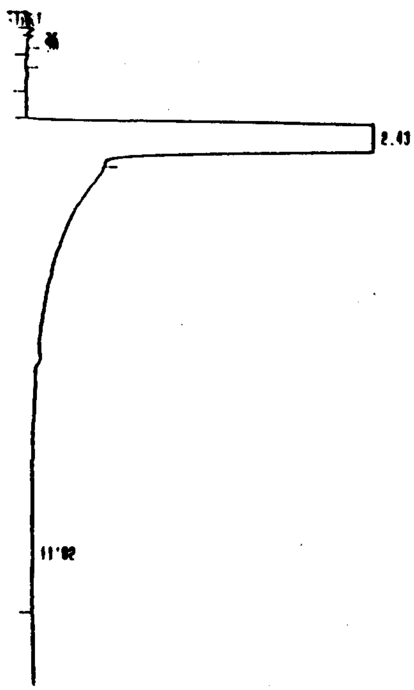
| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.36 | 666 | PB | 0.056 | 0.035 |
| 1.19 | 1904200 | PB | 0.133 | 99.965 |

TOTAL AREA= 1904900
MUL FACTOR= 1.0000E+00

## FIGURE C-09

Gas Chromatogram of
R-142b as Received

R-142b "As Received"




RUN # 8 JUN/11/92 08:42:05

AREA%

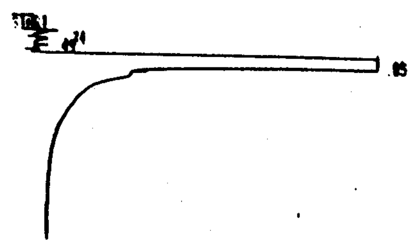
| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.36 | 635 | BP | 0.050 | 0.025 |
| 0.50 | 555 | PB | 0.054 | 0.022 |
| 2.43 | 2519200 | PB | 0.206 | 99.953 |

TOTAL AREA= 2520400
MUL FACTOR= 1.0000E+00

## FIGURE C-10

Gas Chromatogram of
R-143a as Received

R-143a "As Received"




RUN # 1 FEB/19/92 08:44:10

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 1611 | BP | 0.049 | 0.050 |
| 0.49 | 1064 | PB | 0.057 | 0.035 |
| 0.85 | 3070100 | PB | 0.114 | 99.913 |

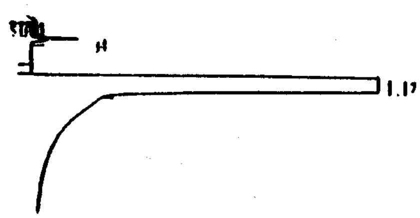
TOTAL AREA= 3072000
MUL FACTOR= 1.0000E+00

Note: 0.49 min peak is probably $CO_2$.

<u>FIGURE C-11</u>

Gas Chromatogram of
R-152a as Received

**R-152a "As Received"**




STOP

RUN # 10 MAR/04/92 10:22:50

AREA%

| RT | AREA | TYPE | AR/HT | AREA% |
|---|---|---|---|---|
| 0.34 | 3322 | BB | 0.050 | 0.134 |
| 1.17 | 2468900 | PB | 0.141 | 99.866 |

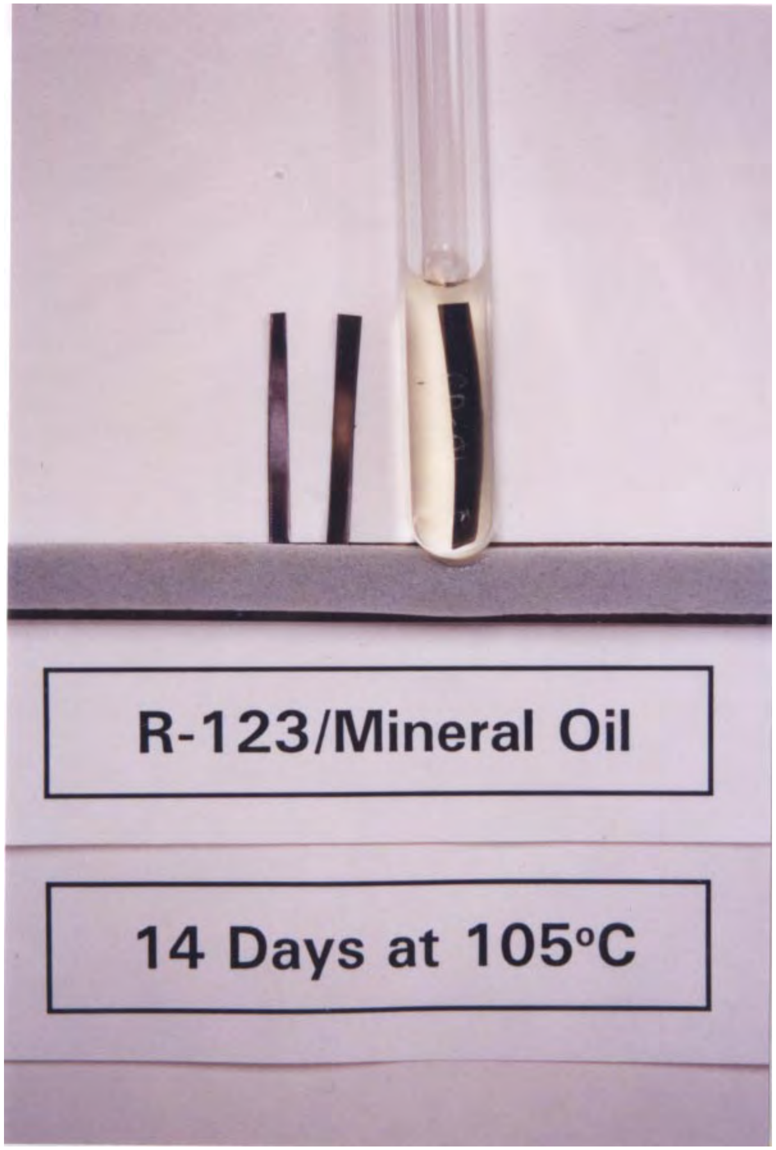
TOTAL AREA= 2472300
MUL FACTOR= 1.0000E+00

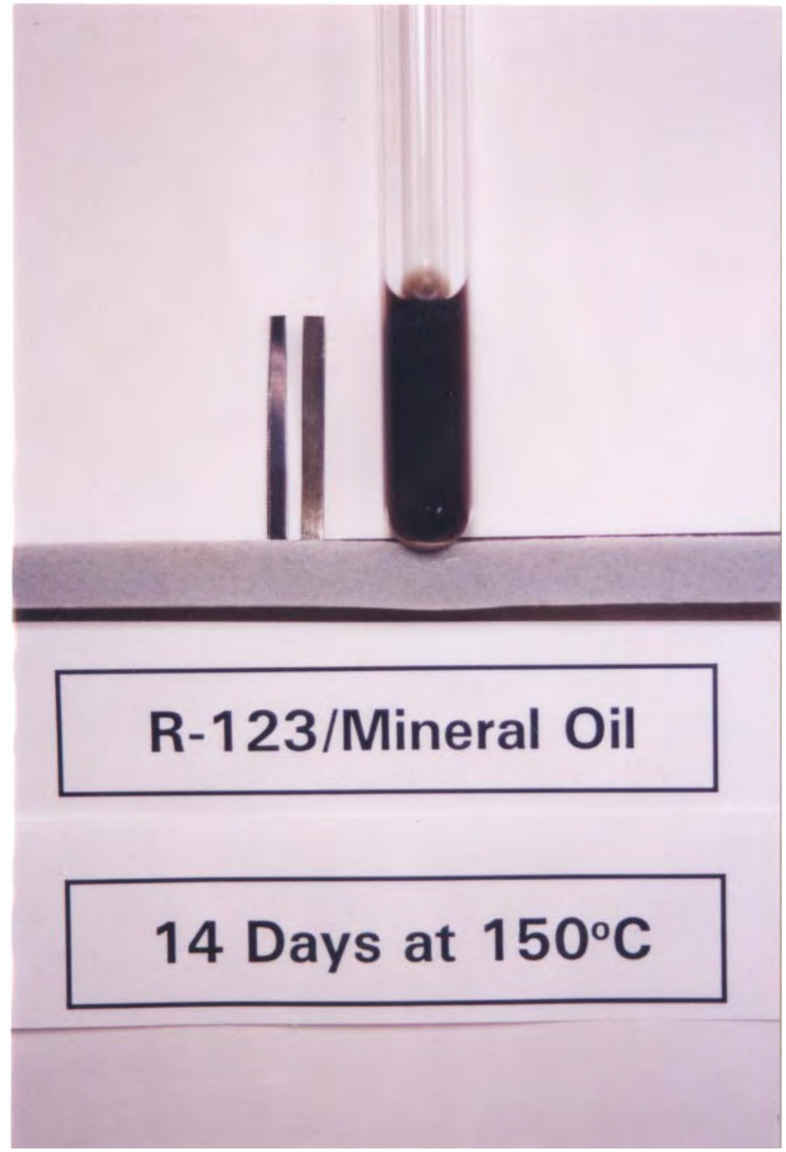
# APPENDIX D

# TABLE OF CONTENTS

APPENDIX D CONTAINS COPIES OF PHOTOGRAPHS FROM THOSE SEALED TUBE SETS THAT EXHIBITED SIGNIFICANT VISUAL CHANGES AFTER THE THERMAL AGING OF THE RESPECTIVE REFRIGERANT-OIL MIXTURES, AS FOLLOWS:

FIGURE D-1


R-123/Mineral Oil
14 Days at 105°C


R-123/Mineral Oil
14 Days at 150°C


R-123/Mineral Oil
14 Days at 175°C

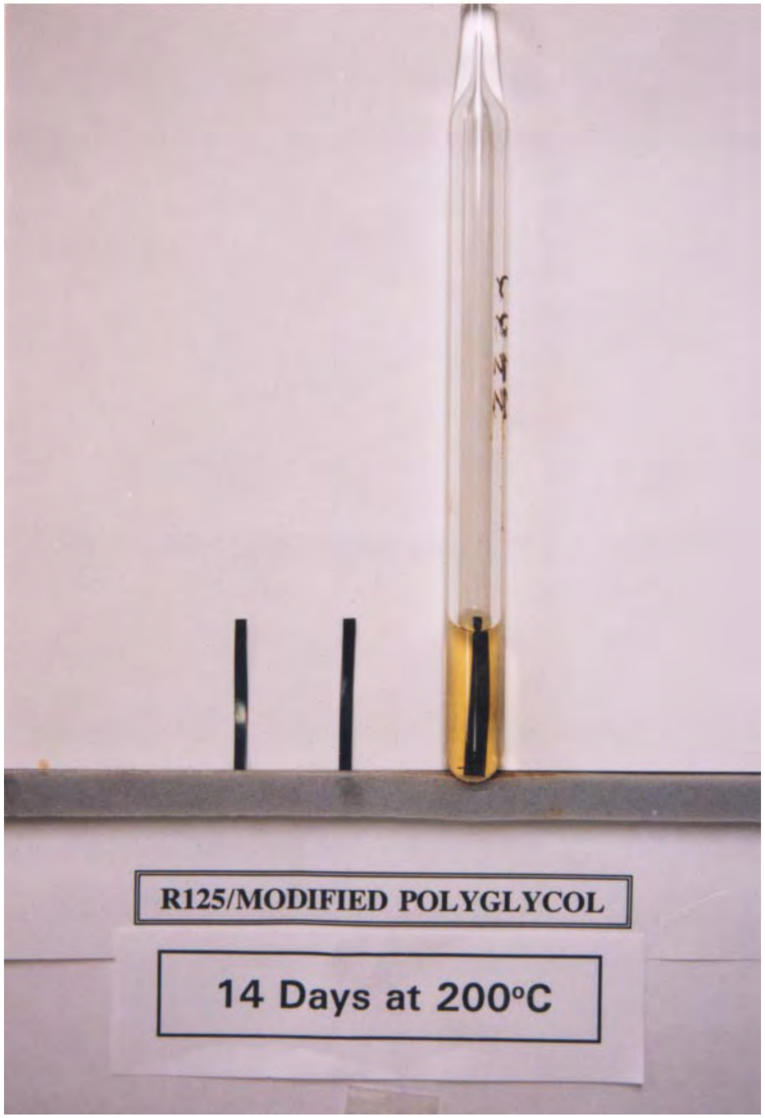

R-123/Mineral Oil
14 Days at 160°C

**FIGURE D-2**


R-124/Alkylbenzene
14 Days at 200°C

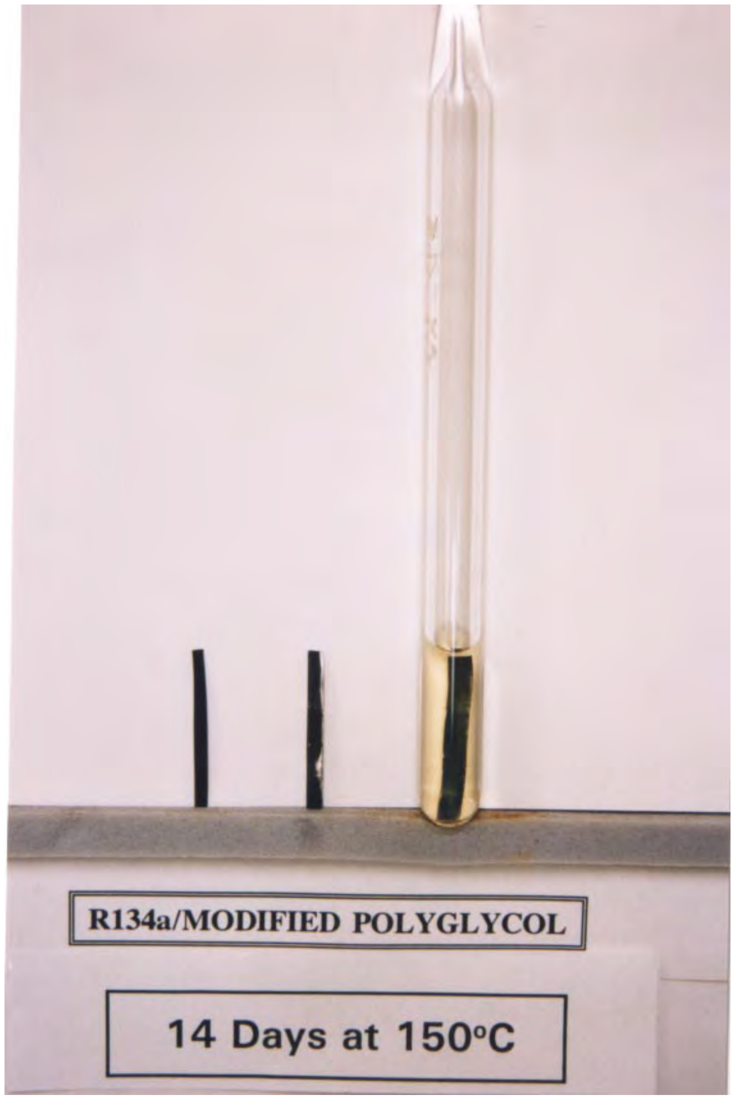

R-124/Alkylbenzene
14 Days at 175°C

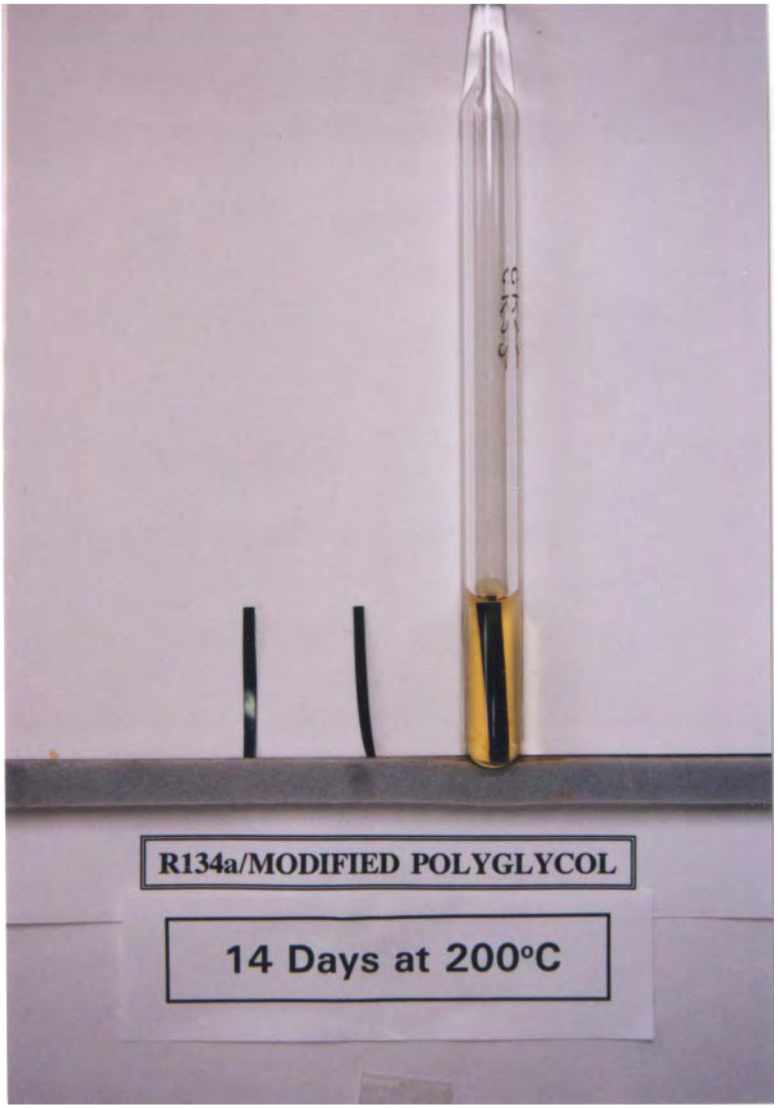
**FIGURE D-3**


R125/MODIFIED POLYGLYCOL
14 Days at 200°C


CG-12
R125/MODIFIED POLYGLYCOL
14 Days at 175°C

## FIGURE D-4




R134a/MODIFIED POLYGLYCOL
14 Days at 200°C

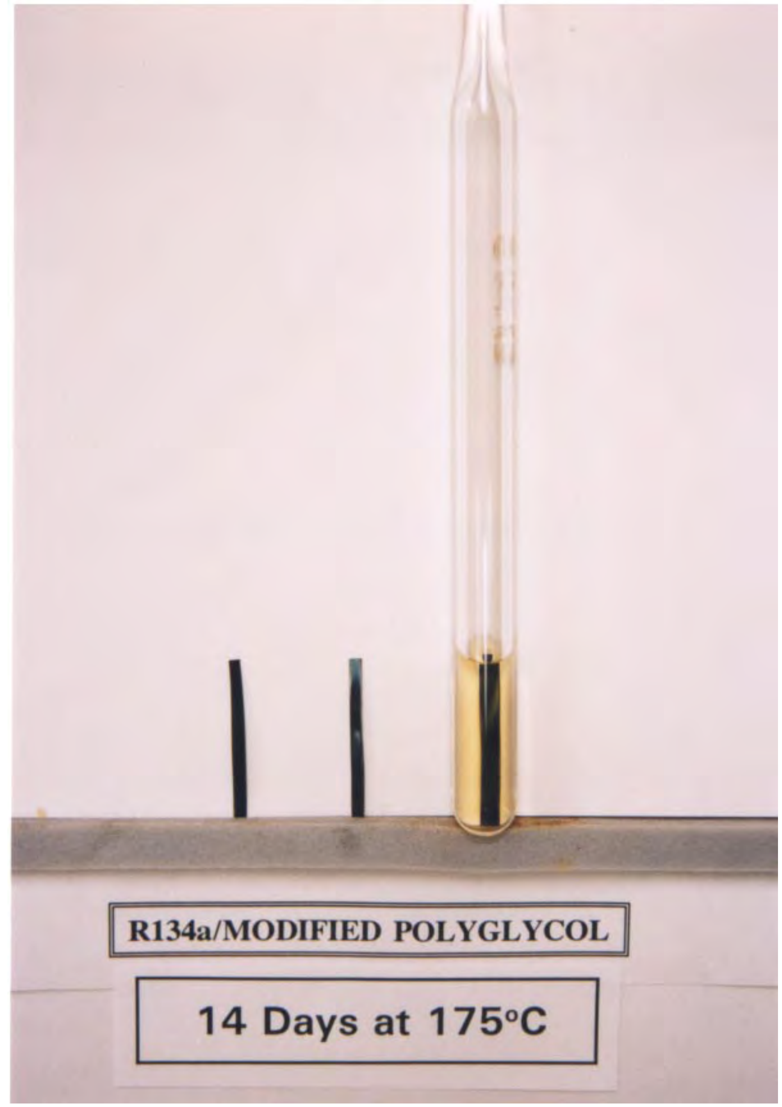
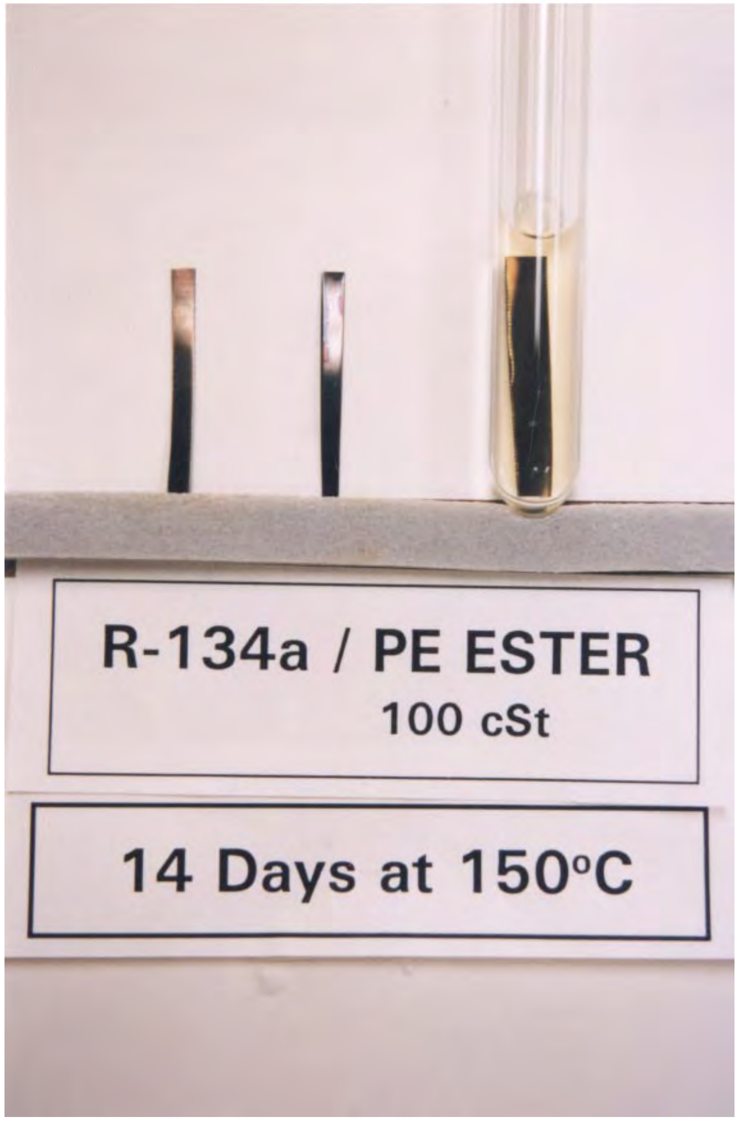

R134a/MODIFIED POLYGLYCOL
14 Days at 175°C

**FIGURE D-5**




R-134a / PE ESTER
100 cSt
14 Days at 200°C

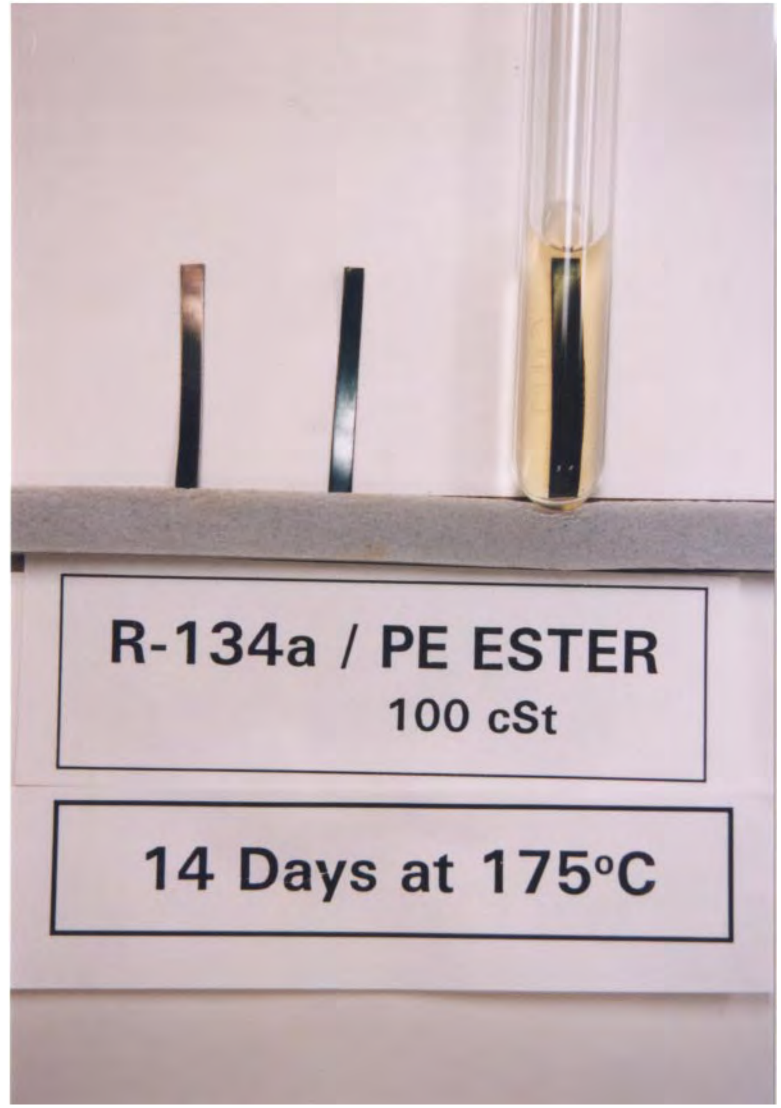

R-134a / PE ESTER
100 cSt
14 Days at 175°C

**FIGURE D-6**


R-11/Mineral Oil
ISO VG32
14 Days at 160○C


R-11/Mineral Oil
ISO VG32
14 Days at 150°C

**FIGURE D-7**


R-11/HEAVY NAPHTHENIC
14 Days at 160○C


R-11/HEAVY NAPHTHENIC
14 Days at 150°C

**FIGURE D-8**


R-123/HEAVY NAPHTHENIC
14 Days at 160○C


R-123/HEAVY NAPHTHENIC
14 Days at 150°C