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Chemical Stability Investigations of Ultra-Low GWP Refrigerants R-1336mzz(Z), R-1336mzz(E), R-514A, R-1233zd(E), and R-1224yd(Z) with Lubricants

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ABSTRACT

AHRTI (Air-Conditioning Research Technology Institute) sponsored a research program on developing essential low global warming potential (GWP) refrigerant database in collaboration with Oak Ridge National Laboratory (ORNL) and National Institute of Standards and Technology (NIST). In 2019, AHRTI received a US Department of Energy Building Technology Office funding award to carry out this research program. As part of the overall effort, Trane Technologies conducted AHRTI Project 9016 – Chemical Stability of Low GWP Refrigerants with Lubricants.

This paper summarizes the results of highly accelerated life tests (HALT), conducted according to ASHRAE Standard 97 sealed glass tube methodology, for refrigerants R-1336mzz(Z), R-1336mzz(E), R-514A, R-1233zd(E), and R-1224yd(Z) with and without lubricants. R-1233zd(E) and R-1224yd(Z) were evaluated with mineral oil and the rest were evaluated with polyalkylene glycol (PAG), polyolester (POE) and polyvinyl ether (PVE).

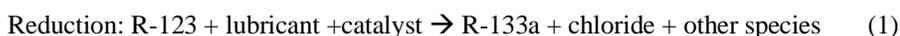
1. INTRODUCTION

Few chemical stability studies exist in literature that describe the comprehensive investigation of R-1336mzz(Z), R-1336mzz(E), R-514A, R-1233zd(E), and R-1224yd(Z) with and without lubricants. The AHRTI 9016 final report provides a literature summary of limited investigations into the stability of these refrigerants (Sorenson et al, 2021). Kujak et al, 2022 provided a summary of the chemical stability data of highly accelerated life tests (HALT), conducted according to ASHRAE Standard 97 sealed glass tube methodology, for refrigerants R-1336mzz(Z), R-1336mzz(E), R-514A, R-1233zd(E), and R-1224yd(Z) with and without lubricants. This paper provides a more detailed examination and discussion of these results. No discussion is provided for the filter drier material exposures in this paper, however little to no reactivity was observed in these experiments, likely as a result of the lower temperature conditions (Table 4).

Olefin or unsaturated (carbon-carbon double bond) refrigerant chemistry introduces the possibility for new and more complicated refrigerant system chemistry as compared to saturated refrigerants. Figure 1 summarizes the chemical structure depiction of the refrigerants studied in this project. R-123 (saturated refrigerant) is shown for reference since it was used as the baseline refrigerant for chemical stability comparisons. Good refrigerant chemical stability in HVACR systems is defined as the ability to perform over a wide range of temperatures, in the presence of materials (necessary for the system operation), with limited deterioration. New refrigerants must have an inherent level of thermal stability before being considered for use in HVACR systems. Once the general thermal stability of the refrigerant alone is established, it is then subjected to a wide range of system materials and potential system containments, like residual air, water, and processing chemicals that could accelerate possible chemical reactions. The refrigerant must not be adversely affected by these materials. Various known and possible refrigerant reaction pathways will be discussed in the following sections.

Reduction and Disproportion Reactions with Olefin Refrigerants

Two types of chemical reactions are common with CFCs, HCFCs, and, to a lesser extent, HFCs, which is the reduction of the refrigerant by lubricant species and disproportionation of refrigerant. Disproportionation reactions will not be addressed as part of this discussion since they are not common given lubricant or other hydrocarbon contaminants are always present in HVACR systems which favors reduction reactions. Reduction reactions are typically thermally initiated with the refrigerant in the presence of oxidizing agent. Reduction reactions involve a direct interchange of chlorine and hydrogen atoms between the refrigerant and the oil (Spauschus and Doderer, 1961). Measurement of the breakdown products formed in the reaction of the refrigerant with the lubricant has become the industry standard method of determining if a material in a system is acceptable for use. The reduction reaction does not always chlorinate the lubricant and aggressive chlorine-based species such as hydrochloric acid can form in systems. Equation 1 shows the reduction reaction for R-123 and the formation of the reduction product R-133a and chloride.



The proton transfer process can be thought of as a simple kind of nucleophilic substitution reaction, which is later discussed, where the halogen is exchanged for a hydrogen. Each molecule of R-123 represents the decomposition of one molecule of R-123. When the chlorine is released, it can then form aggressive chlorine-based species, such as hydrochloric acid, and attack metals and other construction materials. Typically, a lubricant sample can be analyzed for total acid number or chloride to determine how much refrigerant breakdown has occurred in systems, while the refrigerant can be analyzed by gas chromatography to evaluate the formation of reduction product R-133a.

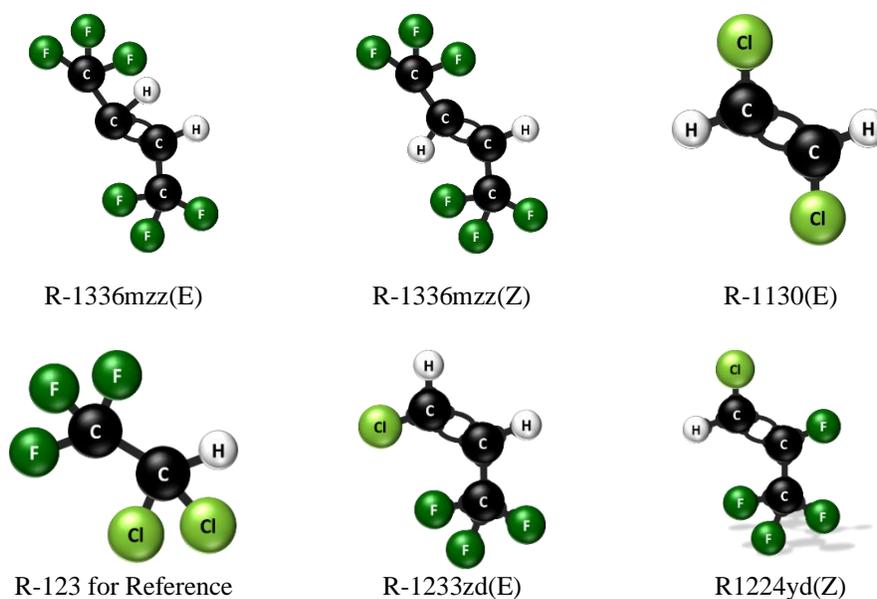


Figure 1: Summary of Olefin Refrigerants Chemical Structures

Unlike the saturated halogenated refrigerants, olefin-based refrigerants have been studied and few refrigerant reduction reactions with the lubricant have been observed in sealed glass tube studies (Majurin et al., 2014, Rohatgi et al., 2012, Fuji-taka, 2010). The reduction products of the various olefins would require the elimination of fluorine or chlorine from the molecule and substitution by hydrogen atom. Table 1 gives summary of potential reduction products if they were to occur with various olefin refrigerants in this study. R-1336mzz(Z and E) do not possess a halogen on the double bond so reduction of a halogen is not possible.

Stereoisomer Rearrangement Reactions with Olefin Refrigerants

Unlike the saturated halogen refrigerants, which are free to rotate into different orientations in three-dimensional space, olefin refrigerants contain a carbon-carbon double bond that does not allow the molecule to rotate freely. This double bond can lead to the formation of stereoisomers with different properties yet the same chemical formulation. Refrigerant nomenclature specifies when there are different stereoisomers by using a designation of “E” (trans, E:

Entgegen (German) meaning opposite) or “Z” (cis, Z: Zusammen (German) meaning together), in the formula. R-1336mzz(E and Z) are examples of stereoisomers. The “Z” isomer has the hydrogen atoms on the same side of the double bond while the “E” isomer has the hydrogen atoms on the opposite side. In general, the “Z” suffix is added to the name if the priority substituents are on the same side of the double bond and the “E” suffix is used when priority substituents are on the opposite sides of the bond. Interestingly, this small change in stereochemistry results in a rather large boiling point difference of $\Delta 26^{\circ}\text{C}$ ($\Delta 47^{\circ}\text{F}$) between the two R-1336mzz’s. In general, trans-alkenes are less polar, more symmetrical and have lower boiling points than cis-alkenes and trans-alkenes in general trans isomers are a more stable lower energy state over cis-alkenes.

Table 1: Summary of Possible Olefin Refrigerant Reduction Products

Refrigerant	Breakdown Product
R-1130(E)	R-1140
R-1336mzz(Z)	Not Applicable
R-1336mzz(E)	Not Applicable
R-1224yd(Z)	R-1234yf or R-1233zd
R-1233zd(E)	R-1243zf

Oligomerization or Polymerization Reactions with Olefin Refrigerants

The presence of a double bond can lead to the potential for oligomerization (small number of monomers joined together) or polymerization (large numbers of monomers joined together) of the olefin refrigerant. Seeton (2018) reported occurrences of R-1234yf polymerization in automotive applications, but it was attributed to the presence of uncured elastomers with residual crosslinking chemistries that lead to the oligomerization/polymerization.

Nucleophilic Substitution Reactions with Olefin Refrigerants

In organic chemistry, nucleophilic substitution is a fundamental class of reactions in which an electron rich nucleophile selectively attacks the positive or partially positive charge of an atom or group of atoms to replace a leaving group. Alkyl halide bonds have electronegativities significantly greater than carbon, with the exception of iodine. This reactivity is based on the covalent bond strength of the carbon and the bonded species’ ability to become a leaving group. The strongest of the carbon-halogen covalent bonds is fluorine. It is roughly 30 kcal/mole stronger than a carbon-carbon bond and about 15 kcal/mole stronger than a carbon-hydrogen bond. As a result, alkyl fluorides and fluorocarbons are generally quite chemically and thermodynamically stable, and do not share any of the reactivity patterns shown by the other alkyl halides. Nucleophilic substitution across a halogenated double bond is possible, but hydrogen, fluorine, and chlorine atoms along with trifluoromethyl ($-\text{CF}_3$) groups attached to the double bond are not favorable leaving groups, whereas bromine and iodine are able to act as favorable leaving groups depending on their bonding characteristics. In addition, the formation of hydrofluoric or hydrochloric acid as the result of limited reduction reactions with olefins is possible, but unlikely to occur because these acids are quickly neutralized by system materials or the filter drier component before they can reach significant concentrations to initiate the reaction. Table 2 shows possible nucleophilic substitution reactions that could occur with the three chlorinated olefins evaluated in this study, R-1130(E), R-1233zd(E), and R-1224yd(Z) where hydrogen and chlorine were substituted across the double bond.

Another example of an olefin reaction is a process called hydrogenation. In a hydrogenation reaction, two hydrogen atoms are added across the double bond resulting in saturated hydrocarbon. Not surprisingly, hydrogenation of a double bond is a thermodynamically favored reaction because it forms a more stable (lower energy) product. Table 2 provides possible hydrogenation reaction products. Although hydrogenation reactions are thermodynamically favorable, they typically will not proceed without a catalyst. The reaction requires first the formation of hydrogen gas followed by the catalysis of the hydrogen-hydrogen bond. Typical catalysts are palladium and platinum.

Table 2: Summary of Possible Olefin Hydrogenation and Chlorine Nucleophilic Substitution Reactions Products

Refrigerant	Hydrogenation Products	Chlorine Substition Product
R-1130(E)	R-150	R-140
R-1233zd(E)	R-253fo	R-243dn & R-243fo
R-1224yd(Z)	R-244eo	R-234bo & R-234en
R-1336mzz (E&Z)	1,1,1,4,4,4 Hexafluorobutane	Not Applicable

2. CHEMICAL STABILITY RESULTS & DISCUSSION

Refrigerants R-1336mzz(Z), R-1336mzz(E), R-514A, R-1233zd(E), and R-1224yd(Z) were evaluated in their pure form (100% refrigerant conditions) and with their appropriate lubricants used or proposed to be used with the candidate refrigerant chemistries. Lubricants evaluated included mineral oil, as well as unadditized and additized polyalkylene glycol (PAG), unadditized polyol ester (POE), and unadditized and additized polyvinyl ether (PVE). Typically, POEs are used without any additives to improve the stability of the lubricant with a refrigerant, while both PAGs and PVEs use additives to stabilize the lubricant with the refrigerant. R-1233zd(E) and R-1224yd(Z) are commercialized products using mineral oil based lubricants and do not use synthetic lubricants like PAGs, POEs and PVEs. As a result, R-1233zd(E) and R-1224yd(Z) were only tested with a mineral oil based lubricant. Likewise, R-1336mzz(Z), R-1336mzz(E) and R-514A were evaluated only with PAG, POE and PVE since they do not have suitable properties with mineral oil. Additionally, the temperature of the exposures were selected based on the intended application of the refrigerant and historical literature evaluation conditions. For refrigerants only applied in water cooled applications an exposure temperature of 127°C (261°F) was determined to be most appropriate while for refrigerants applied in air cooled or heat pump applications, an exposure temperature of 175°C (347°F) was determined to be most appropriate. Table 3 and Table 4 summarize the test conditions used in this study.

Table 3. Summary of Refrigerant and Lubricant Test Conditions with Metal Coupons.

	No Lubricant	Mineral Oil	Unadditized PAG	Additized PAG	POE	Unadditized PVE	Additized PVE	Time (days)	Temp
R-123	X	X						14	127°C
R-514A	X		X		X	X		14	127°C
R-1233zd(E)	X	X						14	127°C
R-1224yd(Z)	X	X						14	175°C
R-1336mzz(Z)	X		X	X	X	X	X	14	175°C
R-1336mzz(E)	X		X	X	X	X	X	14	175°C

Table 4. Summary of Refrigerant and Lubricant Test Conditions with Desiccant Materials.

	No Lubricant	Mineral Oil	Additized PAG	POE	Additized PVE	Time (days)	Temp
R-514A	X		X	X	X	28	100°C
R-1233zd(E)	X	X				28	100°C
R-1224yd(Z)	X	X				28	100°C
R-1336mzz(Z)	X		X	X	X	28	100°C
R-1336mzz(E)	X		X	X	X	28	100°C

R-123 Summary - 14 days at 127°C (261°F)

In general, the R-123 results were as expected with greater instability observed in the conditions containing lubricant. In the lubricant containing conditions, refrigerant decomposition was observed by the elevated concentrations (>100 ppm) of chloride (Figure 2). Visual analysis also indicated potential interactions with iron, copper, and brass; however, no dissolved elements were reported in the lubricant. Additionally, no lubricant decomposition was observed via TAN analysis. The GC/MS analysis of the headspace revealed the presence of R-133a (due to the de-chlorination of R-123) in lubricated conditions. The pure refrigerant analysis revealed unique decomposition products with the elimination of hydrogen and chloride as (HCl) from the R-123 and the formation of R-1112 isomers. Overall, the 100% refrigerant conditions showed to be 5 to 10 times more stable compared to 20%/80% refrigerant/lubricant conditions based on observed decomposition products via GC/MS. Despite observations of refrigerant breakdown, R-123 has been successfully used for 30 years in water cooled centrifugal chillers.

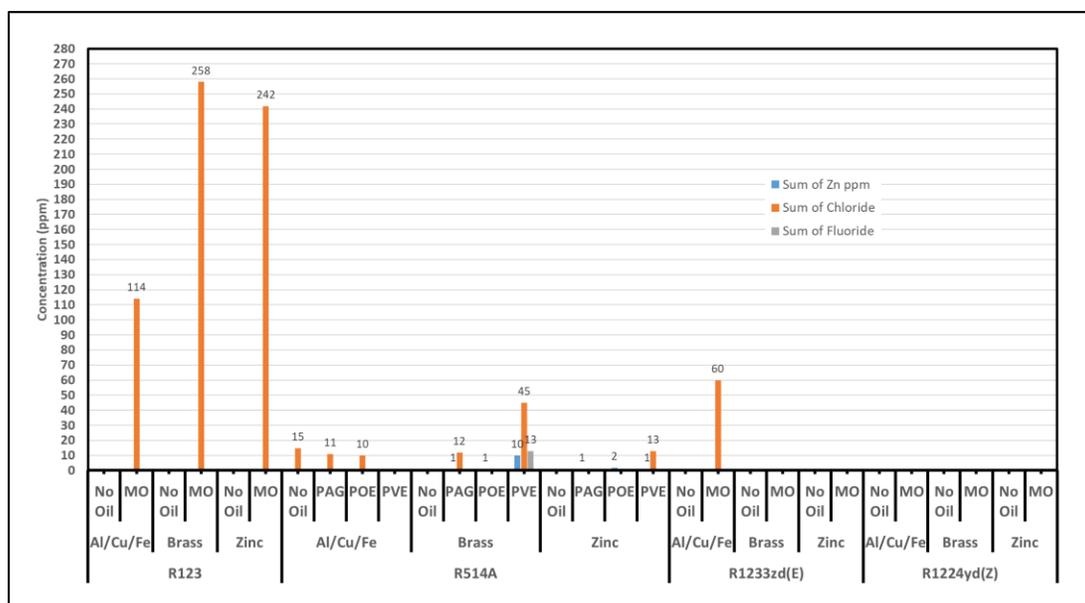


Figure 2: Summary of Dissolved Species of Interest for R123, R514A, R1233zd(E) and R1224yd(Z)

R-514A Summary - 14 days at 127°C (261°F)

Visual analysis of the metal coupons revealed subtle interactions with copper, iron, and brass in select conditions. No lubricant decomposition was measured via TAN and only small amounts of dissolved zinc (≤ 10 ppm) were measured in PAG, POE, and PVE conditions containing brass and the zinc alloy. Refrigerant decomposition was detected in several conditions by anion analysis with the detection of chloride, primarily in conditions containing PAG and PVE lubricants. GC/MS did not reveal significant insights to refrigerant decomposition or stereoisomerization; however, a late eluting compound noted as R-1336mzz-PVE unknown was measured in all conditions containing PVE. Additional impurities in the chromatograms were identified as PAG breakdown in all PAG exposures.

Based on the data generated in this study, it was concluded that R-514A is comparatively about 5 to 10 times more stable than R-123 if using the formation of chloride after aging (10-45 ppm for R-514A vs 100-250 ppm for R-123) as the primary criteria (Figure 2). In addition, the appearance of the metal coupons was not as affected in the evaluation with R-514A compared to R-123, as no copper plating of the iron was observed in the three metal condition with refrigerant/lubricant. No refrigerant decomposition was observed by anion analysis and limited insights to the degree of stereoisomerization were provided via GC/MS analysis. Some degree of lubricant decomposition was observed in all POE conditions due to an elevation in TAN, most significantly in the POE/activated alumina condition where an elevated TAN (0.20 mg KOH/g oil) was accompanied by the presence of organic acids. No dissolved elements were detected in the lubricant, except slightly elevated levels of silicon, mainly in the additized PVE conditions. Dissolved silicon is an indication of possible refrigerant reactions with the sealed glass tube.

R-1233zd(E) Summary- 14 days at 127°C (261°F)

Visual analysis indicated potential interactions of copper in the 100% refrigerant condition and of iron and brass in conditions containing lubricant. Limited refrigerant decomposition was observed by anion and GC/MS analysis with the exception of the mixed metal (Cu, Fe, Al) with lubricant, where 60 ppm of chloride was present. No lubricant decomposition via TAN analysis or dissolved metals was observed in the lubricant analysis. Limited refrigerant decomposition was observed by anion and GC/MS analysis with the exception of low levels of 3,3,3-trifluoro-1-propyne detected by GC/MS in all pure refrigerant conditions (potential H-Cl elimination reaction). Slight elevation in TAN (< 0.15 mg KOH/g oil) was observed in all conditions containing lubricant.

R-1224yd(Z) Summary - 14 days at 127°C (261°F)

Visual analysis indicated potential interactions of copper in the 100% refrigerant condition and with brass in both 100% refrigerant and refrigerant/lubricant exposures with some discoloration of the catalyst surfaces, however no corresponding dissolved elements were observed in the lubricant. Limited refrigerant decomposition was observed by anion and GC/MS analysis with the exception of low levels of 3,3,3-trifluoro-1-propyne detected in multiple

conditions and low levels of 3,3,3-trifluoropropene (R-1243zf) in conditions containing zinc. Conclusions were not able to be drawn regarding the changes in the stereoisomer (R-1224yd(E)) concentrations due to its elevated concentration of the R-1224yd(E) in the baseline refrigerant, variability with the analytical method utilized, and the peak area response. No lubricant decomposition was noted in the TAN analysis.

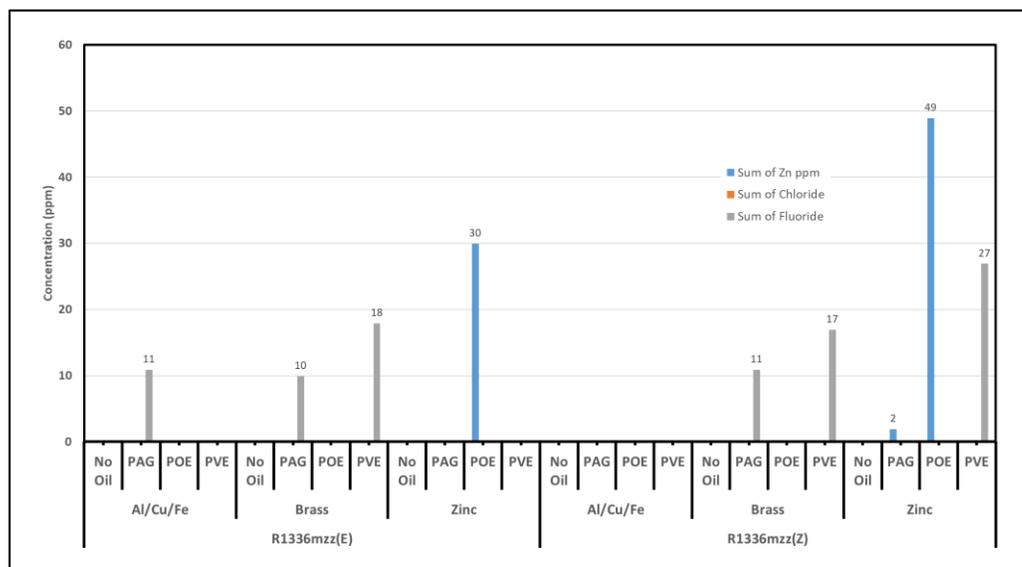


Figure 3: Summary of Dissolved Species of Interest for R-1336mzz(E) and R-1336mzz(Z)

R-1336mzz(Z) Summary - 14 days 175°C (347°F)

Visual analysis indicated potential interactions with the metal coupons in many exposure conditions. The use of additized PAG and PVE lubricants lessened the extent of visual changes in the three metal and zinc conditions, however interactions with brass were still observed with and without the additives. Refrigerant decomposition was observed through elevated levels of fluoride in conditions containing both unadditized and additized PAG and PVE lubricants (Figure 3). The additive package in the PAG lubricant showed comparable or increased levels of fluoride, while the additive package in the PVE lubricant appeared to reduce the amount of measured fluoride. Minimal to no increase in TAN or dissolved metals was observed in all lubricant conditions except in the POE/zinc condition where slight elevation in TAN (0.20 mg KOH/g oil) and low levels of organic acids were observed, indicating lubricant decomposition. Additionally, dissolved zinc (49 ppm) was measured in the POE lubricant indicating interactions with the zinc coupon. POE reactivity with zinc is well known and has been seen with other refrigerants in the AHRTI 9016 study.

GC/MS analysis indicated potential stereoisomerization of the R-1336mzz(Z) to the R-1336mzz(E). Increase levels of R-1336mzz(E) were observed in tests with POE lubricants and when no oil was present. This was an expected result since E-isomers are typically more chemically stable than Z-isomers. However, due to the difference in boiling points between the two stereoisomers, this test method used limited the ability to appropriately quantify the extent of reactivity without the use of response calibration. It would be recommended to further study the stereoisomerization rate of R-1336mzz(Z) in a separate time temperature study. Two additional potential breakdown products of interest were detected in various concentrations in the unadditized and additized PVE conditions, 1,1,1,4,4,4-hexafluorobutane (possible hydrogenation product of R-1336mzz) and a late eluting compound noted as a R-1336mzz-PVE unknown. Finally, additional impurities in the chromatograms were identified as PAG and PVE breakdown and the presence of additives had limited effect on the reduction of these components.

R-1336mzz(E) Summary - 14 days at 175°C (347°F)

Limited interactions with the metal coupons were observed, except for minor interactions with iron, brass, and zinc in select conditions. Refrigerant decomposition was observed through elevated levels of fluoride in conditions containing unadditized and additized PAG and unadditized PVE lubricants (Figure 3). The additive package in the PVE lubricant appeared to reduce the amount of measured fluoride, while the impact of the PAG additive package was difficult to quantify due to the low levels of fluoride measured. The only increase in TAN was observed in the conditions

containing POE with all metals indicating lubricant breakdown. A large increase in TAN (0.22 mg KOH/g oil), measurable organic acids, and dissolved zinc was observed in the POE/zinc condition. In all conditions except with POE and zinc, no dissolved elements were detected in the lubricant except for elevated levels of silicon, mainly in the additized PVE conditions.

GC/MS analysis detected the stereoisomer of R-1336mzz(E), R-1336mzz(Z), in all conditions as well as the baseline refrigerant, however no conclusions could be drawn regarding changes in concentration due to the differences in vapor pressure of the components. Given the observation of 1,1,1,4,4,4-hexafluorobutane (possible hydrogenation of R-1336mzz) and a late eluting compound, noted as a R-1336mzz-PVE unknown, present in the R-1336mzz(Z) exposures, special care was taken to observe increases in concentrations of these components. Consistent with the R-1336mzz(Z) findings, these components were observed in the greatest concentrations in the PVE test conditions with both additized and unadditized lubricants. Finally, additional impurities in the chromatograms were identified as PAG and PVE breakdown and the presence of additives had limited effect on the reduction of these components.

3. CONCLUSIONS

In general, better chemical stability was seen with all refrigerants and refrigerant/lubricant combinations as compared to R-123 based on the amount of fluoride, chloride, or formation of refrigerant decomposition products after exposures. Small potential interactions with iron, copper, brass and zinc samples in the 100% refrigerant and refrigerant/lubricant exposures were seen in some cases. Potential for reactivity was observed with R-1336mzz(Z) based on the observations of increased fluoride in multiple conditions relative to the same conditions tested with R-1336mzz(E). In addition to elevated fluoride, high concentrations of stereoisomerization up to 6% was observed in the 100% refrigerant conditions with R-1336mzz(Z).

This study revealed the role of lubricant chemistry on the breakdown of the various hydrofluoroolefin (HFO) refrigerants tested. A unique breakdown product was observed in all the HFO-PVE conditions and suspected to be an interaction of the refrigerant and lubricant. The addition of the additive package in the PVE had no impact on the presence of this unknown compound. An additional indicator of refrigerant decomposition is the generation of fluoride. Fluoride was consistently generated in conditions containing the unadditized PAG and unadditized PVE lubricants, however, the same trend was not observed in conditions containing POE. The addition of the PVE additive package resulted in a reduction in fluoride generation, but the same was not observed with the PAG additive package, which had varying impact on the fluoride generation.

The presence of zinc-containing materials produced increased refrigerant and lubricant decomposition in many conditions, suggesting the presence of zinc will accelerate reactivity. With select refrigerants, R-1233zd(E) and R-1224yd(Z), unique carbon-carbon triple bond (propyne) breakdown products were identified in the GC/MS analysis. Lubricant decomposition of the POE in the presence of zinc containing materials was also observed with elevated TANs and organic acids. Based on these results, caution should be taken when applying zinc containing materials in HVAC&R systems with HFO refrigerants.

Based on these findings, further work was recommended in the AHRTI Project 9016 final report. For the refrigerants presented in this paper, it was recommended to further explore the unique HFO-PVE interactions that were observed in the GC/MS analysis, the impact of lubricant additives and/or refrigerant stabilizers on stability, and additional materials of construction that may result in unique interactions given observations with brass and zinc. Additionally, there remains a gap in the assessment of semi or non-volatile breakdown products that remain in the lubricant phase and are not detected in the assessment of the headspace via GC/MS.

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NOMENCLATURE

ppm	=	Parts per million
TAN	=	Total Acid Number, mg KOH/gram oil

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