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(54) Titre : INHIBITION DE LA CORROSION EN PHASE VAPEUR
(54) Title: VAPOR PHASE CORROSION INHIBITION

(57) Abrégé/Abstract:
The disclosed technology relates to the inhibition of corrosion on electrically conductive componentry subjected to a lubricant composition, but that is not submersed in the lubricant composition, or in other words, vapor phase corrosion inhibition. The technology more particularly relates to the use of azole compounds capable of inhibiting corrosion of the electrically conductive componentry in the vapor space above a lubricant composition, and often in the liquid phase of the lubricant composition as well.

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TITLE

VAPOR PHASE CORROSION INHIBITION

BACKGROUND OF THE INVENTION

[0001] The disclosed technology relates to the inhibition of corrosion on electrically conductive componentry subjected to an automotive lubricant composition, but that is not submersed in the lubricant composition, or in other words, vapor phase corrosion inhibition. The technology more particularly relates to the use of azole compounds capable of inhibiting corrosion of the electrically conductive componentry in the vapor space above an automotive lubricant composition, and often in the liquid phase of the lubricant composition as well.

[0002] Corrosion is of increasing relevance in the automotive industry due to electrification of vehicle drivelines, whether in full electric vehicles, hybrid vehicles or even internal combustion vehicles. Although transmission lubricating oils are designed to protect metal (most often copper or iron) surfaces submerged in the oil from corrosion, some lubricants can be still be corrosive. Further, issues are arising due to corrosion of parts not submerged in the oil. For example, the evolution of transmissions is such that there are more sensors being used that are not immersed in the lubricant but are exposed in the vapor space to corrosive species. Since such electronics are typically not submerged in the lubricant, these electronics are not protected. Corrosion inhibitory performance for non-submerged electronics is not currently encompassed in vehicle lubricant specifications, but it is anticipated that vapor phase corrosion performance will become increasingly important, particularly with respect to sensitive electronics where even slight corrosion can interrupt the function of the electronics. Corrosion has been studied in the vapor phase, however the corrosion phenomena that have thus far been described are primarily due to atmospheric corrosion (e.g. based on humidity, oxidation and salts), while the corrosion with respect to electronics in the headspace above an automotive lubricant will have significantly different set of environmental contributors (e.g., low humidity, low oxygen, volatile lubricant and lubricant degradation products).

[0003] A need exists to provide corrosion protection to electrically conductive componentry in automotive vehicles that are not submerged in a protective lubricant composition. That is, a need exists for vapor phase corrosion protection to electrically conductive componentry in vehicles.

SUMMARY OF THE INVENTION

[0004] It has been found that certain azole compounds having sufficiently high vapor pressure to escape the liquid phase of a lubricant composition can provide vapor phase corrosion protection above the lubricant composition.

[0005] The disclosed technology, therefore, solves the problem of vapor phase corrosion of electrically conductive componentry in vehicles by providing a lubricant composition containing an azole compound capable of inhibiting corrosion of the electrically conductive componentry in the vapor space above the lubricant composition and a method therewith.

[0006] The lubricant composition can include an oil of lubricating viscosity and an azole compound capable of escaping the lubricant composition and inhibiting corrosion in a vapor space above the lubricant composition.

[0007] In an embodiment, the azole compound can be a low molecular weight triazole or low molecular weight tetrazole compound. In some embodiments, the azole compound can be an N-substituted azole compound that will decompose to a low molecular weight triazole or low molecular weight tetrazole compound under the operating conditions of an automotive device. In further embodiments, the azole compound can be an N-substituted azole compound that will decompose in the presence of a compound that reacts with the N-substituted azole compound resulting in a low molecular weight triazole or low molecular weight tetrazole compound.

[0008] The lubricant composition can further include a compound that reacts with the N-substituted azole compound resulting in a low molecular weight triazole or low molecular weight tetrazole compound.

[0009] In embodiments, the composition can further contain a volatile compound corrosive to electrically conducting componentry.

[0010] There is also provided a method of lubricating an automotive device having electrically conducting componentry. The method includes providing an automotive device having electrically conducting componentry, some portion of said componentry being dry, then delivering to the automotive device a lubricant composition as set forth above, and operating the automotive device.

[0011] The electrically conducting componentry can include, for example, electrical wires, electrical sensors, printed circuit boards, or an electric motor.

[0012] The electrically conducting componentry can, for example, contain copper or a copper alloy.

[0013] In embodiments, the method can be applied where the automotive device contains a transmission, such as, for example, a dual clutch transmission, or a transmission that is driven by an electric motor.

[0014] In further embodiments, the method can be applied where the automotive device contains an axle. In some embodiments, the axle can be driven by an electric motor.

DETAILED DESCRIPTION OF THE INVENTION

[0015] Various preferred features and embodiments will be described below by way of non-limiting illustration.

[0016] One aspect of the technology encompasses a lubricant composition of 1) an oil of lubricating viscosity, 2) an azole compound capable of inhibiting corrosion of electrically conductive componentry, both in the liquid phase of the lubricant composition and in the vapor space above the lubricant composition, and 3) a volatile compound corrosive to the electrically conductive componentry.

Oil of Lubricating Viscosity

[0017] One component of the disclosed technology is an oil of lubricating viscosity, also referred to as a base oil. The base oil may be selected from any of the base oils in Groups I-V of the American Petroleum Institute (API) Base Oil Interchangeability Guidelines (2011), namely

Base Oil Category	Sulfur (%)		Saturates (%)	Viscosity Index
Group I	>0.03	and/or	<90	80 to less than 120
Group II	≤0.03	and	≥90	80 to less than 120
Group III	≤0.03	and	≥90	≥120
Group IV	All polyalphaolefins (PAOs)			
Group V	All others not included in Groups I, II, III or IV			

[0018] Groups I, II and III are mineral oil base stocks. Other generally recognized categories of base oils may be used, even if not officially identified by the API: Group II+, referring to materials of Group II having a viscosity index of 110-119 and lower volatility than other Group II oils; and Group III+, referring to materials of Group III having a viscosity index greater than or equal to 130. The oil of lubricating viscosity

can include natural or synthetic oils and mixtures thereof. Mixtures of mineral oil and synthetic oils, e.g., polyalphaolefin oils and/or polyester oils, may be used.

[0019] In one embodiment the oil of lubricating viscosity has a kinematic viscosity at 100 °C by ASTM D445 of 2 to 7.5 or 10, or 3 to 6, or 3.25 to 6, or 3.5 to 5 mm^2/s, or from 2 to 7 or 3 to 6 or 3 to 5. In one embodiment the oil of lubricating viscosity comprises a poly alpha olefin having a kinematic viscosity at 100 °C by ASTM D445 of 2 to 7.5 or any of the other aforementioned ranges.

Azole Compound

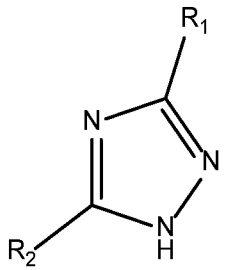
[0020] The lubricant composition also contains an azole compound capable of inhibiting corrosion of electrically conductive componentry in the space above the lubricant composition.

[0021] The phrase "electrically conductive componentry" is used to refer to components in an automobile engine or driveline that conduct electricity, such as, for example, electrical wires, electrical sensors, printed circuit boards, electric motors, etc. Such components are generally kept "dry," meaning the components are not submerged in a lubricant composition, but they will in many cases be in close proximity and exposed to a lubricant composition. Such electrically conducting componentry can be prepared from copper or other electrically conductive material, such as, for example, copper alloys (brass, bronze), silver, aluminum, gold, platinum, tin, and alloys of any of the foregoing, or other like electrically conductive materials.

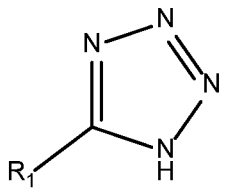
[0022] Many azole compounds will exhibit corrosion inhibition in the liquid phase of a lubricant composition. However, not all azole compounds will exhibit corrosion inhibition in the vapor space above the lubricant composition. To exhibit such vapor phase corrosion inhibition, the azole compound first must have sufficiently high vapor pressure to vaporize, i.e., escape the liquid phase of the lubricant composition and enter the vapor phase. More than just escaping the liquid phase, the azole compound must also be capable of coating the electrically conductive componentry to protect the componentry from other volatile compounds present in the vapor phase that would otherwise be corrosive to the electrically conductive componentry.

[0023] While not wishing to be bound by theory, the coating of the electrically conductive componentry may arise when the azole compound includes more than 2 ring nitrogens, and has a proton available on the azole ring to interact with the metal of the electrically conductive componentry. Azole compounds capable of inhibiting

corrosion of the electrically conductive componentry in the vapor space above a lubricant composition thus can include low molecular weight triazoles and low molecular weight tetrazoles. By low molecular weight, it is meant a compound having a molecular weight between about 50 and 350 daltons, or between about 55 and 250 daltons, or between about 60 and 150 or 200 daltons. Such compounds include, for example, those of formulas I or II:



I



II

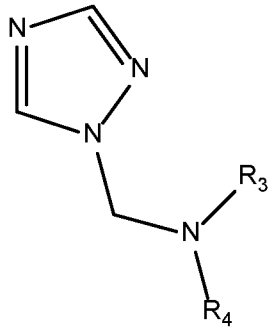
where R_1 and R_2 can be, individually, H or a C_1 to C_9 alkyl group

[0024] Examples of azole compounds of formula I can include, for example, 1,2,4-triazole, 3-methyl-1,2,4-triazole and the like. Examples of formula II can include, for example, 1H-tetrazole, 5-methyltetrazole, and the like.

[0025] The azole compound capable of inhibiting corrosion of the electrically conductive componentry can also include N-substituted azole compounds. N-substituted azole compounds may provide vapor phase corrosion protection on their own, or decompose to a low molecular weight triazole or low molecular weight tetrazole compound under an operating condition of the automotive device; or decompose to a low molecular weight triazole or low molecular weight tetrazole in the presence of a compound that reacts ("reactive compound") with the N-substituted azole compound resulting in the release or formation of a low molecular weight triazole or low molecular weight tetrazole compound.

[0026] Formulas I and II may be reacted with an alkyl (meth)acrylate to obtain a compound having an alkyl (meth)acrylate substituent on a ring nitrogen. The formulas may also be reacted to obtain a formula with an amine substituent on a ring nitrogen, for example, by reacting with formaldehyde and the desired amine.

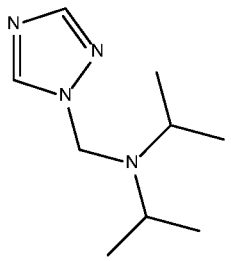
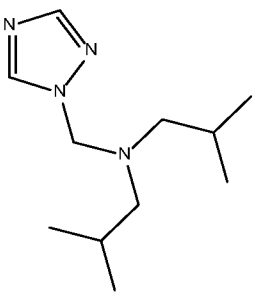
[0027] An example N-substituted azole compound with an amine substituent can include 1,2,4 triazoles of formula III:

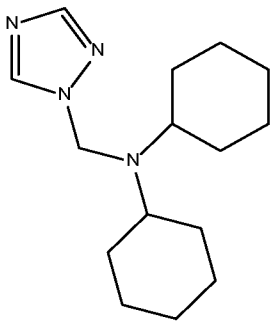
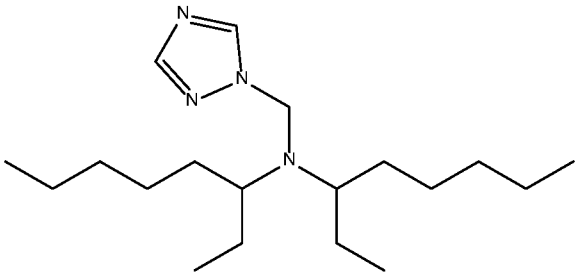
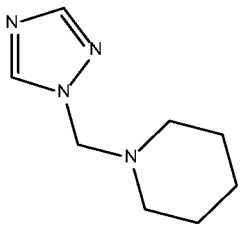
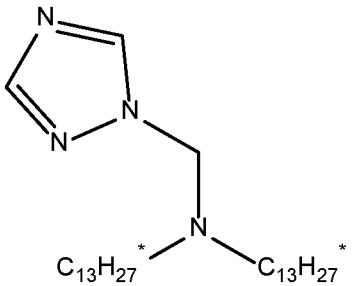


III

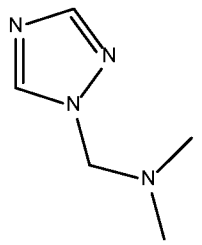
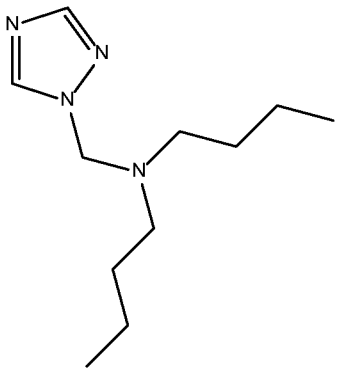
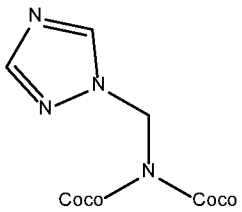
where R_3 and R_4 can be, independently C_1-C_{22}, or C_2-C_{20}, or C_3-C_{18}, or C_3-C_{16} or C_{12}, either linear or branched hydrocarbon groups, phenyl group, or two ends of a hydrocarbon chain forming a cyclic structure, or where at least one of R_3 and R_4 can be H.

[0028] In some embodiments, the N-substituted azole compound can be an N-branched substituted 1,2,4 triazole, such as those of formula III where R_3 and R_4 can be, independently C_1-C_{22}, or C_2-C_{20}, or C_3-C_{18}, or C_3-C_{16} or C_{12} branched hydrocarbon groups, or two ends of a hydrocarbon chain forming a cyclic structure. Example structures of formula III can include:

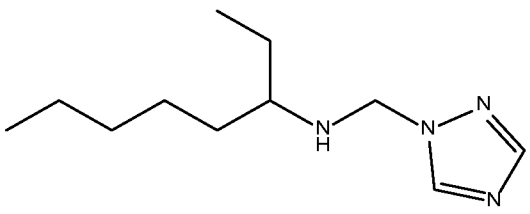
N,*N*-Bis(1-methylethyl)-1*H*-1,2,4-triazole-1-methanamine	
N,*N*-diisobutyl-1*H*-1,2,4-triazole-1-methanamine	

N,N-dicyclohexyl-1H-1,2,4-triazole-1-methanamine	
N,N-bis(2-ethylhexyl)-1*H*-1,2,4-Triazole-1-methanamine	
1-((1H-1,2,4-triazol-1-yl)methyl)piperidine	
N,N-bis(tridecyl)-1*H*-1,2,4-Triazole-1-methanamine *mixed branched isomers	

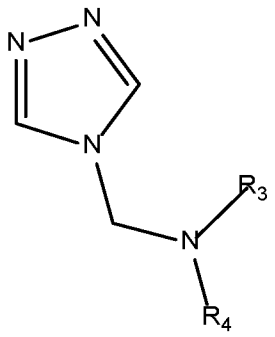
[0029] In some embodiments, the N-substituted azole compound can be an N-linear substituted 1,2,4 triazole, such as those of formula III where R_3 and R_4 can be, independently linear C_1-C_{22}, or C_2-C_{20}, or C_3-C_{18}, or C_3-C_{16} or C_{12} hydrocarbon groups (including carbonyl groups or acrylamide groups). Example structures of formula III can include:

N,N-dimethyl-1-(1H-1,2,4-triazol-1-yl)methanamine	
N,N-dibutyl-1*H*-1,2,4-triazole-1-methanamine	
N,N-dicoco-1-(1H-1,2,4-triazol-1-yl)methanamine	

[0030] In other embodiments, the N-substituted azole compounds can include, for example, N-single substituted 1,2,4 triazoles, such as those of formula III where R_3 and R_4 can be, independently C_1-C_{22}, or C_2-C_{20}, or C_3-C_{18}, or C_3-C_{16} or C_{12}, either linear or branched hydrocarbon groups, or two ends of a hydrocarbon chain forming a cyclic structure, and where at least one of R_3 and R_4 is H. Example structures of formula III can include:

N-((1H-1,2,4-triazol-1-yl)methyl)octan-3-amine	

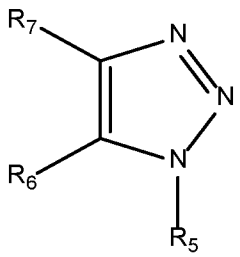
[0031] The azole compound can also include N-substituted 1,2,4 triazoles, where the N-substitutent is at the 4 position, as in formula IV:



IV

where R_3 and R_4 are as defined above. Compounds of formula IV may, in some embodiments, be naturally occurring impurities or minor isomers formed during the manufacture of compounds of formula III.

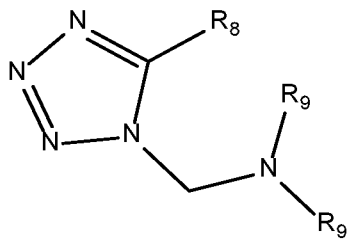
[0032] Other N-substituted azole compounds can include 1,2,3 triazoles of formula V:



V

where R_5, can be, independently C_1-C_{22}, or C_2-C_{20}, or C_3-C_{18}, or C_3-C_{16} or C_{12}, either linear or branched hydrocarbon groups, phenyl group, or two ends of a hydrocarbon chain forming a cyclic structure, R_6 and R_7 can be C_1-C_4, or C_1-C_3, or C_1-C_2, or where at least one of R_5, R_6 and R_7 can be H, or where both R_6 and R_7 are H, or at least one of R_5, R_6 and R_7 can include carbonyl or acrylamide groups, such as in methyl propionate or ethylhexyl propionate and the like, which may be formed by contacting the azole compound with an acrylate, acrylic acid, acrylamide or combination thereof.

[0033] Further N-substituted azole compounds include tetrazoles of formula VI:



VI

where R_8 and R_9 can be, independently C_1-C_{22}, or C_2-C_{20}, or C_3-C_{18}, or C_3-C_{16} or C_{12}, either linear or branched hydrocarbon groups, a phenyl group, or two ends of a hydrocarbon chain forming a cyclic structure, or where at least one of R_8 and R_9 can be H.

[0034] The azole compounds are formulated into a lubricant composition at a level sufficient to provide suitable corrosion protection in the vapor phase when in use. In general, levels of about 30 ppm to 5 wt% of the azole compound are suitable in most applications. In some embodiments, the azole compound can be incorporated at a level of about 50 ppm to 4wt%, or about 250ppm to 3wt%, based on the total weight of the lubricant composition, or even from 500ppm to 2wt% or 1000 ppm to 1 wt%. In some embodiments, the azole compound can be incorporated at a level of from about 100 ppm to 5000 ppm, or 250 ppm to 2500 ppm, or 500 to 2000 ppm.

Azole Decomposition

[0035] The azole compounds above may decompose to provide a low molecular weight azole compound. Decomposition can occur, for example, due to temperatures encountered during operation of the automotive device.

[0036] Decomposition can also occur due to the presence of a compound ("reactive compound") that reacts with the N-substituted azole compound resulting in release or formation of a low molecular weight triazole or low molecular weight tetrazole compound. Thus, the lubricant composition can include a compound that reacts with the N-substituted azole compound resulting in decomposition to a low molecular weight triazole or low molecular weight tetrazole compound.

[0037] In some embodiments, the reactive compound may be an electrophile, a nucleophile, or a combination thereof. Thus, the lubricant composition can also include a compound that is electrophilic to the azole compound and/or a nucleophilic to substituents on the azole compound, or a combination thereof.

[0038] Electrophilic compounds may include, for example, Lewis acids and Bronsted acids. Examples of electrophilic compounds can include, for example, hydrogen (whether on its own or as an "onium" compound such as NH_4^+ or H_3O^+); metal cations such as Li+, Cu(I/II), Ti(IV), Fe(II/III), etc.; trigonal planar species such as BF_3 and the like; α,β-unsaturated carbonyls; polar molecules like carbon dioxide, etc. Such compounds can arise in the lubricant from other additives in the lubricant, such as,

for example from detergent substrates and antiwear additives and their decomposition products.

[0039] Nucleophilic compounds can include Lewis bases. Examples of nucleophiles can include iodine, alcohols, such as, for example, methanol, ethanol or higher alcohols, amines, such as ammonia, or an amine from the head group of a dispersant or surfactant. Here again, such compounds can arise in the lubricant from other additives in the lubricant, such as, for example from friction modifiers and their decomposition products.

Liquid Phase Corrosion Inhibitor

[0040] The lubricant composition can also include corrosion inhibitors that work in the liquid to prevent corrosion in the liquid phase. An example of such a liquid phase corrosion inhibitor can include, for example, a substituted thiadiazole, such as a dimercaptothiadiazole (DMTD) derivative. DMTD derivatives may be used to impede corrosion of copper. The dimercaptothiadiazole derivatives typically are soluble forms or derivatives of DMTD. Materials which can be starting materials for the preparation of oil-soluble derivatives containing the dimercaptothiadiazole nucleus can include 2,5-dimercapto-[1,3,4]-thiadiazole, 3,5-dimercapto-[1,2,4]-thiadiazole, 3,4-dimercapto-[1,2,5]-thiadiazole, and 4,-5-dimercapto-[1,2,3]-thiadiazole. Of these the most readily available is 2,5-dimercapto-[1,3,4]-thiadiazole. Various 2,5-bis-(hydrocarbon dithio)-1,3,4-thiadiazoles and 2-hydrocarbyldithio-5-mercapto-[1,3,4]-thiadiazoles may be used. The hydrocarbon group may be aliphatic or aromatic, including cyclic, alicyclic, aralkyl, aryl and alkaryl. Similarly, carboxylic esters of DMTD are known and may be used, as can condensation products of alpha-halogenated aliphatic monocarboxylic acids with DMTD or products obtained by reacting DMTD with an aldehyde and a diaryl amine in molar proportions of from about 1:1:1 to about 1:4:4. The DMTD materials may also be present as salts such as amine salts. In other embodiments, the DMTD compound may be the reaction product of an alkyl phenol with an aldehyde such as formaldehyde and a dimercaptothiadiazole. Another useful DMTD derivative is obtained by reacting DMTD with an oil-soluble dispersant, such as a succinimide dispersant or a succinic ester dispersant.

[0041] The amount of the DMTD compound, if present, may be 0.01 to 5 percent by weight of the composition, depending in part on the identity of the particular compound, e.g., 0.01 to 1 percent, or 0.02 to 0.4 or 0.03 to 0.1 percent by weight. Alternatively, if the DMTD is reacted with a nitrogen-containing dispersant, the total weight of the combined product may be significantly higher in order to impart the

same active DMTD chemistry; for instance, 0.1 to 5 percent, or 0.2 to 2 or 0.3 to 1 or 0.4 to 0.6 percent by weight.

[0042] In some embodiments, the substituted thiadiazole can be present in a formulation that is substantially free or free of reactants that could react with the substituted thiadiazole to form a volatile corrosive thiol, such as, for example, a hydrogen phosphite.

Volatile Corrosive Compound

[0043] The lubricant composition will, by virtue of the problem statement, also include a volatile compound that is corrosive to the electrically conducting componentry, or "volatile corrosive compound" for short. Volatile, in reference to the volatile corrosive compound, has the same meaning as with the azole compound, that is, the volatile corrosive compound must have sufficiently high vapor pressure to vaporize, i.e., escape the liquid phase of the lubricant composition under operating conditions in an automobile and enter the vapor phase.

[0044] The volatile corrosive compound can be a compound added to the lubricant composition and intended for other bulk fluid purposes, such as, for example, bulk phase rust inhibition, friction modification, or wear and oxidation prevention. The volatile corrosive compound can also be generated *in situ*, for example, as a natural degradation product of the components of the lubricant composition, or from the reaction of two or more components in the lubricant composition.

[0045] In an embodiment, the volatile corrosive compound can be a volatile sulfur-containing compound, such as a thiol. Other sulfur-containing compounds that can cause corrosion include, for example, sulfurized olefins, disulfides, sulfurized esters, mercaptans, thioethers, dialkyldithiophosphoric acids and their salts, and dithiocarbamates.

[0046] The volatile corrosive compound can also include hydrogen sulfide arising from the degradation or hydrolysis of any of the sulfur-containing compounds. Likewise, the volatile corrosive compound can be a low molecular weight mercaptan arising from the degradation of sulfurized olefins, or a sulfur dioxide compound from thermal degradation of a sulfonate or sulfone. In an embodiment, the volatile corrosive compound can be the reaction product of a substituted thiadiazole and a hydrogen phosphite.

Methods of Vapor Phase Corrosion Inhibition

[0047] A further aspect of the present technology encompasses a method of lubricating an automotive device having electrically conducting componentry. The method includes providing an automotive device comprising electrically conducting componentry with some portion of the electrically conducting componentry being dry (i.e., not submerged in a lubricant composition). A lubricant composition, as disclosed above, can be delivered to the automotive device, and the automotive device is operated.

[0048] The automotive device is, in one embodiment, a driveline device. The driveline device can be, for example, a gear, an axle, a drive shaft, an automatic or manual transmission, or a driveline of an off-highway vehicle (such as a farm tractor). Such driveline devices are lubricated by gear oils, axle oils, drive shaft oils, traction oils, manual transmission oils, automatic transmission oils, or off highway oils (such as a farm tractor oil).

[0049] In one embodiment a method of lubricating a manual transmission that may or may not contain a synchronizer system is provided. In one embodiment there is provided a method of lubricating an automatic transmission. In one embodiment the invention provides a method of lubricating an axle.

[0050] Automatic transmissions that may be encompassed by the disclosed method include, for example, continuously variable transmissions (CVT), infinitely variable transmissions (IVT), toroidal transmissions, continuously slipping torque converter clutches (CSTCC), stepped automatic transmissions or dual clutch transmissions (DCT).

[0051] The automatic transmissions can contain continuously slipping torque converter clutches (CSTCC), wet start and shifting clutches and in some cases may also include metal or composite synchronizers. Dual clutch transmissions or automatic transmissions may also incorporate electric motor units.

[0052] With respect to axles and gears, the method can include employing a gear oil or axle oil in a planetary hub reduction axle, a mechanical steering and transfer gear box in utility vehicles, a synchromesh gear box, a power take-off gear, a limited slip axle, and a planetary hub reduction gear box. Axles may also incorporate electric motors units. Motors may be placed, for example, “in-wheel” or on the front or rear axle. The electric motor may also be incorporated into the driveshaft.

[0053] The amount of each chemical component described is presented exclusive of any solvent or diluent oil, which may be customarily present in the commercial material, that is, on an active chemical basis, unless otherwise indicated. However, unless otherwise indicated, each chemical or composition referred to herein should be interpreted as being a commercial grade material which may contain the isomers, by-products, derivatives, and other such materials which are normally understood to be present in the commercial grade.

[0054] It is known that some of the materials described above may interact in the final formulation, so that the components of the final formulation may be different from those that are initially added. For instance, metal ions (of, e.g., a detergent) can migrate to other acidic or anionic sites of other molecules. The products formed thereby, including the products formed upon employing the composition of the present invention in its intended use, may not be predisposed to easy description. Nevertheless, all such modifications and reaction products are included within the scope of the present invention; the present invention encompasses the composition prepared by admixing the components described above.

[0055] As used herein, the term "about" means that a value of a given quantity is within ±20% of the stated value. In other embodiments, the value is within ±15% of the stated value. In other embodiments, the value is within ±10% of the stated value. In other embodiments, the value is within ±5% of the stated value. In other embodiments, the value is within ±2.5% of the stated value. In other embodiments, the value is within ±1% of the stated value.

[0056] The invention herein is useful for inhibiting corrosion of non-submerged electrically conductive componentry in lubricated driveline devices, which may be better understood with reference to the following examples.

EXAMPLES

[0057] **Sample Preparation -** General Procedure for coupling azoles to alkylamines with paraformaldehyde: 1.0 mole of azole is combined with 1.0 mole of alkyl- or dialkylamine and the mixture is heated to 60-80°C with agitation. Paraformaldehyde, from 0.94 to 1.0 equivalent, is then added. For low mw amines, the paraformaldehyde charge is divided into two equal portions, and the second portion is not charged until the reaction exotherm from the 1st charge has subsided. The equivalent weight of the paraformaldehyde is calculated as follows:

$$\text{Eq wt} = 30.0264/(\text{wt\% } CH_2{=}O \text{ in paraformaldehyde})$$

Once the exotherm from the paraformaldehyde addition has subsided, heating is continued until all solids have dissolved. Vacuum is then applied to the reaction mixture to remove the by-product water generated by the reaction. No further purification of the product is necessary. Samples prepared by this method are provided below.

[0058] **Sample 1**: A commercial sample of 1,2,4-triazole was obtained from Tokyo Chemical Industry Company. Ltd.

[0059] **Sample 2**: 1,2,4-Triazole (0.81 mole) was reacted with piperidine (0.81 mole) and 91% paraformaldehyde (0.761 equivalent) per the general procedure to yield 131.66 g (98% yield) of clear, slightly yellow liquid that froze upon cooling. The product, 1-((1H-1,2,4-triazol-1-yl)methyl)piperidine, had a melting point of 61-63 °C. ^{1}H and ^{13}C NMR of the product showed that it was exceptionally pure.

[0060] **Sample 3**: 1,2,4-Triazole (0.746 mole) was reacted with diisopropylamine (0.738 mole) and 91% paraformaldehyde (0.701 equivalent) per the general procedure to yield 105.13 g (77% yield) of clear, faintly yellow liquid. ^{1}H and ^{13}C NMR of the product showed that it was impure. Primary impurities were unreacted 1,2,4-triazole and di(1H-1,2,4-triazol-1-yl)methane, the product of two moles of triazole coupling with formaldehyde.

[0061] **Sample 4**: 1,2,4-Triazole (0.653 mole) was reacted with dibutylamine (0.654 mole) and 91% paraformaldehyde (0.614 equivalent) per the general procedure to yield 131.64 g (96% yield) of clear, colorless liquid. The product, *N,N*-dibutyl-1*H*-1,2,4-triazole-1-methanamine, was substantially pure by ^{1}H and ^{13}C NMR.

[0062] **Sample 5**: 1,2,4-Triazole (0.654 mole) was reacted with diisobutylamine (0.654 mole) and 91% paraformaldehyde (0.615 equivalent) per the general procedure to yield 131.68 g (96% yield) of a colorless, low-melting, crystalline solid, *N,N*-diisobutyl-1*H*-1,2,4-triazole-1-methanamine, having a melting point of < 45 °C. The product was substantially pure by ^{1}H and ^{13}C NMR, the only impurity being a trace of the triazole-to-triazole coupled compound.

[0063] **Sample 6**: 1,2,4-Triazole (0.653 mole) was reacted with 2-ethylhexylamine (0.654 mole) and 91% paraformaldehyde (0.615 equivalent) per the general procedure to yield 131.48 g (96% yield) of clear, almost colorless liquid. The ^{1}H and ^{13}C NMR spectra showed, however, that the desired product, N-((1H-1,2,4-triazol-1-yl)methyl)-2-ethylhexan-1-amine was not obtained in high purity. The NMR spectra indicate that the product obtained contained a mixture of at least three compounds in addition to the desired compound.

[0064] **Sample 7**: 1,2,4-Triazole (0.534 mole) was reacted with dicyclohexylamine (0.533 mole) and 91% paraformaldehyde (0.501 equivalent) per the general procedure to yield 139.07 g (99.5% yield) of a nearly colorless, crystalline solid, N,N-dicyclohexyl-1H-1,2,4-triazole-1-methanamine, having a melting point of > 65 °C. The product showed good purity by ^{1}H and ^{13}C NMR, the only impurities being trace amounts of the triazole-to-triazole coupled compound and unreacted triazole.

[0065] **Sample 8a**: The commercial corrosion inhibitor Irgamet® 30 from BASF Corporation, CAS Number 91273-04-0, is the formaldehyde-coupled product of 1,2,4-triazole with bis(2-ethyhexyl)amine. ^{1}H and ^{13}C NMR spectra of this sample show that it is very pure.

[0066] **Sample 8b:** The general procedure was used to prepare a product analogous to Irgamet 30. 1,2,4-Triazole (0.439 mole) was reacted with bis(2-ethyhexyl)amine (0.440 mole) and 91% paraformaldehyde (0.411 equivalent) to give 142.71 g (100%) of clear, colorless liquid product. The 1H and 13C NMR spectra of this material showed that the purity was comparable to the commercial product of Sample 8a.

[0067] **Sample 9**: 1,2,4-Triazole (0.404 mole) was reacted with oleylamine (0.404 mole) and 91% paraformaldehyde (0.403 equivalent) per the general procedure to yield 141.79 g (99.8% yield) of waxy product. The ^{1}H and ^{13}C NMR spectra showed that the product was a mixture of several compounds.

[0068] **Sample 10**: 1,2,4-Triazole (0.313 mole) was reacted with ditridecylamine (0.312 mole) and 95% paraformaldehyde (0.313 equivalent) per the general procedure to yield 144.26 g (100% yield) of clear, nearly colorless liquid product. The ditridecylamine used in this Sample was obtained from BASF; it is a complex mixture of isomers. The ^{1}H and ^{13}C NMR spectra confirm that the product is a complex mixture.

[0069] **Sample 11**: 1,2,4-Triazole (0.315 mole) was reacted with dicocoamine (0.316 mole) and 95% paraformaldehyde (0.314 equivalent) per the general procedure to yield 142.15 g (98.9% yield) of clear, pale amber liquid product. The alkyl groups on the dicocoamine are primarily a mixture of saturated C_{12}-C_{14} linear hydrocarbon chains. The ^{1}H and ^{13}C NMR show that the product is very pure.

[0070] **Sample 12:** A commercial sample of 5-Methyltetrazole was obtained from Tokyo Chemical Indu*stry Co*mpany. Ltd.

[0071] **Sample 13:** Using the general procedure, 5-methyltetrazole (0.490 mole) and bis(2-ethylhexyl)amine (0.494 mole) were coupled with 91% paraformaldehyde (0.458 mole) to give 165.75 g (99.4%) of clear, almost colorless liquid product. The ^{1}H NMR spectrum shows that the product is very pure. Due to the lack of hydrogen atoms directly on the azole ring, however, it was not possible to say conclusively whether the formaldehyde-coupled substituent was attached to N1 or N2 of the ring. The NMR showed a single ring methyl group, though, indicating that only one of the two possible substitution isomers had formed.

[0072] **Sample 14:** 5-Phenyltetrazole (0.358 mole) and bis(2-ethylhexyl)amine (0.359 mole) were coupled with 91% paraformaldehyde (0.336 mole) using the general procedure to give 142.81 g (99.2%) of clear, almost colorless liquid product. The ^{1}H NMR spectrum shows that the product is very pure. The position of the formaldehyde-coupled substituent on the ring is not definitive, however a single positional isomer is observed in the spectrum.

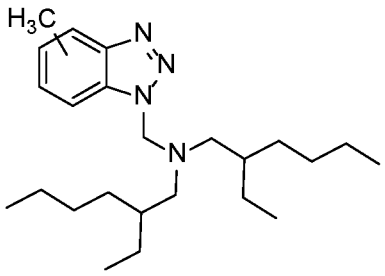
[0073] **Sample 15**: A commercial sample of 1,2-Dimethylimidazole was obtained from Alfa Aesar.

[0074] **Sample 16**: A commercial sample of 2,4-Dimethylimidazole was obtained from Alfa Aesar.

[0075] **Sample 17**: A commercial sample of 1-Butylimidazole was obtained from Alfa Aesar.

[0076] **Sample 18**: A commercial sample of 1-Methyl-1,2,4-triazole was obtained from Alfa Aesar.

[0077] **Sample 19**: A commercial oil-soluble corrosion inhibitor, Skosanor KSP-93, was obtained from Lubrizol Corporation. This product is the reaction product of tolyltriazole, bis(2-ethylhexyl)amine, and paraformaldehyde as shown below.



[0078] **Formulation A for testing vapor phase corrosion**: The formulation shown below, which is representative of a typical automotive transmission fluid, causes severe vapor-space corrosion of copper within a few days at 65°C, despite

having two known copper corrosion inhibitors (indicated by the asterisks "*"). In some tests described below, Formulation A without the tolyltriazole is used.

Formulation A	
Ingredient Generic Name	**wt%**
3 cSt Group III oil	60.61
4 cSt 100N Group III oil	21.90
Viscosity modifier	10.96
Dispersant	3.00
Friction modifier	1.40
Antioxidant	0.60
Substituted thiadiazole *	0.50
Seal swell agent	0.35
Antiwear agent (phosphite based)	0.22
Detergent	0.12
Pour Point depressant	0.09
Ethoxylated amine	0.10
Mineral acid	0.10
Tolyltriazole *	0.03
Foam inhibitor	0.02

[0079] **Semi-submerged Test**: Formulation A is top-treated with the Sample vapor phase corrosion inhibitors listed above at the reported treat rates. Freshly sanded copper strips are half immersed in the top-treated fluids in 4-oz jars which are capped and placed in a controlled temperature oven. Corrosion of both the liquid-immersed portion and vapor-space portion of the coupons is assessed per the ASTM D130 rating scale after a specified period. A blank (Formulation A with no top treat) is used as a standard in each test.

[0080] **Example 1**: Several Sample corrosion inhibitors were incorporated at a level of 500 ppm into Formulation A without tolyltriazole (listed as "Baseline" in Table 1). A semi-submerged test was conducted on each of these fluids at 65 °C for seven days. ASTM D130 ratings for both the liquid and vapor space portions of each coupon are shown in table 1 below.

Table 1

	Back		Front	
Sample	Vapor	Liquid	Vapor	Liquid
Baseline	4C	1A	4C	1A
1	1A	1A	1A	1A
2	1A	1A	1A	1A
4	1A	1A	1A	1A
5	1A	1A	1A	1A
7	1A	1A	1A	1A
10	1A	1A	1A	1A
11	1A	1A	1A	1A
13	2A	1A	2A	1A
14	4C	1A	4C	1A

[0081] **Example 2:** The performance of three sample corrosion inhibitors in a semi-submerged test as a function of treat level in Formulation A without tolyltriazole are shown in Table 2. The semi-submerged test was carried out at 80°C for seven days. ASTM D130 ratings for both the liquid and vapor space (front side only) are listed.

Table 2

	Sample 8		Sample 5		Sample 19	
Top Treat	Vapor	Liquid	Vapor	Liquid	Vapor	Liquid
0 ppm	4C	1A	4C	1A	4C	1A
500 ppm	1A	1A	1A	1A	4C	1A
250 ppm	1A	1A	1A	1A	4C	1A
125 ppm	1A	1A	1A	1A	4C	1A
62.5 ppm	1A	1A	1A	1A	4C	1A

[0082] **Example 3:** Performance of the sample corrosion inhibitors was demonstrated in both low and high viscosity fluids. All three formulations contained the same performance additives as Formulation A, with the exception of the base oil and viscosity modifier. The base oil in Example 3a was a 2cSt polyalpha olefin, the base oil in Example 3b was a low viscosity petroleum alkylate and the base oil in Example 3c was an 8cSt polyalpha olefin. None of these formulations contained a polymeric viscosity modifier.

Table 3

Example 3a: 2.1 cSt Fluid				
	Back		Front	
	Vapor	Liquid	Vapor	Liquid
none	4C	1B	4C	1B
500 ppm	1A	1A	1A	1A
100 ppm	1A	1B	1A	1B
50 ppm	1A	1B	1A	1B
Example 3b: 1.9 cSt Fluid				
none	4C	1B	4C	1B
500ppm	1A	1A	1A	1A
Example 3c: 9.1cSt Fluid				
	Back		Front	
none	4C	1B	4C	1B
500ppm	1A	1A	1A	1A

[0083] **Example 4:** Performance of the sample corrosion inhibitors was also demonstrated in an alternate fluid. Formulation B is a gear oil formulation that contains the additives listed below.

Formulation B	
Lubicant Oil	96.42
Extreme pressure agent	2.9
Pour point depressant	0.3
Rust inhibitor	0.2
Corrosion inhibitor	0.1
Friction modifier	0.05
Antifoam	0.03

[0084] Formulation B was top treated with 500ppm of Sample 8a in one instance and 500ppm of Sample 19 in another instance. These top treated gear oil formulations were subjected to the semi-submerged test at 80°C for seven days. Results are listed in Table 4.

Table 4

	Gear Oil Formulation B			
	Back		Front	
Sample	Vapor	Liquid	Vapor	Liquid
None (blank)	3B	3B	3B	3B
Sample 8	1A	1A	1A	1A
Sample 19	3A	3A	2E	2E

[0085] **Example 5**: This semi-submerged test is a short-duration comparison of several low molecular weight azoles in the Formulation A without tolyltriazole. The test was run at 80°C for 24 hours. Each Sample was added to Formulation A as a top treat at 1000ppm.

Table 5

Sample	Vapor	Liquid
1	1A	1A
12	1A	1A
15	4A	1B
16	4A	4A
17*	4A	1A
18	4A	1B

* Used Formulation A with tolyltriazole

[0086] **Example 6**: A 4-oz uncapped jar of the Formulation A was placed inside a ½-gallon wide-mouth jar. A freshly polished copper coupon was laid across the top of the small jar such that it was not in contact with the liquid. The outer large jar was capped and the entire assembly placed in an 80 °C oven for two days. The coupon turned black (4C rating). This test proves that the corrosive species from the Formulation A is acting through volatilization of the corrosive species into the vapor space rather than through a mechanism whereby the corrosive species climbs up the surface of the coupon from the liquid phase.

[0087] **Example 7**: Two 4-oz uncapped jars of Formulation A, one top-treated with 1000 ppm of Sample 8 were placed inside of a ½-gallon wide-mouth jar. A freshly polished copper coupon was laid across the top of the small jars such that it was not in contact with the liquids. The outer large jar was capped and the entire assembly placed in an 80 °C oven for two days. The coupon remained in pristine condition (1A rating). This test, in conjunction with Example 6 proves that the vapor-space inhibiting species from the top-treated Formulation A is acting through volatilization into the vapor space rather than through a mechanism whereby the inhibiting species climbs up the surface of the coupon from the liquid phase. This test also rules out a mechanism whereby the corrosion inhibiting species reacts with or neutralizes the corrosive species in the liquid phase before it can volatilize.

[0088] **Example 8**: One 4-oz uncapped jar of Formulation A, and a second 4-oz uncapped jar of 4 cSt Group III oil top-treated with 1000 ppm of Sample 8 were placed inside of a ½-gallon wide-mouth jar. Anhydrous calcium sulfate pellets were also scattered on the bottom of the ½-gallon jar to maintain an anhydrous environment inside the jar. A freshly polished copper coupon was laid across the top of the

small jars such that it was not in contact with the liquids. The outer large jar was purged with nitrogen, then capped and the entire assembly placed in an 80 °C oven for 6 days. The coupon slowly turned black (4 rating). This test, in conjunction with Examples 6 and 7 proves that Sample 8 is not inherently capable of providing vapor-space corrosion inhibition.

[0089] Example 9**:** Performance of the sample corrosion inhibitors was also demonstrated in an alternate fluid. Formulation C represents a baseline manual transmission fluid.

Formulation C	
Ingredient generic name	**Wt %**
Base oil	Balance to 100
Viscosity modifier (PMA Type)	7.58
Antioxidant (aminic)	0.3
Detergent (580 TBN calcium sulfonate)	0.58
Dispersant (PIB succinimide type)	1.7
Extreme pressure agent (sulfurized olefin)	0.3
Antiwear agent (phos containing, non-phosphite type)	0.91
Foam inhibitor	300ppm

[0090] Fluids E to K were generated by adding a thiadiazole corrosion inhibitor and/or inventive Sample 8b to Formulation C. Each fluid was tested & rated in accordance with ASTM D130 at 121 & 150°C for 3 hours as well as being subjected to the semi-submerged test at 80°C for 168 hours. The results are given below:

Fluid/Test	Baseline	E	F	G	H	I	J	K
Thiadiazole corrosion inhibitor (wt%)	0	0.3	0	0	0	0.3	0.3	0.3
Inventive inhibitor 8b (ppm)	0	0	250	500	1000	250	500	1000
ASTM D130 at 121C	3B	1A	2D	2D	1B	1A	1A	1A
ASTM D130 at 150C	4A	1B	3B	3B	3B	1A	1A	1A
Semi submerged test								
Liquid	3B	1B	3B	3B	1A	1A	1A	1A
Vapor	4C	4A	3B	2C	2C	1A	1A	1A

[0091] Each of the documents referred to above is incorporated herein by reference, including any prior applications, whether or not specifically listed above, from which

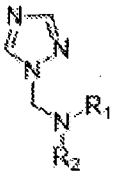
priority is claimed. The mention of any document is not an admission that such document qualifies as prior art or constitutes the general knowledge of the skilled person in any jurisdiction. Except in the Examples, or where otherwise explicitly indicated, all numerical quantities in this description specifying amounts of materials, reaction conditions, molecular weights, number of carbon atoms, and the like, are to be understood as modified by the word "about." It is to be understood that the upper and lower amount, range, and ratio limits set forth herein may be independently combined. Similarly, the ranges and amounts for each element of the invention can be used together with ranges or amounts for any of the other elements.

[0092] As used herein, the transitional term “comprising,” which is synonymous with “including,” “containing,” or “characterized by,” is inclusive or open-ended and does not exclude additional, un-recited elements or method steps. However, in each recitation of “comprising” herein, it is intended that the term also encompass, as alternative embodiments, the phrases “consisting essentially of” and “consisting of,” where “consisting of” excludes any element or step not specified and “consisting essentially of” permits the inclusion of additional un-recited elements or steps that do not materially affect the essential or basic and novel characteristics of the composition or method under consideration.

[0093] While certain representative embodiments and details have been shown for the purpose of illustrating the subject invention, it will be apparent to those skilled in this art that various changes and modifications can be made therein without departing from the scope of the subject invention. In this regard, the scope of the invention is to be limited only by the following claims.

What is claimed is:

1. A method of lubricating an automotive device having electrically conducting componentry comprising
    a. providing an automotive device comprising electrically conducting componentry, some portion of said componentry being dry,
    b. delivering to the automotive device a lubricant composition comprising
        i. an oil of lubricating viscosity,
        ii. an azole compound capable of inhibiting corrosion of the electrically conductive componentry in the vapor space above the lubricant composition comprising
            1. an N-substituted 1,2,4 triazole



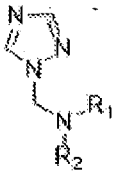
where R_1 and R_2 are C3-C22 hydrocarbon groups, either linear or branched, or two ends of a hydrocarbon chain forming a cyclic structure, or where one of R_1 or R_2 is H, or

            2. an azole-acrylic adduct formed by contacting an azole compound with an acrylate ester, acrylic acid, acrylamide or combination thereof; and said adduct has at least one nitrogen-alkyl group comprising at least one acyl group,
    c. operating the automotive device.
2. The method of claim 1, wherein the electrically conducting componentry comprises electrical wires, electrical sensors, printed circuit boards, or an electric motor.
3. The method of claim 1 or 2, wherein the electrically conducting componentry comprises copper or a copper alloy.
4. The method of claim 1 or 2 or 3, wherein the N-substituted 1,2,4 triazole comprises *N,N*-bis(2-ethylhexyl)-1*H*-1,2,4-Triazole-1-methanamine.

AMENDED SHEET

Date Reçue/Date Received 2020-12-07

5. The method of claim 1 or 2 or 3 or 4, wherein the lubricant composition further comprises a volatile compound corrosive to the electrically conducting componentry.
6. The method of claim 5, wherein the volatile compound corrosive to the electrically conducting componentry comprises a volatile sulfur-containing compound.
7. The method of claim 5, wherein the source of volatile sulfur comprises a thiol, or the reaction product of a substituted thiadiazole and a hydrogen phosphite.
8. The method of claim 1 or 2 or 3 or 4 or 5 or 6 or 7, wherein the automotive device comprises a transmission.
9. The method of claim 8, wherein the transmission is a dual clutch transmission.
10. The method of claim 8, wherein the transmission is driven by an electric motor.
11. The method of claim 1 or 2 or 3 or 4 or 5 or 6 or 7, wherein the automotive device comprises an axle.
12. The method of claim 11, wherein the axle is driven by an electric motor.
13. The method of any previous claim, further comprising a thiadiazole corrosion inhibitor in the absence of a phosphite.
14. A lubricant composition comprising
    a. an oil of lubricating viscosity,
    b. an azole compound capable of escaping the lubricant composition and inhibiting corrosion in a space above the lubricant composition comprising
        i. an N-substituted 1,2,4 triazole



where R_1 and R_2 are C3-C22 hydrocarbon groups, either linear or branched, or two ends of a hydrocarbon chain forming a cyclic structure, or where one of R_1 or R_2 is H, or

        ii. an azole-acrylic adduct formed by contacting an azole compound with an acrylate ester, acrylic acid, acrylamide or combination thereof; and said adduct has at least one nitrogen-alkyl group comprising at least one acyl group, and

c. at least one of a Lewis acid, Bronsted acid, or Lewis base.

15. The composition of claim 14, wherein the N-substituted 1,2,4 triazole comprises *N*,*N*-bis(2-ethylhexyl)-1*H*-1,2,4-Triazole-1-methanamine.

16. The composition of claim 14 or 15, further comprising a volatile compound corrosive to electrically conductive componentry.

17. The composition of claim 16, wherein the volatile compound corrosive to electrically conductive componentry comprises a volatile sulfur-containing compound.

18. The composition of claim 16 or 17, wherein the source of volatile sulfur comprises a sulfurized olefin, a thiol, such as those formed from the reaction product of a substituted thiadiazole and a hydrogen phosphite, or a combination thereof.

19. The composition of any of claims 17 to 26, further comprising a thiadiazole corrosion inhibitor in the absence of a phosphite.

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