

ANTIFREEZES AND DEICING FLUIDS

An antifreeze is defined as a chemical which, when added to a water-based fluid, reduces the freezing point of the mixture. Antifreezes are used in a wide variety of mechanical equipment during the winter months to prevent freezing of aqueous heat-transfer fluids. Most commonly, antifreeze refers to the freeze-protected fluid which cools automotive engines, although antifreeze liquids are also used in ice skating rinks, refrigeration systems (as a secondary coolant), heating and air conditioning systems, solar energy units, and many other applications. Chemical antifreezes include brines, such as calcium chloride; alcohols, such as methanol, ethanol, and 2-propanol; and glycerol and glycols. Since 1960 ethylene glycol has held the majority of the antifreeze market share because of its availability and superior performance characteristics.

1. Colligative Properties

Many chemicals when added to water cause a freezing point depression, as shown in Table 1, and thus are termed antifreezes. The antifreeze properties of these chemicals vary widely as a function of their colligative, or concentrative, properties. The reduction in freeze point depends both on the chemical itself and the concentration of the chemical in water. The freeze point depression increases as the antifreeze chemical is added to the water, until a characteristic concentration is achieved. Further addition of the antifreeze chemical to water will either result in insolubility or serve to increase the freezing point of the mixture, as illustrated in Figure 1.

The colligative properties of antifreeze chemicals may also result in boiling point elevation. As the chemical is added to water, the boiling point of the mixture increases. Unlike the freeze depression, the boiling elevation does not experience a maximum; the boiling point versus concentration curve is a smooth curve that achieves its maximum at the 100% antifreeze level. The boiling point elevation can be another important characteristic for antifreeze fluids in certain heat-transfer applications.

2. History of Antifreeze

Antifreeze solutions are found in many heat-transfer applications. For heat transfer, plain water provides superior performance, low cost, and universal availability. However, water alone has the very limited operating temperature range of 0 to 100°C. The antifreeze fluid increases this range by depressing the freezing point and elevating the boiling point, while still providing good heat transfer. This enables a water-based heat-transfer system to operate outdoors during the winter months, as does an automobile; and to operate at higher temperatures and thus higher efficiencies, as do large internal combustion engines.

Early antifreeze solutions were predominantly salt brines and alcohols. Glycerol and sugar solutions were used to a limited extent. Salt brines were relatively inexpensive and widely available, and gave efficient freeze point depression. However, salt solutions proved to be corrosive to the metals of construction and caused scaling

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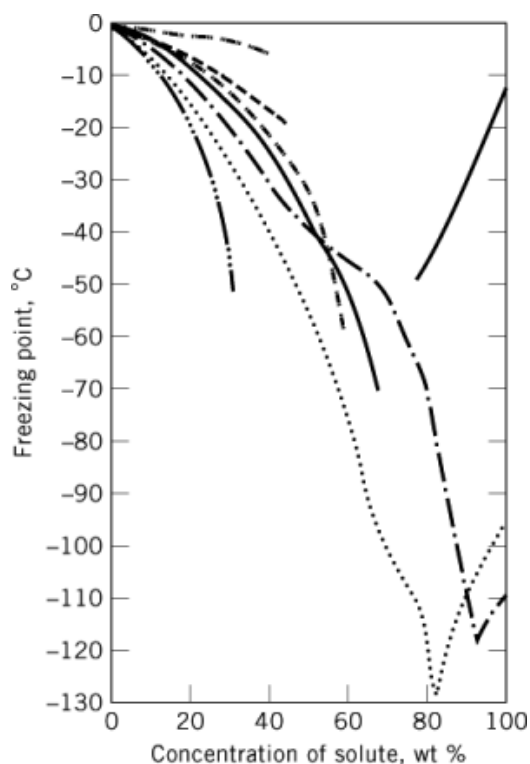


Fig. 1. Freeze point depression as a function of solute concentration (1, 2). Calcium chloride (.....), sucrose (.....), and urea (.....) become insoluble above maximum concentration given. Ethylene glycol (—) forms an eutectic at 65–80% solute concentration. Freezing points cannot be measured in this region. Propylene glycol (— · — · —) supercools at higher concentrations. Freezing points cannot be measured. Ethanol (—) and methanol (.....) are also included.

on heat-transfer surfaces. Scaling and precipitation of the salt drastically reduces heat transfer and system efficiency. Today, because of its low cost, brine solutions still find use in some antifreeze applications including refrigeration systems, off-shore heat-transfer systems (seawater), and roadway deicing; but are not used for cooling of automotive engines and other internal combustion engines.

Like brines, alcohols were readily available and widely used as antifreeze liquids in the early 1900s. Both methanol and ethanol offer excellent heat transfer and efficient freeze point depression. However, the alcohols have the distinct disadvantage of their low boiling points. During the summer months when the engines operate hot, significant amounts of the alcohols are lost because of evaporation. These evaporative losses result in costly make-up requirements. Additionally, the alcohols have very low flash points and potentially flammable vapors. These safety concerns have, particularly in recent years, caused the use of alcohols to be completely discontinued for most heat-transfer systems.

The use of ethylene glycol as an antifreeze was first initiated in 1925. Although not as efficient a freeze point depressant as methanol, ethylene glycol has a very low vapor pressure so that evaporative losses from a cooling system are water vapor rather than glycol vapor. This reduces make-up costs and ensures that the freeze protection is maintained in the system. Thus, glycol-based antifreezes are termed permanent antifreezes. Ethylene glycol (EG) also eliminated the flammability concerns associated with methanol-based antifreeze. Ethylene glycol-based antifreeze solutions (50%) have no flash point.

Table 1. Freeze Point Depression of Antifreeze Chemicals^a

Component	CAS Registry Number	Molecular formula	Concentration in water, wt %	Freeze point depression, °C
calcium chloride	[10043-52-4]	CaCl ₂	32 ^b	-50
calcium magnesium acetate	[76123-46-1]	C ₂ H ₄ O ₂ ·xCa·xMg	32.5 ^b	-28
ethanol	[64-17-5]	C ₂ H ₆ O	50	-38
ethylene glycol	[107-21-1]	C ₂ H ₆ O ₂	50	-22
glycerol	[56-81-5]	C ₃ H ₈ O ₃	50	-22
methanol	[67-56-1]	CH ₄ O	50	-50
potassium acetate	[127-08-2]	C ₂ H ₄ O ₂ ·K	50	-50
potassium chloride	[7447-40-7]	KCl	13 ^b	-6.5
propylene glycol	[57-55-6]	C ₃ H ₈ O ₂	50	-32
seawater			100 (6% salt)	-3
sodium chloride	[7647-14-5]	NaCl	23 ^b	-21
sucrose	[57-50-1]	C ₁₂ H ₂₂ O ₁₁	42 ^b	-5
urea	[57-13-6]	CH ₄ N ₂ O	44 ^b	-18

^a Refs. (1–3).^b Saturated solution at this concentration. Becomes insoluble at higher concentrations.**Table 2. United States Consumption of Ethylene Glycol-Based Automotive Antifreeze^a**

Year	Consumption, 10 ⁶ L ^b
1965	534
1970	672
1975	824
1980	738
1985	881
1986	727
1987	731
1988	818
1989	606

^a Ref. 5.^b To convert from liters to U.S. gallons, divide by 3.79.

For automotive engine cooling, ethylene glycol or methanol were the predominant products from the late 1920s to early 1950s. In 1927, the first formulated EG-based fluid was introduced by Union Carbide (4). *Prestone* brand antifreeze contained ethylene glycol with additives to prevent corrosion and foaming in the engine. By 1960, 90% of the automotive antifreeze market was using formulated ethylene glycol-based products. The consumption of EG-based antifreeze is shown in Table 2, as defined by the Chemical Specialty Manufacturers Association (5). In 1990 automotive antifreeze represented approximately 33% of the total ethylene glycol consumption in the United States (6). The primary antifreeze manufacturers include BASF Corp., First Brands Corp., Old World Trading Co., Shell Chemical Co., and Texaco Chemical Co.

Ethylene glycol continues to be the antifreeze of choice for cooling of internal combustion engines. EG-based antifreezes may contain a few percent of other glycols, specifically diethylene and triethylene glycols. However, these other ethylene-based glycols, or the propylene-based glycols (propylene glycol (PG) and dipropylene glycol), are not widely used as the primary component of engine coolants. Ethylene glycol provides both the most effective freeze point depression, as well as the best heat-transfer characteristics (see Glycols). Propylene

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glycol-based antifreezes do have some limited usage in Europe (specifically Switzerland and Austria), where human toxicity concerns override the performance and environmental benefits of ethylene glycol.

3. Engine Coolants

Besides freeze protection, antifreezes provide many other performance properties that enhance the operation of a heat-transfer system. Because the internal combustion engine is by far the largest antifreeze application, and ethylene glycol is the predominant antifreeze in use, the following focus on the performance properties of an ethylene glycol-based antifreeze and their relationship to engine cooling.

3.1. Heat Transfer

The primary role of the antifreeze liquid in an internal combustion engine is to remove heat and thus cool the engine. If heat is not removed, the engine will be destroyed. The cooling system must remove about one-third of the heat generated by the engine; one-third is removed by the exhaust system; and the balance of the heat is converted into mechanical energy. The antifreeze fluid removes heat by passing through the engine where it picks up heat, then through the radiator where it is cooled by air. The fluid operates in a closed loop system and thus is continuously reused. To provide efficient cooling, the antifreeze must have a high specific heat and thermal conductivity and low viscosity at operating temperatures. Water alone provides the most efficient heat transfer, as defined by the heat-transfer coefficient (HTC). The higher the heat-transfer coefficient, the greater the efficiency of the system. The HTC is directly proportional to the thermal conductivity (k), the specific heat (C_p), and the density of the fluid (ρ), and is inversely proportional to the viscosity (μ). The addition of antifreeze to the cooling system results in a slight penalty in heat-transfer efficiency by decreasing the HTC because of the lower specific heat and higher viscosity of the aqueous glycol solution.

3.2. Freeze Point Depression

The slight heat-transfer penalty incurred when an antifreeze is added to the aqueous heat-transfer fluid is necessitated by the need for increased operating temperature range in most internal combustion engines. Because most parts of the world achieve temperatures below freezing during some time of the year, an antifreeze fluid is required to keep equipment operational in these subfreezing temperatures.

The colligative properties of antifreeze fluids dictate the freeze point depression of the antifreeze solution. The freeze point depression offered by a chemical is dependent on the concentration of that substance in the solution; ie, as the concentration of the solute is increased, the freezing point of the solvent is depressed. Most solutes reach a maximum freezing point depression at a characteristic concentration. When this concentration is exceeded, either the solute becomes insoluble or the freezing point begins to increase. For ethylene glycol solutions, the maximum freezing point is achieved by using a 68% aqueous solution; this concentration is known as the eutectic concentration. Antifreeze concentrations in excess of 68% have higher freezing points; lower concentrations offer less freeze point depression, as indicated in Figure 1. Thus it seems ideal to use this 68% solution to give the maximum freeze protection. However, because water has the best heat transfer and heat transfer is sacrificed as the antifreeze concentration is increased, it is recommended that lower concentrations be used if local weather conditions permit. For automotive applications, a 50% solution is usually standard: the -36.7°C freezing point provides adequate freeze protection in most areas of the continental United States; heat-transfer characteristics do not compromise engine efficiency; this is typically the concentration used as the basis for design of the cooling system. In larger internal combustion engines, particularly stationary compression engines, where tremendous fuel savings can be achieved by maximizing cooling efficiency, the concentration of

coolant chosen usually gives a freezing point 3–6°C (5–10°F) below the lowest historical ambient temperature for the region.

When defining the desired concentration and thus the use temperatures for an antifreeze fluid, several factors should be taken into consideration. First, the freezing point of an antifreeze is defined as the temperature at which the first ice crystal is formed in the fluid. Below this temperature, the fluid will not function efficiently as a heat-transfer fluid; however, at this temperature the fluid will not freeze to a completely solid state, thus protecting the system from the expansion that occurs upon complete freezing. In contrast, water freezes solid when the temperature is maintained at its freezing point of 0°C (32°F), and undergoes an expansion of 9% (7). The addition of an antifreeze to the water significantly reduces the amount of expansion that the fluid undergoes, thus protecting the system from bursting. For glycol solutions, the amount of expansion upon freezing decreases as the amount of glycol in solution increases (Fig. 2). For example, a 20% glycol solution expands 5% as the temperature is lowered 19.4°C below its freezing point; a 30% solution expands 3.3% at 22°C below its freezing point (8). After the first ice crystal formation at the fluid's freezing point, a continued drop in temperature is accompanied by additional crystal formation to produce a thick, slushy state. For higher glycol concentrations, a completely solid state is never achieved; rather the fluid becomes thick and taffylike. The temperature at which the fluid ceases to flow, defined as the pour point, is significantly lower than the freezing point. In systems that are not operational during the winter months, a fluid concentration is chosen to protect the system against bursting, not freezing, since some crystal formation is not harmful. However, it is important to remember that the use of a fluid down to its pour point may require significant pumping power to restart the system during these cold temperatures because of the drastic increase in viscosity as the fluid becomes slushy. Additionally, because of the poor heat transfer of these slushy fluids, systems that anticipate cold weather operation should choose a fluid having a freezing point below the coldest expected winter temperature.

3.3. Boiling Point Elevation

Boiling point elevation may also be beneficial in heat transfer applications. As engine power increases, the heat given off by the engine also increases. This increase in engine operating temperature increases engine efficiency by making more heat available to power the engine. However, if too much heat remains in the engine, overheating of both the engine and the oil may occur and engine efficiency decreases. The extra cooling required can be supplied either by increasing the cooling system capacity, or by enabling the coolant to accept more heat. An increase in the capacity of the cooling system is a costly alternative that can require physical changes to the system. Enabling the coolant to remove more heat most often requires no capital changes, and can be achieved by either increasing the flow rate of the coolant, or increasing the system pressure, and thus the boiling point of the coolant, which would allow the coolant to circulate at a higher maximum temperature. Today, most engines operate slightly pressurized to increase the maximum use temperature of the coolant and thus eliminate boiling of the coolant under extreme operating conditions. Under these operating conditions, the very low vapor pressure of glycol-based coolants significantly reduces the evaporative losses of the coolant, when compared to high vapor pressure liquids such as methanol and ethanol.

3.4. Corrosion Inhibition

Another important property of antifreeze solutions is the corrosion protection they provide. Most cooling systems contain varied materials of construction including multiple metals, elastomeric materials, and rigid polymeric materials. The antifreeze chosen must contain corrosion inhibitors that are compatible with all the materials in a system. Additionally, the fluid and its corrosion inhibitor package must be suitable for the operating temperatures and conditions of the system.

Of the commonly used chemical antifreezes, salt brines are inherently the most corrosive. The alcohols and glycols, in their pure forms, are generally noncorrosive to common metals of construction. However, as

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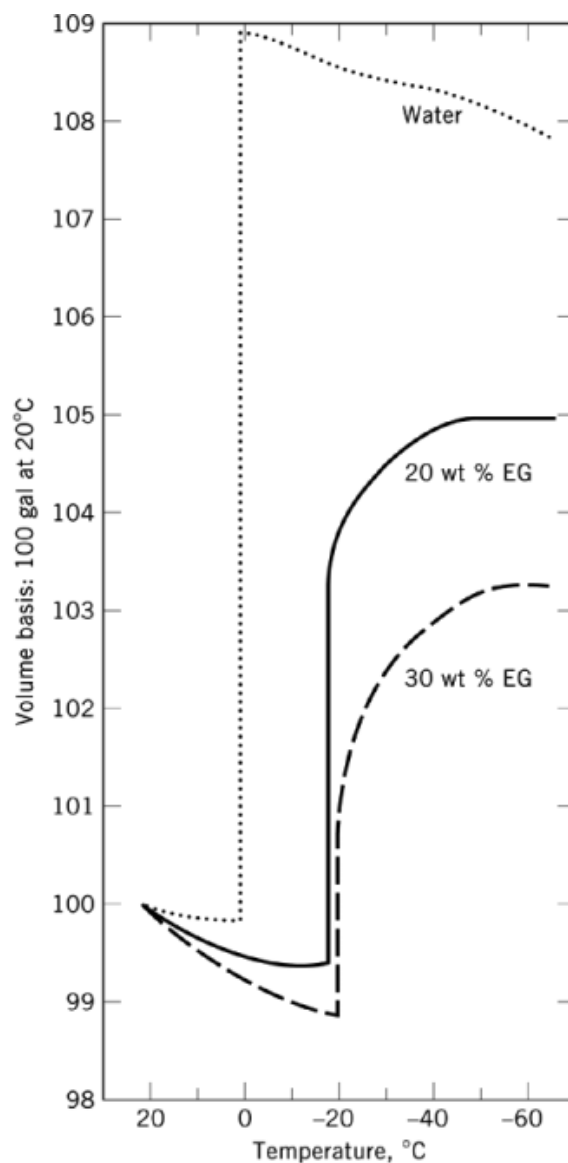


Fig. 2. Expansion of water and ethylene glycol (EG) on freezing (7, 8). 100 gal=377 L.

water is added to these compounds to reduce the freezing point, the aqueous antifreeze solution assumes the corrosivity of the water of dilution. Because water sources contain varying amounts of corrosive ions such as chloride and sulfate ions, antifreeze solutions contain inhibitors to counteract these ions. Additionally, in the case of glycol solutions, some corrosion inhibitors also act as buffering agents to counteract the slow oxidation of the glycol. At high temperatures, in the presence of oxygen, all glycols are slowly oxidized to form acids of degradation. These acids, if not adequately neutralized, are corrosive to the metals in the system.

The corrosive effects of water and heat-transfer fluids, if untreated, can be detrimental to the heat-transfer system. Corrosion in a system is first evidenced by the presence of corrosion products in solution, either in the

form of solid particles in the fluid, or as metal ions in solution with the fluid. The corrosion products in the fluid alter the heat-transfer characteristics of the fluid and may reduce its heat-transfer efficiency. Heat transfer is further reduced because of the irregular metal surface left behind as corrosion products are entrained by the fluid. The higher surface area of these irregular surfaces require a higher concentration of corrosion inhibitors to cover and protect the metal surface. Inadequate concentrations of corrosion inhibitors result in the presence of bare metal surfaces, an open invitation to corrosion. Corrosion is further accelerated as the metal ions in solution react with the active corrosion inhibitor ions, and render them incapable of protecting the system against further corrosion. The solid corrosion products flowing with the fluid may also cause erosion corrosion, in which the solid particles impacting the metal surfaces break down the protective inhibitor layers and allow corrosion to occur. These erosive effects are most pronounced in high velocity points within the system. Another corrosion phenomena is known as transport deposition, where the corroded metal is dissolved in the fluid (usually at the engine), then deposited in another point of the system (usually the radiator), thus reducing cooling efficiency. As these various forms of corrosion continue unthwarted, the amount of solid corrosion products in the system increases, and eventually builds to the point where the solids may clog lines and restrict flow through the system. Ultimately, the lack of cooling in the engine as a result of all of these factors results in engine failure.

Corrosion reduction can be achieved either by altering the fluid or the system operation. During operation of the heat-transfer system, many factors may accelerate corrosion. Because corrosion is an oxidative process, the most critical factor is the amount of oxygen in the system. The presence of oxygen allows metal surfaces to oxidize or corrode; additionally, in glycol systems, this oxygen accelerates the oxidative degradation of the glycol to form corrosive acids. Both of these processes are accelerated by the elevated temperatures in the engine. Another factor affecting the tendency for a system to corrode is the presence of contaminants in the system. Contaminants can range from dirt and corrosion particles to oils to the ions in poor quality water. These contaminants affect the system in varied ways, such as air entrainment, erosion corrosion, reaction with the corrosion inhibitors, and glycol or inhibitor degradation. These factors make it important to operate the system as cleanly, and as oxygen-free as possible.

The first glycol-based antifreeze, preformulated with corrosion inhibitors, was introduced in the United States in 1927. Since that time, many and varied antifreeze formulations and brands have been used in automotive cooling systems. Today, all antifreezes contain inhibitors designed to minimize corrosive attack, including both organic and inorganic compounds. The most common corrosion inhibitors for glycol-based antifreezes include borates, molybdates, nitrates, nitrites, phosphates, silicates, amines, triazoles, and thiazoles (9, 10). Since no single compound provides complete corrosion protection to all metals under all conditions, antifreeze formulations contain a combination of these inhibitors. Additionally, since cooling system design varies from automobile to automobile, and from year to year, the antifreeze formulation must have enough latitude to account for these changes and yet maintain adequate corrosion protection for most systems.

The choice of corrosion inhibitors for an antifreeze is also dependent on the type of service of the engine. Engine cooling systems can be categorized into three types: light-duty automotive, heavy-duty diesel, and continuous stationary engine operations. For automotive applications, the engine operates intermittently. The corrosion inhibitors must protect the system during operation and while idle. For this service, film-forming inhibitors such as silicates are predominantly used. Silicates are particularly useful for corrosion protection of heat-emitting aluminum surfaces such as that found in an aluminum head engine. Silicates have been more widely used as automobile manufacturers have tried to reduce car weight to improve fuel efficiency by increasing the use of aluminum in engines. The film-forming silicates effectively protect the system while idle (most automobiles remain idle greater than 50% of the time), but have the disadvantage of reducing the heat-transfer efficiency of the coolant. Additionally, with time, silicates react adversely with the glycol and any salts or hardness ions in the fluid (phosphates, calcium) to form a gel. The gel severely reduces heat transfer and may clog lines. If untreated, silicate gel formation may cause engine failure. Many antifreeze manufacturers now

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include a silicate stabilizer to retard this reaction, and recommend frequent (annual or biannual) antifreeze replacement. Nonetheless, for light-duty automotive service, silicates work effectively to inhibit corrosion.

For heavy-duty diesel service, on the other hand, high concentrations of silicates are usually not recommended because of the silicate–glycol reaction. Heavy-duty diesel service implies more continuous operation of the engine, and therefore, a heavy film-forming compound is not required to protect during long idle periods and is undesirable because of its adverse effects on heat-transfer efficiency. Also, aluminum is typically not used in heavy-duty engines. For diesel service, a low silicate formulation is used in conjunction with a supplemental coolant additive (SCA). SCAs are important in systems where cavitation of water pumps and cylinder liners is frequently evidenced. SCAs supplement the concentration levels of inhibitors in the base coolant, and are frequently replenished throughout the life of the coolant.

Inhibitor replenishment is also important in stationary engine operation, where the large volume and continuous operation of the engine prohibit frequent fluid replacement, because of the high cost and down time constraints. For stationary engines, such as those used in natural gas transmission applications, even minute concentrations of silicates must be avoided, because of the rapid gel formation and loss of heat transfer during the continuous operation of these engines. Coolant formulations for stationary engines are different from automotive and diesel formulations; they predominantly consist of phosphate for ferrous metal protection and buffering and a triazole for protection of the yellow metals. Periodic analysis of these fluids is used to determine the type and amount of inhibitor replenishment required.

3.5. Foaming

Another factor in maintaining heat-transfer efficiency and good system operation, is minimizing the foaming tendency of the fluid. Foaming or air entrainment in the fluid minimizes the amount of fluid that is in contact with the heat-transfer surface, and thus reduces the amount of heat that the fluid can remove from the system. Air entrainment may also cause a form of corrosion in the system: as the air bubbles break against the walls of the system, the pressure of them breaking may cause a phenomena called cavitation erosion corrosion. Although air entrainment alone can probably not cause system failure from cavitation, it may work in conjunction with mechanical forces such as vibration. The combination of these two forces is detrimental and frequently causes system failure, particularly of the cylinder liner.

Although glycol–water solutions are not inherently prone to foaming, mechanical and chemical conditions may cause foam to form in the system. To minimize foaming and thus the possibility of cavitation failure and the loss of heat transfer, the amount of air entrained in the coolant must be minimized. Air is often entrained in the coolant expansion tank; increasing the level in this tank to recommended levels minimizes the air entrained. Other causes of foaming include the use of organic corrosion inhibitors and the presence of various contaminants in the fluid. To counteract these effects, most antifreeze formulations contain antifoaming agents, such as silicones, polyglycols, or oils. Often the antifoamer functions by being insoluble in the glycol solution and appears as a separate phase when not circulating. In the system, the antifoamer is well-dispersed and not evident as a second phase (see Defoamers).

3.6. Effect on Elastomers

The antifoamers and corrosion inhibitors used in antifreeze formulations are designed not only to function effectively in their assigned role, but also to be compatible with the other materials of construction in the system, such as the elastomers and plastics. Various components in the coolant can swell and soften elastomers or cause them to become brittle and crack. In general, ethylene glycol causes the elastomer to shrink when compared to the effect of pure water. Therefore, once a system is charged with a specific type of coolant, replacement with another chemical may require replacement of the gaskets and hoses in the system. This is most often seen when plain water is used during the summer months to achieve the maximum heat-transfer efficiency, then

Table 3. Compatibility of Ethylene Glycol with Elastomeric Materials^a

Trade name or common name	Material	Temperature		
		25°C	80°C	160°C
Adiprene L-100 ^b	urethane	good	poor	poor
Black Rubber 3773	polyisoprene, synthetic	good	poor	poor
Buna N	acrylonitrile–butadiene	good	good	
Buna S	styrene–butadiene	good	fair	poor
Butyl Rubber	isobutylene–isoprene	good	good	
EPDM	ethylene–propylene–diene	good	good	good
EPR Rubber	ethylene–propylene rubber	good	good	good
Hycar, D-24 ^b	butadiene–styrene, butadiene–acrylonitrile, acrylate emulsion	good	fair	
Hypalon ^b	chlorosulfonated polyethylene	good	poor	poor
Kalrez ^b	tetrafluoroethylene, perfluoromethyl vinyl ether cure site monomer	good	good	good
Natural Rubber Gum	polyisoprene, natural	good	poor	poor
Neoprene 7797	chloroprene	good	fair	
Red Rubber #107	polyisoprene, synthetic	good	poor	poor
Silicone #65	polydimethylsiloxane	good	good	
Viton A ^b	vinylidene fluoride and hexafluoropropylene	good	good	poor

^a Ref. 2 (see Elastomers, synthetic).^b Registered trademarks.

glycol is added for winter operations. The shrinkage of the elastomeric gaskets with the addition of the glycol may cause leakage in the system. Table 3 shows the compatibility of various elastomers with ethylene glycol.

3.7. Service Life

The service life offered by a coolant is dependent on many factors, including the initial condition of the coolant and the cooling system, the type of water used for dilution, the metals of construction in the system, the type of corrosion inhibitors and SCAs used, the system operating temperature, and the type of operation the coolant is in: light-duty/intermittent, heavy-duty/diesel, or continuous stationary. Before initial charging of a cooling system with fresh coolant, the system should be free of dirt, oil, and other contaminants. New cooling systems probably only require a water flush to remove solids introduced during assembly of the system. Older systems may require chemical cleaning to remove corrosion products, residual spent coolant, and other contaminants such as scale and oils. The addition of fresh coolant to an improperly prepared system results in rapid deterioration of the condition of the fresh fluid.

Similarly, the antifreeze itself must be prepared properly to extend the life of the fluid. Because many antifreeze solutions contain inorganic corrosion inhibitors such as phosphates and molybdates, the fluids are sensitive to dilution by hard water. Hard water ions, calcium and magnesium, complex with the corrosion inhibitors and render them ineffective in inhibiting corrosion. Additionally, the calcium and magnesium salts of the corrosion inhibitors are typically insoluble in aqueous solutions, and frequently cause scale formation on the walls of the system, thus reducing heat transfer. Consequently, the use of very hard water should be avoided, particularly in the larger engines that require greater heat-transfer efficiency. In addition to the hardness ions, poor quality water of dilution can result in the introduction of corrosive ions, chloride and

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sulfate, to the system. The use of corrosive water must be avoided to ensure adequate corrosion protection and to extend the life of the fluid and the system.

The service life of an antifreeze can be affected both by the absolute time a coolant is in a system, and by the actual service hours of the engine. Thus, the type of engine operation and the type of corrosion inhibitors in the fluid play a significant role in determining the life of the fluid. Because of the presence of the silicate inhibitors, antifreeze in light-duty service is designed to be replaced frequently (annually or biannually). However, the size of the automotive engine cooling system ensures that the coolant replacement is not cost prohibitive. For larger engines, yearly fluid replacement would be costly in terms of both the fluid cost and the engine downtime cost. Consequently, antifreeze for heavy-duty diesel and continuous stationary operations are designed to be longer lasting and the corrosion inhibitors easily replenished. Additionally, because stationary engines have the capability of operating hotter to increase efficiency, coolants for these engines typically have greater buffering capacity to counteract the effects of the accelerated glycol degradation caused by the higher temperatures.

Despite all these safeguards to extend the service life of the antifreeze, fluid replacement is required periodically. Typically, fluids are replaced because of irreversible damage caused by one of four conditions: contamination, gel formation because of glycol/silicate reaction, extensive glycol degradation caused by over-heating or excessive oxygen exposure, or inhibitor depletion.

3.8. Fluid Maintenance

The frequency of fluid replacement can be minimized and the heat-transfer efficiency and overall condition of the system maximized by proper maintenance of the engine cooling system. The most critical requirement for an antifreeze is that it provides adequate freeze protection. Because of evaporative losses of water and system makeup, it is necessary to monitor the freezing point of the antifreeze to ensure adequate protection. The freezing point of a glycol-based fluid can be determined in the field by specific gravity or refractive index methods. Both methods measure the amount of glycol in the solution, and from this the freezing point can be determined from the literature or from precalibrated measuring devices. An automotive hydrometer, measuring the fluid's specific gravity, is calibrated to read the freezing point of the ethylene glycol-based fluid directly. Because of differences in the various brands of antifreeze, the automotive hydrometer will provide the freezing point within 4.4°C (8°F) of the actual freezing point, but should not be relied on for greater accuracy. A laboratory hydrometer or pycnometer, which reads in specific gravity, provides a high degree of accuracy; however, literature values correlating specific gravity to freezing point for the specific brand of coolant are required. A handheld refractometer is another convenient method for field determinations of the coolant's freezing point. The refractometer is more accurate and more expensive than the automotive hydrometer, reading to within 1.67°C (+3°F) of the freezing point, but less accurate than a laboratory hydrometer or pycnometer. Because the handheld refractometers are also calibrated directly in freezing point, the refractive index to freezing point correlation must account for the differences in the available coolants, and thus accuracy is sacrificed.

The freezing point of the coolant should be monitored for coolants in all types of service. Additionally, maintenance of the corrosion inhibitor levels is required of the heavy-duty service coolants and the stationary engine coolants. Because corrosion inhibitors and combinations of corrosion inhibitors work most effectively at given concentrations and specific ratios to the other inhibitors, appropriate concentrations must be maintained to maximize corrosion protection. Many manufacturers of coolants for stationary engines, and manufacturers of SCAs, provide an analytical service to monitor the effective inhibitor concentrations in the system periodically. Recommendations can then be made for proper maintenance and inhibitor replenishment.

3.9. Fluid Specifications

The performance characteristics of all antifreeze solutions are governed by fluid specifications, that have been developed over the years by industry standards committees, such as the American Society for Testing and Materials (ASTM) and the Society of Automotive Engineers (SAE). Additionally, most engine and/or cooling system manufacturers have their own compositional specifications to which the fluids must conform.

ASTM D-3306 is a long-standing performance specification for engine coolants, under the jurisdiction of ASTM Committee D-15 on Engine Coolants (11). Most recently, as the varying performance requirements for light-duty versus heavy-duty engines were defined, D-3306 has been renamed and reclassified to apply specifically to coolants for light-duty and automotive applications. A separate specification, D-4985, has been developed to account for the needs of heavy-duty service. In addition to this reclassification of the performance requirements for engine coolants, several additional specifications are being developed to respond to the changing needs of the industry including a specification for prediluted coolants (diluted by the coolant manufacturer) that has been adopted by the ASTM committee. The need for specifications for propylene glycol-based coolants is also being reviewed. PG-based fluids currently find limited application in Switzerland and Austria where human toxicity concerns prohibit the use of EG-based coolants. PG is also being proposed in several developmental applications, such as anhydrous cooling systems that permit engine operation at higher temperatures and cavitation control in heavy-duty service, where the higher viscosity of PG is reported to be beneficial. Lastly, a specification for the use of recycled coolants is being explored. The many and varied modes of recycling provide vastly different performance characteristics of the recycled product, and a standard is needed to govern these fluids.

4. Deicing Fluids

Antifreeze chemicals, because of their characteristic freeze point depression, find additional applications in the deicing of aircraft and runways. Because of the stringent requirements of corrosion protection of aircraft surfaces and the necessity of a low flash point for use around aircraft engines, aircraft deicing fluids use glycols exclusively (over brines and alcohols) to achieve freeze point depression; runway deicing is commonly achieved with either urea or glycol-based fluids that contain urea, although two acetate salts are being studied for runway usage. Calcium magnesium acetate and potassium acetate are being reviewed by the Federal Aviation Administration (FAA) and the Air Transport Association (ATA).

4.1. Runway Deicing

Deicing compounds are applied to the runway under certain conditions to assist in the removal of frozen accumulations of snow and ice that cannot be readily removed by mechanical means such as plowing. The primary purpose of chemical deicing is not to melt the surface ice and snow, but rather to diffuse through the snow and ice to break the bond between the ice and the runway. Once the ice-surface bond is broken, the remaining frozen accumulation can then be removed by mechanical means. The use of chemical deicing often reduces the amount of time and equipment used in snow removal. Runway antiicing can also be achieved by applying certain deicing compounds in anticipation of freezing rain, snow, or icing conditions. It is critical that both deicing and antiicing be conducted in a closely controlled manner where the application rate is monitored and the runway is monitored for slipperiness. Improperly applied runway deicing fluids may create a slippery condition.

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4.2. Aircraft Deicing

Aircraft deicing fluids typically contain at least 90% glycol, with the balance being water, corrosion inhibitors, wetting agents, and an orange dye. In North America, ethylene glycol is the most commonly used freeze point depressant, although other glycols, such as diethylene glycol, triethylene glycol, and propylene glycol, as well as mixtures of these, are also used. The corrosion inhibitors are necessary to protect the materials of construction of the aircraft; wetting agents ensure complete coverage of the surfaces being deiced. Deicing fluids are typically diluted prior to use, to achieve maximum freeze point depression; aqueous glycol solutions achieve their minimum freezing point at about 68 wt % deicing fluid and 32% water.

Deicing is defined as a method for removing ice, snow, and frost accumulations from the surfaces of aircraft during ground operations. These frozen deposits, if untreated, significantly affect the aerodynamic performance of the aircraft by reducing lift and altering thrust and drag characteristics. Additionally, severe damage to the engines may occur if ice buildup on the wings dislodges during the takeoff run and strikes the turbine fan blades. Removal of frozen accumulations from aircraft surfaces prior to takeoff is required by the FAA's clean aircraft concept which states that "flight following ground operations conducive to aircraft icing should not be attempted unless it is ascertained that the aircraft is free of any ice formations that may degrade the aircraft's performance or change its flight characteristics" (12).

To remove frozen accumulations, deicing fluids are applied at high temperatures ($\sim 71 - 82^{\circ}\text{C}$) and pressures to melt the ice, snow, and frost, and to break the adhesion to the surface of the aircraft. Deicing leaves the aircraft surfaces wet and therefore, to guard against refreezing, the deicing fluid must contain sufficient freeze point depressant to lower the freezing point of the residual fluid on the aircraft below the ambient or surface temperatures. The FAA Advisory Circular 20-117 recommends that "the freeze point of the residual fluids should not be greater than 11°C (20°F) below ambient or surface temperature, whichever is less" (12). In the absence of further precipitation, the low freezing residual fluid will guard against refreezing. However, during precipitation or frost-forming conditions, deicing fluids offer time-limited protection against refreezing and ice or snow buildup. Precipitation will dilute the residual fluid, raising its freezing point, and thus freezing on the surface of the aircraft may occur. Additionally, dilution of the residual fluid by precipitation lowers the viscosity of the fluid, reduces the film thickness of the fluid remaining on the aircraft, and eventually flushes the fluid from the aircraft surfaces. For these reasons, the FAA recommends that a close inspection of the aircraft be performed immediately prior to takeoff to ensure that it is aerodynamically clean and free of ice, snow, or frost accumulations.

During precipitation or frost-forming conditions, several techniques may be used to maintain an aerodynamically clean aircraft following deicing and until rotation and takeoff. Most desirably, the deicing operation should be performed as close to the takeoff point as possible. End of the runway deicing is practiced at several airports in the United States. This procedure eliminates the possibility of refreezing prior to takeoff, since the takeoff roll commences immediately following deicing. End of the runway deicing is remote deicing since the deicing operation is removed from the gate and terminal area. Remote deicing can be performed either using a mobile facility which includes several deicing trucks working in tandem, or using a fixed facility, usually a "drive thru" station. One type of fixed facility, referred to as a "car wash" system, is a large structure which surrounds the aircraft and deices using multiple spray nozzles fixed to the structure at adjustable angles. Both systems, mobile and fixed, have distinct advantages. Mobile facilities lend flexibility for reconfiguring depending on storm conditions; and fixed facilities enable collection of spent deicing fluid and thus ease the management of storm water runoff.

Although end of the runway deicing is the only reliable method for ensuring that an aircraft is aerodynamically clean at takeoff during precipitation conditions, many airports are configured such that they do not permit end of the runway deicing. At these facilities, when long taxi times and delays are expected, antiicing techniques must be employed if takeoffs during precipitation are to be permitted. Antiicing is most often performed using a two-step procedure, in which the aircraft is first deiced using a hot, high pressure fluid, and

then antiiced using a cold fluid. Antiicing must only be performed following complete deicing. Antiicing fluids must only be applied to aerodynamically clean surfaces, and serve to protect the clean surfaces from further accumulations of frozen deposits for a finite period of time.

Traditionally, in North America, antiicing has been performed using cold, undiluted deicing fluids containing high glycol concentrations, so that when diluted with precipitation, the freezing point decreases and the residual fluid provides protection against refreezing. However, during certain weather conditions, because of the low film thickness and low viscosity of the fluid, the fluid can run off the surface of the aircraft before its freeze protection is depleted. Nonetheless, the use of neat deicing fluids for antiicing does provide some limited protection against refreezing during light precipitation conditions. At low volume airports where taxi times are low, neat deicing fluids work effectively to retard refreezing during taxiing and the takeoff roll under light precipitation and frost-forming conditions, and thus allow airport operations to proceed uninterrupted. Deicing fluids should never be used for antiicing purposes during heavy precipitation or freezing rain conditions.

Because of the time-limited protection that deicing fluids provide, and because of the rapidly increasing air traffic and subsequent longer taxi times, thickened, non-Newtonian antiicing fluids are being investigated for use at several of the larger airports in North America. The current antiicing fluids (AAFs) contain glycols for freeze point depression, with added water, corrosion inhibitors, wetting agents, and stabilizers. AAFs also contain a polymeric thickening agent that gives the fluid its antiicing properties. The thickening agent increases the fluid's viscosity and allows the fluid to adhere to the aircraft surfaces. Current AAFs contain a minimum of 50% glycol (usually 50–60%), and adequate water as required to solubilize the thickening agent. Because of this added water for solubilizing the thickener, the neat antiicing fluid has already achieved its minimum freezing point. Further dilution of the fluid increases the freezing point and reduces its protection against refreezing. Antiicing fluids are typically undyed; if dyed, they cannot be orange or blue, thus distinguishing AAFs from aircraft deicing fluids and runway deicing fluids.

The antiicing fluid's thickening agent gives the fluid its non-Newtonian behavior, and thus its antiicing protection. Non-Newtonian fluids are defined as fluids with a viscosity dependent on shear as shown in Figure 3. The fluid has a higher viscosity at low shear rates and a lower viscosity at higher shear rates. The non-Newtonian design is critical to its performance. When the aircraft is stationary or taxiing, the fluid is at low shear, the fluid's viscosity is high, and its high viscosity prevents flow and enables it to adhere to the aircraft surface. During the takeoff run, the fluid experiences high shear, its viscosity is reduced, and it flows from the aircraft (13).

Non-Newtonian antiicing fluids have been used in Europe for many years. Early non-Newtonian fluids had high viscosities of up to 50 Pa·s (500 P) at -25°C ; these fluids have been found to have adverse effects on the aerodynamic performance of the aircraft, reducing the lift coefficient at takeoff by greater than 12%. Current non-Newtonian fluids have substantially lower viscosities ($<10 \text{ Pa}\cdot\text{s} = 100 \text{ P}$) and lower lift losses of 2–7%, as compared to ethylene glycol-based deicing fluids, which have lift losses of 1–3% (14). The antiicing fluids, when applied to a clean aircraft, form a high viscosity, protective layer on the surface of the aircraft and block the adhesion of frozen deposits. However, the protection provided by these fluids is also time-limited. The amount of antiicing protection they provide, defined as the holdover time, varies tremendously with weather conditions (temperature and type and rate of precipitation), type of fluid used, and type of aircraft antiiced. Because of these varied and ever-changing conditions, the holdover times for antiicing fluids cannot be predicted. Inspection of the aircraft immediately prior to takeoff is necessary at all times to ascertain that all aerodynamic surfaces are free of adhering ice, snow, or frost.

Non-Newtonian antiicing fluids are currently being used in the United States on a limited, experimental basis. Antiicing fluids require special handling and application equipment and techniques, and therefore deicing equipment modification and personnel training are required before these fluids are used by an airline at an airport.

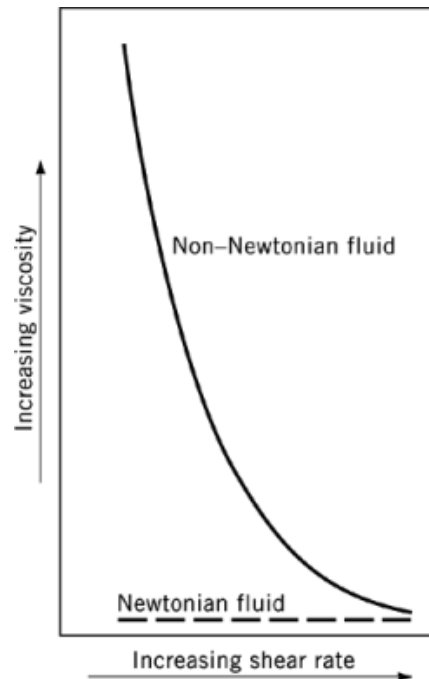


Fig. 3. Shear dependence of Newtonian versus non-Newtonian fluids (13).

4.3. Materials Compatibility

In addition to the runway deicing and aircraft deicing/antiicing fluids' primary roles of removing frozen accumulations and protecting against the refreezing of aircraft and runway surfaces, these fluids are subject to many other performance criteria, as defined by the airlines, the aircraft manufacturers, and the aircraft engine manufacturers. The fluids must contain corrosion inhibitors to minimize the corrosive effects on aircraft metals of construction. Additionally, the fluids must not adversely affect the acrylics and polycarbonates used for aircraft windows by crazing or staining, and should not stain, discolor, or blister painted and unpainted aircraft surfaces.

4.4. Deicing and Antiicing Specifications

The performance criteria for deicing fluids have traditionally been governed by specifications of the Aerospace Division of Society of Automotive Engineers (SAE) (15). These specifications are widely used by most airlines in North America. The SAE specifications incorporate the materials' compatibility requirements set forth by the airframe manufacturers (Douglas CSD#1 and Boeing D6-17487), as well as the performance criteria recommended by the airlines, including minimum flash points, maximum freezing points, and hard water compatibility. A new aerodynamic performance criteria, as defined by the airframe manufacturers, is being added to all deicing and antiicing fluid specifications.

In 1987, a proposal was made to the International Standards Organization (ISO) to prepare international specifications for deicing and antiicing fluids, equipment, and procedures, in an effort to standardize winter ground operations and set minimum requirements for deicing and antiicing at airports throughout the world, and thus aid the pilot in understanding and defining the protection against refreezing as a function of the fluid

used at a given airport. These specifications for worldwide standardization are currently being developed by the ISO.

5. Toxicity and Environmental Issues

The toxicity of antifreeze and deicing fluids is predominantly a function of the main component, the freezing point depressant. For ethylene glycol-based fluids, the toxicity is well-defined, as the toxicity of ethylene glycol has been studied extensively because of its wide usage in varied applications (16).

Ethylene glycol is acutely toxic to humans and animals by oral exposure. For humans, the lethal dose is estimated to be 1.4 mL/kg of body weight, or about 3.2 fl oz (95 mL) for a 150 lb (68 kg) person. For antifreeze applications, oral toxicity is a concern primarily because of the possibility of accidental ingestion by children and pets. Legislation is currently being proposed to study the effectiveness of adding bittering agents or emetic agents in consumer antifreeze, to reduce the likelihood of ingestion of lethal doses (17). For deicing fluid applications, oral ingestion is unlikely. Ethylene glycol is defined as an animal teratogen meaning that at high concentrations in laboratory animals, ethylene glycol has been shown to cause birth defects.

For deicing fluid applications, exposure to vapors and mists is the more likely means of exposure. Ethylene glycol has a threshold limit value (TLV) of 50 ppm for vapors. When proper deicing procedures are followed and proper protective equipment worn, the exposure of deicing personnel to vapor and mist is expected to fall well below this TLV value. This mode of exposure is unlikely for engine cooling applications.

Because of these toxicity concerns, propylene glycol is sometimes used instead of ethylene glycol in these applications. Studies on propylene glycol have shown no evidence of teratogenic effects and it has a lower oral toxicity, with an estimated lethal value of 7.0 mL/kg or 16.2 fl oz (479 mL) for a 150 lb person. No TLV has been defined for propylene glycol, but an AIHA WEEL Guide of 50 ppm has been determined. Propylene glycol has, however, been shown to be slightly more irritating to the skin than ethylene glycol (16).

5.1. Aquatic Toxicity

Aquatic toxicity is different from human toxicity and must be taken into consideration when choosing a chemical that has the potential to enter lakes, streams, or rivers, such as deicing fluids. Unlike human toxicity issues, aquatic toxicity is not strictly a function of the primary component of the fluid, ie, the freeze point depressant. In formulated deicing and antiicing fluids, the additives play a significant role in defining the aquatic toxicity. In particular, many surfactants or wetting agents are highly toxic to aquatic life; whereas both ethylene and propylene glycol are essentially nontoxic: concentrations exceeding 10,000 mg/L show no effect on aquatic life (16). Thus it is important when choosing a deicing or antiicing fluid to obtain aquatic toxicity data on the formulated product and not just the glycol itself.

5.2. Biodegradation and Oxygen Demand

Another important consideration in the use of all chemicals, including antifreezes and deicing fluids, is the rate and extent of biodegradation, or the length of time the chemical persists in the environment. Organic chemicals, such as the glycols, biooxidize to give carbon dioxide and water; however, the rate at which they degrade and the amount of oxygen used in the process determine their effect on the environment. In the case of ethylene glycol-based fluids, laboratory tests indicate complete biooxidation occurs within 20 days. The rate of biooxidation is steady throughout this 20-day period and thus exerts no undue oxygen demand on waterways (causing fish kill) or waste treatment facilities. Propylene glycol, on the other hand, biodegrades rapidly during the first five days of the test (62%), then the biooxidation rate slows down substantially such that only 79% biooxidation is indicated at the end of the 20-day test (16). The rapid initial biooxidation imposes a great

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oxygen demand on the receiving stream or waste treatment facility. Airports using propylene glycol-based deicing fluids must reduce the rate at which they feed the runoff to the waste treatment facility by about half when compared to the rate at which a solely ethylene glycol deicing fluid runoff can be fed to the waste treatment facility.

Human toxicity, aquatic toxicity, and the environmental impact of engine coolants and deicing fluids are typically measured on the fresh fluid only. Spent fluids contain varied contaminants that can drastically affect the toxicity and environmental impact of the fluid. Most pronounced is the impact of heavy-metal contaminants in spent antifreeze. Data on spent and recycled antifreeze, compiled by the ASTM Committee on Engine Coolants, show an average lead level 11 ppm, as well as various other metal contaminants (iron, copper, zinc) (18). The presence of these contaminants in a used fluid may require special disposal techniques for the fluids.

6. Fluid Recycling

When antifreeze becomes unsuitable for use, either because of depletion of inhibitors, presence of corrosion products or corrosive ions, or degradation of the fluid, recycling and reuse of the antifreeze, rather than disposal, may be considered. Although ethylene glycol is readily biodegraded in typical municipal waste treatment facilities, antifreeze disposal becomes problematic because the coolant may contain hazardous quantities of heavy metals picked up from the cooling system. Recycling may be economically preferred over coolant disposal and reduces the concern for environmental impact.

Ideally, a system for recycling spent antifreeze consists first of the removal of the deleterious contaminants such as the corrosion products, corrosive ions, degradation products, and remaining inhibitors. Then the clean fluid could be reinhibited to a known concentration of both inhibitors and glycol.

Two methods of recycling spent antifreeze, filtration or distillation, have been reported (19, 20). The filtration method consists of a filter and a system for reinhibition of the filtered glycol. It may be a portable or skid-mounted recycling machine located in a shop environment. The system filters out particles and other visible contaminants, but does not remove the corrosive ions, dissolved metals, degradation products, or corrosion inhibitors. Typically, supplemental coolant additives consisting of premixed quantities of corrosion inhibitors are added to the filtered glycol. However, because all residual inhibitors are not removed in the filtration process, the addition of premixed inhibitor packages to the filtered fluid may not result in the appropriate balance of inhibitors in the recycled coolant. This is especially critical in silicated fluids, where the excess silicate may result in rapid gelation.

The other recycling method is fractional distillation. This is more complex and capital intensive than a filtering system but produces pure refined glycol which is free of all the deleterious contaminants. The refined material is then reinhibited to exactly the desired coolant formulation. Often this is accomplished through centralized locations where the spent coolant from various sources is collected, combined, then refined and reinhibited. A distillation method is also currently in limited use for the recycling of aircraft deicing fluids. The *Gantry* deicing system, a central deicing car wash-type system, collects the used deicing fluid through a porous pad, then distills the used fluid to remove many of the contaminants. The distilled product is then mixed with virgin deicing fluid before use on the aircraft for deicing.

Because of the problems with some recycling options, the ASTM committee on engine coolants is exploring a specification for recycled coolants. Some automobile manufacturers have found the in-shop recycling equipment to be ill-advised; one service bulletin warns of the deterioration of engine-coolant ethylene glycol to the extent that no additive can restore it to an acceptable state (21).

With the increasing emphasis on recycling in general and environmental awareness in the United States, further growth in the recycling of antifreezes and deicers is anticipated. Emerging technologies may allow

portable shop systems to properly clean spent antifreeze to provide a good base to build an acceptable antifreeze product.

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