

Overview

Engine coolant usage continues to increase on a worldwide basis as the overall vehicle population becomes larger. Many off-highway vehicles and stationary equipment facilities also use engine coolant. Water preservation and environmental concerns are reflected in a gradually expanded use of coolant for industrial cooling applications. Vehicle cooling predominates and is the major concern for the symposium. Average vehicle size and coolant capacity has recently reduced in the United States. More efficient engine designs tend to use less coolant volume for equivalent heat rejection purposes. Modern automobiles are made with newer and lighter weight materials. The importance of aluminum alloy protection by engine coolant has become evident, together with an increased use of composite plastics. Meanwhile, the average age of vehicles on the highway has increased, and these older vehicles require engine coolant replacement at regular intervals. The engine coolant specialist has therefore many technical challenges and the technology is developing sufficiently that a meeting to present advances and discuss current problems was needed.

The first symposium was held in Atlanta, GA, in 1979. It was well supported and resulted in ASTM *Engine Coolant Testing: State of the Art*, (STP 705), which still provides a good reference. Success led to a second conference in 1984, held in Philadelphia, PA, at which the rapid changes in material usage and testing requirements were expounded upon by many of the authors. This symposium resulted in ASTM *Engine Coolant Testing: Second Symposium*, (STP 887), and probably the most important development was the basis of a new standard for evaluating hot surface protection for aluminum engine alloys that has now become an international standard for the coolant industry. Propylene glycol was introduced as an alternate base fluid for coolants. Electrochemical test methods were evaluated and discussions of specific needs for heavy duty engines highlighted.

The third symposium was held in Scottsdale, AZ, *Engine Coolant Testing: Third Symposium*, 6–8 Nov. 1991, and was well attended with presentations from European, Japanese, and United States authors.

Papers presented at the conference covered advances in the development, testing, and application of engine cooling fluids for automobiles and heavy duty engines that have occurred since the last meeting.

A keynote opening address by Hannigan set a good tone to the conference by presenting a brief history of ethylene glycol engine coolant. Ethylene glycol was first suggested for use as an engine coolant in military aircraft in England in 1916. Other aircraft applications followed, with the Curtiss Hawk PIA in 1926 being of particular note. Use of ethylene glycol in automobiles began in 1927. Wide adoption occurred in the period 1949 through 1955 as a factory fill in place of methanol. Developments have continued, and Hannigan presents the highlights bringing us up to the present time.

Four authors presented papers on new families of engine coolant that operate in a medium pH range. Maes and Van Den Ven's work in Europe on the use of low depletion monoacid diacid inhibitor technology reveals good high-temperature corrosion protection of aluminum when acids are properly balanced. An evaluation program included ASTM Test Method for Corrosion of Cast Aluminum Alloys in Engine Coolants Under Heat-Rejecting Conditions (D

4340) hot surface tests, a dynamic heat transfer test, and a coolant aging test. These were used with top quality commercial engine coolants and the monoacid dibasic acid technology, for direct comparisons. Static heat transfer testing gave good results with all technologies. Dynamic heat transfer testing was more discriminating and favored the new monoacid and diacid combinations for aluminum corrosion protection under hot surface conditions with apparently better heat transfer at the metal/coolant interface.

Burns carried out an extensive fleet test with a carboxylic acid long life coolant formulation with very good results. Two hundred and three Ford Crown Victoria Taxi cabs were used. The chief objective of the program was to evaluate coolant pump failure with respect to the new carboxylic long life coolant, when compared to more traditional formulations. Coolant installation was color coded and pump failures from each group identified. Four conditions and a factory fill were involved. The new carboxylic formulation resulted in the lowest pump failure rate, although reasons why could only be speculated upon. A new coolant pump bench test is recommended for comparative study of coolant formulations.

Mercer examined experimental carboxylic acid inhibitor formulations, to test their efficiency in both laboratory and fleet tests. Problems were encountered with lead based solder and aluminum alloy corrosion, although other metals were adequately protected. Aluminum protection required high levels of acids present. Compatibility of the carboxylic acid formulations and phosphate buffered coolant was poor, and mixtures resulted in reduced protection. No inhibitor depletion was observed, but this did not prevent corrosion of the aluminum and high lead solder alloys in fleet testing.

Extended life coolant with sebacic acid was compared to current high silicate alkaline phosphate coolant prevalent in the United States in a three-year municipal fleet test by Adamowicz and Falla. Results demonstrated no particular advantage with the sebacic acid formulation over current North American coolant. They also concluded that factory fill coolant life can be extended far beyond previous expectations. Metal coupon corrosion losses were minimal for either coolant throughout the test. The relatively high cost of the sebacic acid coolant precludes its use on an economic basis.

Durability of aluminum alloy automotive radiators in service depends on the alloy selected and the expected engine coolant environment. Beal and El-Bourini investigated accelerated testing procedures for alloy development with appropriate coolant conditions. New alloys are continually under development to improve radiator service life. The challenge is to find testing methods that correlate with service experience without resorting to long-term vehicle exposure trials. A combination of electrochemical studies and simulated service has demonstrated a viable approach. Unless related to a particular engine coolant environment, serious mistakes can be made in aluminum alloy radiator materials chosen.

Heavy duty diesel engines use significant quantities of coolant and emphasis on long operation periods continues as engine design changes, resulting in higher efficiencies. Hercamp presented an historical overview of cavitation corrosion in diesel cylinder liners, relating various factors that are involved with liner pitting. A major problem in the 1950s, much work has been done since to identify causes and develop solutions. The paper covers scientific background and theories, and not all workers agree on the damage mechanism. Coolant effects and the use of supplement coolant additives (SCAs) are covered with some reference to engine related factors. Education of maintenance personnel is important to follow prescribed procedures in coolant and SCA practice. Testing can assist in avoiding trouble and is cost effective in checking coolant condition.

Hudgens briefly covered the history of supplemental coolant additives used in heavy duty diesel engines, and then went on to describe a new family of phosphate molybdate packages that are designed to perform better with aluminum components and cause less problems if

overtreatment occurs. A test scheme involved most ASTM standards and the German FVV Test, in addition to bench cavitation work. The phosphate molybdate formula provides better protection in hard water, and the ability to reduce nitrite levels is beneficial to solder protection. Liner pitting is prevented at lower overall inhibitor addition.

Toxicity and disposal of engine coolants is a topical subject that was reviewed and discussed by Hudgens and Bustamante. Properties of ethylene and propylene glycols and major additives used in engine coolants are included. Propylene glycol is not toxic and provides an environmentally acceptable coolant base. However, inhibitors used have varying degrees of toxicity, and after use, when heavy metals are dissolved into the coolant, the resultant fluid is definitely toxic, whether propylene glycol or ethylene glycol are used as the base fluid. Present laws and regulations are referenced, and a discussion on the hazardous concept is included. Used coolant may or may not be hazardous depending on its condition when tested against EPA threshold values. Both ethylene and propylene glycols are biodegradable. 400 million gal (1514 million L) of coolant are sold every year. 10% of coolant may be lost by leakage, 25% or more by improper disposal, and the remainder generally handled according to regulations. Recycling is becoming a commercial feasibility and is being done in the western United States in particular on a large scale. Total volume of recycled coolant is still low compared to coolant sold per year. The paper gives a good overview of facts and concerns regarding handling and disposal of engine coolants.

Test strips have been developed for rapid on-site analysis of engine coolants for some specific attributes. Hemmes et al. described their efforts. Strips for nitrite and molybdate measure protection for cavitation erosion. One has been developed for MBT in conjunction with chloride level identification. Test strips for pH and RA also are available. Measurement of freeze point has been carried out for over ten years. The wider range of test strips assist in maintenance programs and for identifying when SCAs should be used in heavy duty vehicles.

Engine coolant analysis techniques use standard equipment with particular procedures for accurate results. Advances coincide with new analytical instrumentation. Problems in coolant systems can be solved by application of inductively coupled plasma (ICP) emission spectroscopy and laser ablation ICP according to Zamechek and McKenzie. Coolant analysis by ICP is enhanced by specially developed software for interferences and data reduction. Aqueous standards are used with 50-fold dilution of the analytical samples. Preparation methods are described. The laser ablation system was used for spacial mapping of elements on the surfaces of water pump seals. A uniquely adapted sample cell and target area was devised with optical focusing and alignment.

Oxalic acid cleaning of engines removes inhibitors, rusts, and other deposits. Some concern has been expressed on the post cleaning effect of the process when vehicles are used for coolant testing. Woyciesjes reviewed the chemistry involved. Oxalates form a variety of complexes with typical metals in the engine circuit. Ferrous oxalate can be 10 μm or more in thickness. Borate conditioning removes some of the oxalate. Oxalates can affect subsequent coolant properties by having a detrimental influence on pH, RA, and inhibitor levels. A high pH, borate conditioning fluid minimizes the consequences, and in new vehicles the effect is small. Vehicles exhibiting heavy corrosion should not be used for coolant testing, because cleaning will not be totally effective.

Pump seal failures are a contemporary problem with disagreements on causes and solutions. This topic was received with much interest by the attendants. Deposits on water pump seal faces were examined by Stafford from heavy duty diesel on-highway engines. Coolant leakages were traced to deposit films built up on the siliconized graphite seals. Surface analyses of the buildup revealed elements from the coolant, coolant additives, corrosion metals from the engines and calcium from hard water. Mileage at pump removal ranged from 28 000 to

199 000 miles (45 060 to 320 251 km). A calcium-iron-phosphate complex precipitated during nucleate boiling episodes was determined as the cause of seal leakage because of seal face separation caused by the deposit.

Kiryu et al. examined the effect of coolant on water pump mechanical seals in a very thorough investigation. There is an urgent problem attributed to coolant formulation contamination and an increase in engine operation condition severity. Leakage occurs by deposit formation and growth of the film, which creates a gap at the seal face. Oxygen-rich conditions at 150°C can cause inhibitor solidification that deposits on the seal. Test work confirmed that high-temperature seal operation causes deposits related to silicates, when they are present. In triethanolamine coolant, copper and iron salts were the culprit, usually from breakdown acids promoting corrosion of copper parts. A third coolant formulation resulted in precipitation of dibenzothiazyl disulfide on seal ring surfaces leading to leakage. All the deposition problems were solved by designing seals with lower interface operating temperature, controlling materials used, and reducing surface roughness at the seal face.

Depletion of engine coolant inhibitors, contamination, and breakdown of the glycols occurs during the use of engine coolant in service. Vehicle makers provide recommendations on changing coolant on a regular basis. These changes provide the waste stream that can be used for recycling. Statistical analysis of used coolants gathered from New England through Georgia was performed by Woodward and Gershun. A total of 2500 vehicles was reviewed in the results. Standard laboratory techniques were used for the analyses with appropriate conditions for accuracy of data collected. A wide range with nonnormal distribution was found for residual inhibitors. Corrosive contaminants, such as chloride and sulfate, varied widely with chloride levels similar to ASTM corrosive water and sulfates significantly higher. Degradation of the glycol to acetates, glycolates, and formates depletes the reserve alkalinity. A prediction is made that 20% of used engine coolant will have lead in excess of 5 ppm, and thus be regarded as hazardous waste. Suspended solids are found regularly with over 25% of those coolants tested having 500 ppm or more. Recycling needs careful consideration because of variations in fluid conditions and the need for a balanced product.

Extension of coolant life in automobiles is feasible when a three-step examination is made that determines coolant has not been used for 65 000 km, is not oily, murky, or rusty, or is less than 25% concentrated with a reserve alkalinity of less than 3. Under these conditions Hercamp and Remiasz show that a supplemental coolant additive package can provide at least a further year of life to the coolant. Standard ASTM tests were used for verification, and field experience has been satisfactory. The additive is used in conjunction with a closed-loop coolant flushing system attached to the vehicle.

Richardson described a recycling process for used coolant that involves a multistage process with dual bed deionization. The process purifies the coolant removing contaminants and particulates. The resulting fluid has very low concentrations of all species providing a clean fluid for reinhibition. The process used and data obtained are described. The author considers that efficient removal of contaminants to a low total dissolved solids level is necessary for a consistent finished product.

Recycling processes were discussed by Bradley. The paper reviewed several different approaches to providing the service. Awareness of environmental issues in the disposition of spent engine coolant prompted a study to examine the efficiency of various systems. A reference coolant was utilized that was collected from many vehicles, resulting in a mixture of several types of inhibitor packages and degradation products. All recycling processes used the same coolant for test purposes. Processes evaluated were filtration, filtration-flocculation-coagulation, deionization, reverse osmosis and vacuum distillation. Some systems were combinations of these processes. These systems are described. Off site coolant recycling is performed on a large scale typically by fractional distillation, and these companies are included

in the G.M. approval program. Recycled coolants must meet or exceed GM 1825 M coolant specification. A progressive test program was undertaken. Coolants failing any test in the sequence were rejected. Physical tests, followed by ASTM Test Method for Corrosion Test for Engine Coolants in Glassware (D 1384) and ASTM D 4340 hot surface evaluation were performed. Only those passing proceeded to pump cavitation and simulated service. Results were not available at the conference and will be published later.

Two keynote papers were invited covering automotive and heavy duty vehicles technologies. The objective was to educate the newcomers and remind the veterans of coolant technology development over the years to the present time. Both papers were timely and a success at the symposium. Hannigan covered automotive cooling technology in which he has been personally involved over many years making a good presentation of history and finishing with highlights of present challenges. A summary of current heavy duty technology in coolants was ably addressed by Kelley with discussion on liner pitting, silicate drop-out, water pump seal leakage, and other problems. He discussed the value of ASTM standards and new requirements for the future with a good overview.

The symposium was a success and reflected advances in coolant technology and present areas of concern. A special thanks to all the authors, the symposium subcommittee, chairmen of the individual sessions, and the ASTM staff is warmly given. Jenny Beal, Denise Steiger, and Gloria Collins deserve specific mention for the organization of the conference and social events. This volume will make a valuable contribution to publicly available information on coolant technology.

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