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BACKGROUND

In 1967, because of the success of silicone fluids in other hydraulic systems, producers of silicones became interested in developing silicone based fluids for use in vehicle brake systems. It was recognized that silicone fluids possessed physical and chemical properties that were desirable for this application. Some of these were low-temperature viscosity, high-boiling point, low-water sensitivity, and good chemical stability. Preliminary laboratory studies showed that the first silicone experimental brake fluids performed well except in the areas of lubricity and rubber compatibility. Each of the three major silicone fluid manufacturers centered their brake fluid research around the development of additives to improve these two properties. Subsequent fluids were developed by each of the three manufacturers which performed satisfactorily in laboratory tests. The US Army set up a series of field tests in vehicles operating

in (1) the tropical rain forest (Panama) (2) the desert (Yuma Proving Ground, Arizona) and (3) the Arctic (Fort Greeley, Alaska). Results reported in 1975 (reference 1) and 1976 (reference 2) indicated that the silicone brake fluids were far superior to the conventional polyglycol brake fluids in the areas of low hygroscopicity and corrosion protection. Lubricity and rubber compatibility were equal to or superior to that found with conventional brake fluids. The Society of Automotive Engineers (SAE) publications (references 3 and 4) reflect all phases of early army research on silicone brake fluids.

As a result of these studies a specification, MIL-B-46176, Brake Fluid, Silicone, Automotive, All Weather, Operational and Preservative, was published (reference 5). In late 1980 the army opted to switch from conventional brake fluids to silicone brake fluids. A technical consideration concerning the cost effectiveness of retrofit procedures was resolved. Successful resolution of this

ABSTRACT

An investigation was conducted to determine the compatibility of recently specified silicone brake fluids with elastomers expected to be found in vehicles submitted to the US Army for test and evaluation to compare their performance with conventional fluids. More than 1500 immersion tests were conducted at temperatures ranging from 0°F to 248°F (-18 to +120°C) with 14 different elastomers and 5 different brake fluids. It was found that

the silicone brake fluids performed as well as/or better than the conventional fluid in all tests involving vehicle brake system elastomers. Brake performance of systems in which silicone brake fluids and conventional brake fluid become mixed will operate normally with no fluid/elastomer related problems. Contaminants such as petroleum based fluids will cause undesirable attacks on brake system elastomers regardless of the fluid in the system.

question allows fluid meeting MIL-B-46176 to displace the three existing military fluids (references 6, 7, and 8) from the inventory.

The SAE, automotive manufacturers, military vehicle developers, and test engineers have been concerned about the compatibility of the silicone brake fluid with the elastomers which are found in various vehicle systems. In order to resolve these concerns and to corroborate the validity of the basic laboratory and field test data, more discriminating tests in this area were deemed advisable. An in-depth study of fluid-elastomer behavior was needed, which would point out shortcomings such as excessive and uneven swelling, possible leaching of elastomer ingredients, and minor degradation of specific types of elastomers under unusual or unforeseen operational situations.

Findings from this study could be applied to the analysis of failures occurring in future vehicle tests, and aid brake system design engineers and brake fluid researchers in their evaluation of the performance of the new silicone brake fluids.

MATERIALS TESTED

The compatibility of representative silicone brake fluids with the new elastomeric materials used in automotive vehicles was determined through a series of immersion tests.

The five brake fluids listed in Table 1 were evaluated.

Table 1. Fluids

Fluids	
Code A	Silicone
Code B	Silicone
Code C	Silicone
SAE RM 70	Silicone
SAE RM 66-03	Conventional polyglycol

Fourteen elastomers were used in the study, representing all elastomers found in current automotive systems. The elastomers are listed in Table 2.

IMMERSION TESTS

Twelve series of immersion tests were conducted. One-inch slabs of each of the elastomers were washed with isopropyl alcohol, weighed in air and water, hardness

determined using a Shore "D" durometer, and immersed in each of the fluids (in duplicate) under the following conditions.

TEST NO. 1. This test was conducted at ambient conditions. Test jars were stored on the laboratory shelf. Rubber specimens were removed after 1 week, 3 weeks, 2 months, 6 months, and 12 months; the specimens were wiped with a clean lint-free cloth, weighed in air and water to determine change in volume, and the hardness was measured. After each storage period the specimens were examined for evidence of disintegration and then placed back in the test jar.

Table 2. Elastomers

Type	Use	Shore A Durometer Hardness
SBR (styrene butadiene rubber)	Wheel cylinder cups	50
SBR	Master cylinder seals	70
SBR (SAE)	Disc brake seals	70
EPDM (ethylene propylene rubber)	Disc brake seals	70
EPDM	Brake valve parts and seals	80
EPDM (SAE, RM 69)	Referee test slabs	70
VITON	O-rings	70
Silicone rubber	Seals and O-rings	60
N.R. (natural rubber based on SAE-ISO-1)	Referee test slab	60
BUNA-N (33% ACN) (nitrile rubber)	Automotive parts	60
BUNA-N (21% ACN)	Automotive parts	70
BUNA-N (41% ACN)	Automotive parts	70
Neoprene (SAE, RM 68)	Brake hose	70-80
Chlorobutyl	Master cylinder diaphragms	

TEST NO. 2. This test was conducted at 70°C (158°F). Rubber specimens were removed after 3 days and 7 days. Test jars were removed from the oven and allowed to cool for 30 minutes. The rubber specimens were then removed, wiped with a clean cloth, weighed in air and water to determine volume, and the hardness was determined; the specimens were examined for disintegration, and after the three day inspection placed back in the test jar; jars were placed back in the oven; after the 7 day inspection the fluids were visually examined for excessive sediment buildup.

TEST NO. 3. This test was identical to Test No. 2 except that the test temperature was 120° C (248°F). Rubber specimens were examined after 3 days and 7 days.

TEST NO. 4. In this test each of the silicone fluids was mixed with an equal quantity of the conventional fluid and placed in the test jars. The two fluids were not miscible so they separated. The volume and hardness of two rubber test specimens were determined. One specimen was placed in the lower fluid layer (conventional fluid), and one specimen was suspended horizontally in

the top fluid layer (silicone). The jar was stored on the laboratory shelf at ambient temperature. The volume and hardness of each of the two rubber test specimens were measured and examined after 2 weeks, 8 weeks, 6 months, and one year.

TEST NO. 5. This test was conducted at -18°C (0°F). Test jars were removed from the cold chamber after 2 weeks, 8 weeks, 6 months, and 1 year. The volume and hardness of rubber specimens was measured within 10 minutes after the jars were taken from the chamber, after which the specimens were examined for evidence of disintegration and then placed back in the test jars. Exposure to cold temperature continued.

TESTS NO. 6, 6A, 7, 7A, and 8. These tests were run on the silicone compatibility fluid and the conventional fluid in order to determine the effect of some common automotive contaminants on the performance of the rubber. In tests No. 6 and 6A, 1% and 5% respectively, by volume, of petroleum oil conforming to grade 10, MIL-L-2104 (reference 9) was added to each of the jars. In tests 7 and 7A, 1% and 5% respectively, of synthetic lubricant meeting MIL-L-46167 (reference 10) was added to each of the jars. In test 8 10% of hydraulic fluid meeting MIL-H-6083 was added to each of the jars (reference 11). Each of these tests was stored at ambient temperature and examined after 1 week, 3 weeks, 7 weeks, and 6 months of storage.

TEST NO. 9. In this test, conventional fluid was mixed with the silicone compatibility fluid to produce conventional fluid concentrations of 5%, 10%, 20%, and 30% by volume. Rubber specimens were immersed as described in test No. 4. Four weeks, eight weeks, and 6 months examination were made.

TEST NO. 10. This test was run on neoprene rubber at 100°C (212°F) in order to correlate with the test temperature prescribed for neoprene in silicone brake fluid specifications. Specimens were examined after 3 days and 7 days exposure.

TESTS NO. 11 and 11A. These tests were conducted with each of the fluids contaminated with 3.5% water. Test 11 was run at 70°C (158°F). Test 11A was run at 120°C (248°F). In each of these tests, specimens of neoprene, SBR (70 duro), EPDM (SAE RM 69) and natural rubber were suspended in the fluid/water mixture and inspected after 3 days and 7 days exposure.

TEST NO. 12. In this test, rubber specimens were soaked in conventional fluid for 3 days at 70°C (158°F). After 3 days the test slabs were removed from the fluid, rinsed in isopropyl alcohol and wiped with a clean, lint-free cloth. Hardness and volume measurements were taken. The slabs were then immersed in the silicone fluids

for 7 days at 70°C (158°F), removed, wiped with a clean lint-free cloth, and change in hardness and volume was measured.

COMPARISON CRITERIA. The criteria listed in Table 3 were established in reference (5) to check performance of silicone brake fluids on some elastomers found in vehicle brake systems. These criteria were used as a basis for comparing the performance of the fluid/elastomer combinations in these tests with known satisfactory performance levels.

Table 3. Criteria for Rubber Performance (Reference 4)

Type of Rubber	Volume Swell (Percent)	Immersion Tests		
		Changes in Hardness (Durometer Points)	Test $^{\circ}\text{C}$	Test $^{\circ}\text{F}$
SBR	+5 to +20	0 to -10	70 ± 2	158 ± 3
	+5 to +20	0 to -15	120 ± 2	248 ± 3
Neoprene	-3 to +6	+3 to -10	70 ± 2	158 ± 3
	-3 to +10	+3 to -10	100 ± 2	212 ± 3
EP	0 to +16	0 to -10	70 ± 2	158 ± 3
Natural	+5 to +20	0 to -10	70 ± 2	158 ± 3

RESULTS AND ANALYSIS

EFFECT ON SBR.

(1) Results. Swelling and softening exhibited by all proprietary silicone fluids on SBR fell in the middle range of reference criteria in all tests. Swelling values for the silicone compatibility fluid were borderline high at 0°F and 248°F (-18 and $+120^{\circ}\text{C}$). Swelling values received with the conventional fluid were low and in some instances at ambient temperature, slight shrinkage occurred.

(2) Analysis. SBR is the most widely used elastomer in drum and shoe brake systems, so the silicone fluid manufacturers adjust the effect-on-rubber properties of the fluids so that the effect on SBR falls in the middle range of reference criteria. The borderline high values received with the silicone compatibility fluid would not cause brake failure. The low swelling values received with the conventional fluid would indicate poor performance because of potential leakage of brake fluid past the cups. In actual vehicle operation no widespread problem has been reported.

EFFECT ON NEOPRENE RUBBER.

(1) Results. Results of tests on neoprene rubber showed that the proprietary silicone fluids gave no excessive shrinkage or swelling regardless of the test temperature. The silicone compatibility fluid gave high swelling values at ambient temperature and 248°F (120°C) after extended exposure. The conventional fluid also gave high values at 248°F . The presence of water also caused high swelling in one of the silicone fluids and the conventional fluid.

(2) Analysis. Neoprene rubber is used in brake hoses so the swelling/softening values are not as critical as those for rubber found in components which move during braking applications. Specifications and reference criteria allow slight shrinkage and moderate swelling. The high values recorded in this series of tests for the silicone compatibility fluid and the conventional fluid at 248°F is beyond the normal test temperatures and exposure temperatures of neoprene rubber.

EFFECT ON EP RUBBER.

(1) Results. All silicone fluids performed satisfactorily on EP rubber at all test temperatures. Swelling of the EP rubber with conventional fluid was low; slight shrinkage occurred at 0°F (-18°C)

(2) Analysis. EP rubber polymers are used in disc brake seals, brake valve parts and in some master cylinder applications. The results received in this test with the silicone fluids were excellent and would indicate that no problems would be expected in the use of silicone brake fluids with EP rubber. The amount of shrinkage found with the conventional fluid at low temperatures would not be expected to cause poor performance. Recent research has been directed toward improving the cold-temperature properties of EP rubber

EFFECT ON NATURAL RUBBER.

(1) Results. Results of all tests on natural rubber/silicone fluid combinations paralleled results found in the SBR tests. Swelling and softening values for natural rubber fell within the middle range of reference criteria with all proprietary silicones. The values for the silicone compatibility fluid were borderline high at 248°F. The results of the conventional fluid/natural rubber tests were satisfactory at all temperatures.

(2) Analysis. Natural rubber is used in the brake system of some foreign vehicles, but domestic use has diminished over the last decade. The switch to SBR was made because of better availability and increased high-temperature properties. No elastomer related problems would be expected in systems using natural rubber and silicone fluids.

EFFECT ON BUTYL RUBBER.

(1) Results. Swelling and shrinkage of butyl rubber was very low in all tests conducted in this program. There was little effect on the elastomer by either the silicone fluids or the conventional fluid.

(2) Analysis. Butyl rubber is used in master cylinder diaphragms and is subjected to static situations only. The results received in this program indicate that there would be no operational difficulties in the use of butyl rubber in the desired application.

EFFECT ON NITRILE RUBBER.

(1) Results. In this investigation, except in isolated instances, the silicone fluids were compatible with the three nitrile rubber formulae. The conventional brake fluid is not compatible with nitrile rubber and caused excessive swelling, softening and some rubber disintegration in most tests, especially those tests conducted at high temperatures.

(2) Analysis. Nitrile rubber (Buna N) is compatible with petroleum based fluids and is used extensively in O rings in systems, such as control systems and weapons recoil systems, which use petroleum base hydraulic fluids. It is also used in various automotive applications such as shock absorbers and fuel systems, but is not used in conventional braking systems; it is incompatible with conventional brake fluids. For the purpose of this study the observed compatibility of silicone brake fluids with nitrile rubber is coincidental, but the data derived in the study would be of interest to engineers in the weapons recoil systems field or other fields which at the present time use petroleum base, synthetic base, or other inflammable hydraulic fluids in the systems. Possible advances could be made in the use of silicone fluids in these applications.

MIXED FLUIDS.

(1) Results. In tests involving mixtures of silicone brake fluids and conventional brake fluid (5%, 10%, 20%, 30%, and 50% conventional fluid) many instances were noted where different amounts of swelling of the rubber test slabs occurred in the two fluids in the same test jar. The swelling which was found was of different magnitude in each layer of fluid and did not match the swelling which occurred when that fluid was tested alone. The differences in swelling were relatively small and showed up gradually over a long period of time. In some cases slight shrinkage occurred in one layer and not in the other layer.

(2) Analysis. The silicone and conventional brake fluids are not miscible. Each fluid contains additives which are placed in the fluid to adjust the rubber swelling and softening to the desired range. When the fluids are mixed, this series of tests showed that one of the fluids extracted the rubber swelling additives from the other fluid and in many instances gave results which showed that the additive had migrated. If the fluids were mixed in a brake system and uneven swelling of the elastomer occurred, the distortion of the brake cup would be gradual and would not contribute to catastrophic brake failure. In cases where shrinkage of the elastomer occurred, fluid leakage would show up in actual operation.

EFFECT OF CONTAMINANTS.

(1) Results. The tests in which engine oil and hydraulic fluids were added to the silicone brake fluids as contaminants showed increased swelling of EP, SBR, NR, and silicone rubber; neoprene and Viton shrunk; the nitriles and butyl rubber remained approximately the same. In the contaminated conventional fluid EP, SBR, NR, and butyl rubber showed increased swelling; neoprene, nitrile, silicone and Viton remained unchanged. In tests in which the shock absorber fluid was used as the contaminant most of the results were similar to the tests where engine oil or hydraulic fluid were added. Some variations occurred; the neoprene rubber swelled more in both the conventional and silicone brake fluids; the silicone rubber swelled slightly more in the conventional fluid; the butyl rubber shrunk slightly in the silicone fluid.

(2) Analysis. Contaminants were chosen which are most apt to be inadvertently added to the brake system of vehicles. The engine oil and hydraulic fluid are petroleum base fluids and are known to be incompatible with EP, SBR, and natural rubber. The shock absorber fluid used in this test is a synthetic diester fluid. Its effect on elastomers is very similar to the petroleum base fluids. Experience has shown that nearly every conceivable type of contaminant can find its way into an automotive brake system. Some of these contaminants can cause catastrophic failures which are entirely independent of the type brake fluid used. Early studies showed that water contamination affects the rubber swelling properties of conventional brake fluids. This study showed that water sometimes affects the rubber swelling properties of silicone fluids, but not to the extent found with conventional fluid. Also, water contamination of silicone fluids is less likely to occur due to their hydrophobic properties. A general analysis of the results of brake fluid contamination is not possible. Past efforts in the training of maintenance personnel in proper handling of brake fluids should continue to be emphasized.

EFFECT ON VITON RUBBER.

(1) Results. In this study Viton was compatible with proprietary silicone fluids, codes A and B, but was not compatible with silicone fluid, code C, and the silicone compatibility fluid. The conventional fluid caused the Viton to swell and soften excessively and was not compatible.

(2) Analysis. Viton rubber is used in O rings, valves, and diaphragms in fuel systems and does not come into contact with brake fluids. The fact that the Viton is compatible with some of the silicone fluids is worthy of note, and this information may

be useful to design engineers in future developmental work in the automotive field.

EFFECT ON SILICONE RUBBER.

(1) Results. The silicone rubber was not compatible with the silicone brake fluid. Excessive swelling, softening, and disintegration occurred. The conventional brake fluid is compatible with silicone rubber; only slight swelling or shrinkage occurred in all tests involving conventional fluids.

(2) Analysis. Silicone rubber is used in O rings and in some hoses found in the automotive systems, such as radiator hoses. Since it is chemically similar to the silicone brake fluids, the "solution effect" renders the fluid and rubber incompatible. The conventional polyglycol fluid has no adverse effect on the silicone rubber, and the rubber can be used in many applications where it is exposed to polar fluids.

EFFECT OF PRESOAKING IN CONVENTIONAL BRAKE FLUIDS.

(1) Results. In Test No. 12, in which the rubber test specimens were first exposed to conventional brake fluids, then exposed to silicone, no degradation, excessive swelling, or excessive softening of the brake system elastomers was found.

(2) Analysis. In the recommended change-over from conventional brake fluids to silicone brake fluids, a question was raised of the effect that pre-exposure of elastomers to conventional fluids would have on silicone fluid compatibility. Test 12 was devised to simulate this situation. The initial exposure of the elastomers to the conventional fluid for three days at 70°C is based on standard accelerated test conditions simulating extended field exposure. These conditions are found in brake fluid specifications (reference 6) and are based on correlating laboratory and field test observations. In this study it was found that the presoaking in conventional fluids would not affect performance in silicone fluids. In the replacement of conventional fluids with silicone fluids, rubber parts already in the system would not need to be replaced.

SUMMARY OF INVESTIGATION

More than 1500 comparative immersion tests were conducted at temperatures ranging from 0°F to 248°F (-18 to +120°C) with 14 different elastomers and 5 different brake fluids. The silicone brake fluid performed as well as or better than the conventional fluid in all tests involving vehicle brake systems elastomers. No discrepancies were revealed in extended exposure periods.

The replacement of conventional brake fluids in automotive vehicles with silicone brake fluids will not adversely affect the

brake performance from the standpoint of fluid/elastomer compatibility.

Mixtures of silicone fluids and conventional fluids could lead to conditions which would affect brake performance due to differences in rubber swelling characteristics of the fluids. The differences in swelling characteristics showed up gradually, were not extreme, and would not cause catastrophic brake failure.

Depending upon the contaminant, inadvertent addition of engine oils, hydraulic fluids, shock absorber fluids, etc., will cause undesirable attack on brake systems elastomers, which will lead to brake failures.

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