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PETROLEUM HYDRAULIC FLUIDS

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Pitman-Dunn Research Laboratories
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February 1967

Inorganic Salts in Mahogany Sulfonates and Their Effect on Petroleum Hydraulic Fluids

A series of mahogany sulfonate rust inhibitors used in compounding qualified petroleum hydraulic fluids was studied in connection with the inoperability of tank hydraulic systems after short storage periods of six to nine weeks. Examination of the disassembled hydraulic components showed severe staining and rusting in valving areas having extremely critical tolerances.

Analyses of the sulfonates indicated the presence of inorganic water soluble salts (calcd. as calcium chloride and calcium sulfate) ranging from 0.02 to 0.19 per cent. It was found that the hydraulic fluids used in the tanks which did not perform satisfactorily contained sulfonates having a relatively high chloride salt content of 0.12 per cent, while the use of hydraulic fluids with sulfonates containing less than 0.03 per cent of the halide salt permitted satisfactory operation. Data are also presented which show that the concentrations of inorganic salts found in the commercial mahogany sulfonates studied do not appear to significantly affect the rust prevention properties of the sulfonates but their presence does promote corrosion due to galvanic action.

A correlation was established between laboratory galvanic corrosion tests and performance tests in tank and mock-up systems.

INTRODUCTION

The use of corrosion preventive additives for lubricating oils has been practiced for many years (1,2) and the literature reveals many types of compounds which are effective corrosion inhibitors (3,4,5). These additives, however, are generally associated with automotive crankcase lubricants, with little emphasis given to corrosion inhibitors for use in lubricating oils for military hydraulic and instrument systems. This has been notably apparent to the military since it has been observed that many instruments and hydraulic units of fire control systems would not function due to severe deficiencies in rust protection of the lubricants used (6,7,8). Thus, a great deal of work was begun on the study and development of rust inhibi-

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tors. Among the materials studied and found to be effective were the high molecular weight petroleum sulfonates, naphthenates, long chain fatty acids, and amines (6,9,10, 11,12,13). Of particular interest were the mahogany sulfonates because of their effectiveness, relatively low cost and availability as by-products of white mineral oils. The mahogany sulfonates are now extensively used by the military in petroleum and synthetic lubricating oils to provide rust protection for fuzes (MIL-L-11734), instruments (MIL-L-60S5), hydraulic systems (MIL-H-13919, MIL-H-60S3), aircraft (MIL-L-7870), and weapons (MIL-L-14107, MIL-L-644).

Recently, however, the use of one of the rust-inhibited petroleum-base hydraulic fluids meeting Specification MIL-H-60S3B, "Hydraulic Fluid, Petroleum Base, Preservative," currently on the list of qualified products for military hydraulic systems, was found to cause inoperabil-

ity of M48A2 tanks (11). (MIL-H-60S3B hydraulic fluid is used by the Army as both a preservative and an operational fluid.) During manufacture of the tanks, two qualified MIL-H-60S3B fluids, A and B, were used in the hydraulic system for storage purposes. After short periods (six to nine weeks), it was noted that a large number of the units would not function. It was found that all of the tanks with fluid A in the hydraulic system were inoperable, while all of those using fluid B performed satisfactorily. Disassembly and examination of the hydraulic systems lubricated with fluid A revealed extensive staining and corrosion on critical areas of those components which control movement of gun and turret. A schematic diagram of the hydraulic systems of the M48A2 tank is illustrated in Fig. 1. Figures 2 and 3 are photographs of some of the rusted components of the hydraulic system lubricated with the MIL-H-60S3B fluid which caused inoperability of the tanks after a storage period of only six weeks. Since mahogany sulfonates are used as rust inhibitors in formulating MIL-H-60S3B fluids, it appeared reasonable to consider such compounds as possible suspects in permitting the rusting and staining of the components which caused inoperability of the tanks.

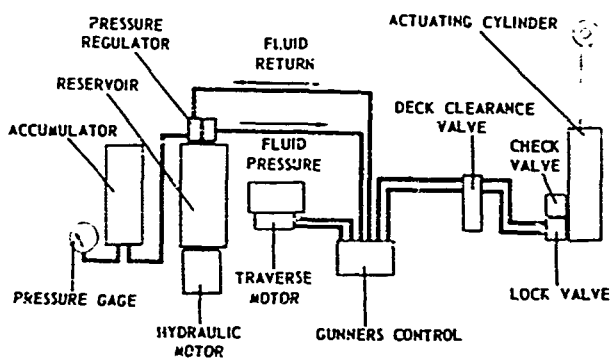


Fig. 1 — M48A2 Tank Hydraulic System

The work reported here describes studies on MIL-H-60S3B hydraulic fluids formulated with commercial mahogany sulfonates, and data are presented to show that the water soluble inorganic salts found as impurities in the rust inhibitors are a primary cause of the rusting and staining of the hydraulic components. Since dissimilar metals (steel, iron, copper and aluminum) are present in the hydraulic system, the inoperability of the tank hydraulic system is considered to be associated with the phenomenon of galvanic corrosion.

EXPERIMENTAL AND TEST RESULTS

Preliminary studies in determining the cause of the rusting and staining of the components were directed toward evaluating the rust preventive properties of the qualified MIL-H-60S3B hydraulic fluids. All of the lubricants (including the MIL-H-60S3B fluid which permitted the stain-

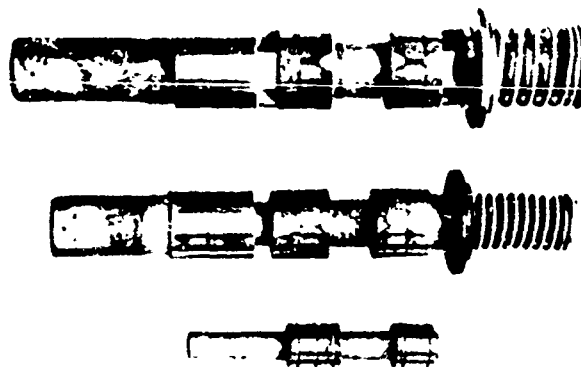


Fig. 2 — Hydraulic Components M48A2 Tank, Elevation and Traverse Spools

ing and rusting of the components) were found to pass the 100-hour rust protection requirement, using the JAN-H-792 humidity cabinet. To further determine the ability of fluid A or B to provide protection against rust of the hydraulic components, an immersion storage test was conducted using the lock valves which have extremely close tolerances (0.00016 in.). Four valves were placed in four separate beakers, two were covered with fluid A and two with fluid B. At the end of nine weeks' storage, an examination of each valve indicated no evidence of stain or rust. These results confirm the previous exposure tests in the humidity cabinet, indicating that fluids A and B provide the hydraulic steel components with adequate protection against rust.

Attention was then directed toward evaluating the qualified fluids for compliance with the other requirements of the specification. It was found that all of the hydraulic fluids were essentially within specification limits. These initial studies indicated that perhaps the present requirements covering MIL-H-60S3B fluids are not entirely satisfactory and that the inoperability of the tank systems may be due to some deficiency not covered by present requirements.

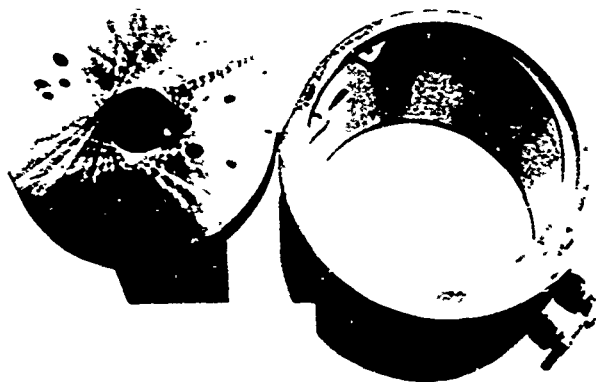


Fig. 3 — Hydraulic Components M48A2 Tank, Superelevation Actuator Assembly

A somewhat similar corrosion problem with the use of instrument-lubricating oil meeting Specification MIL-L-6085 "Lubricating Oil, Instrument, Aircraft, Low Volatility," had been observed in the 1950's when it was noted that considerable corrosion of steel balls and races in brass-retainer ball bearings took place during storage. This problem was studied (15), and the corrosion of the ball bearings was attributed to galvanic action. A test device, consisting of an SAE 52100 steel disc (¼ inch thick x 1 inch in diameter) and a Muntz-metal (alloy of 60% copper + 40% zinc) specimen (½ inch wide, 1½ inches long, and 1/64 inch thick), was developed and utilized in the laboratory to measure the ability of lubricating oils to promote galvanic corrosion. (This method has been adopted by the military and is now described as Federal Standard Test Method 5322T.) Since the tank hydraulic system contains dissimilar metals, this test device was used in evaluating the galvanic corrosion property of the qualified MIL-H-6083B hydraulic fluids (A to F). The results are given in Table 1.

Since the hydraulic fluids (A to F) contained no organic sulfonate rust inhibitors, it appeared probable that the results given in Table 1 (fluid A fails, fluids B to F pass) may be associated with differences in the sulfonates used. Accordingly, analyses of several of the commercially available sulfonates were determined. The test method used was ASTM D1216-56, modified to include quantitative methods for the inorganic salts content. The chlorides were determined as follows: a 50 g ± 0.50 g sample of the sulfonate was placed in a 250 ml beaker. Fifty ml of ethyl ether were added, with stirring, until the sample dissolved. The solution was transferred to a 250 ml separatory funnel using an addition 50 ml of ethyl ether. Twenty-five ml of HNO₃ (1 acid:9 water) was added, with shaking. The mixture was allowed to settle and the acid layer was collected into another 250 ml separatory funnel. The chlorides were then determined, using Volhard's method (16).

The sulfates were determined in accordance with the method described in (17). The data are given in Table 2. (The sulfonate compounds A-1, B-1, and C-1 are the same rust inhibitors used in formulating MIL-H-6083B hydraulic fluids A, B, and C, referred to in Table 1.)

TABLE 1 - GALVANIC CORROSION DATA ON MIL-H-6083B FLUIDS*

| MIL-H-6083B FLUID | RESULT |
|-------------------|--------|
| A ^b | Fail |
| B ^c | Pass |
| C | Pass |
| D | Pass |
| E | Pass |
| F | Pass |

*Fed. Std. Test Method 5322T

^bMIL-H-6083B Fluid which caused inoperability of the tank hydraulic system.

^cMIL-H-6083B Fluid which permitted satisfactory operation of tank hydraulic system.

TABLE 2 ANALYSES OF COMMERCIAL SULFONATES*

| COMPOSITION, % | COMMERCIAL SULFONATES | | | |
|-----------------------|-----------------------|-------|-------|------------------|
| | A-1 | B-1 | C-1 | D-1 ^b |
| Carboxylate Sulfonate | 0.69 | 0.19 | 0.73 | 0.50 |
| Chloride ^c | 0.12 | 0.03 | 0.03 | 0.01 |
| Sulfate ^d | 0.07 | 0.12 | 0.08 | 0.01 |
| Water | 1.00 | 3.60 | 0.40 | 0.26 |
| Mineral Oil | 64.51 | 39.24 | 69.91 | 62.82 |

*Determined by ASTM Method D1216-56 except for chlorides and sulfates. The latter were determined by using test methods described in references (16, 17).

^bSame as A-1 except that this sulfonate was treated to remove soluble inorganic salts. Purified sulfonate currently used in formulating MIL-H-6083 fluids.

^cCalcd. as calcium sulfonate.

^dCalcd. as barium sulfonate.

^eCalcd. as calcium chloride.

^fCalcd. as calcium sulfate.

Galvanic corrosion tests on the sulfonate samples listed in Table 2 showed that samples B-1 and D-1 were satisfactory, while sample A-1 exhibited corrosion and C-1 was considered borderline. Samples were then prepared in the laboratory in which a sufficient quantity of calcium chloride was added to the B-1, C-1, and D-1 samples to make up a total of 0.12 per cent chloride (same quantity of chloride found in the sulfonate used in hydraulic fluid A, which caused inoperability of the tank). The galvanic corrosion test results are given in Table 3. Photographs of discs typical of a sulfonate containing 0.01 per cent and 0.12 per cent calcium chloride are illustrated in Fig. 4. (C-1 sulfonate was considered borderline, since many tests on many different batches showed only one or two small rust spots on the steel disc. This is in contrast with the solid mass of rust usually noted with sulfonates which promote galvanic corrosion as is evident in Fig. 4.)

TABLE 3 - GALVANIC CORROSION DATA ON COMMERCIAL SULFONATES CONTAINING CALCIUM CHLORIDE*

| SULFONATE | RESULT |
|--|------------|
| A-1 (0.12 per cent chloride, as received) | Fail |
| B-1 (0.03 per cent chloride, as received) | Pass |
| B-2 (chloride content of B-1 increased to 0.12 per cent) | Fail |
| C-1 (0.03 per cent chloride, as received) | Borderline |
| C-2 (chloride content of C-1 increased to 0.12 per cent) | Fail |
| D-1 (0.01 per cent chloride, as received) | Pass |
| D-2 (chloride content of D-1 increased to 0.12 per cent) | Fail |

*Fed. Std. Test Method 5322T

An over-all analysis of the data to this point in our study showed that to resolve this tank inoperability problem, many tests on the tank hydraulic systems would be required. In order to avoid costly expenditures in the use of tanks for test purposes, it was decided to construct a mock-up of the tank hydraulic system for laboratory test purposes. The parts used in the construction of the mock-up were the same as those shown in the schematic diagram of Fig. 1. The mock-up unit, therefore, contained the identical major components found in the M48A2 tank hydraulic system. Two units were constructed. Tests were conducted on the mock-ups in duplicate, with all of the qualified MIL-H-6083B hydraulic fluids listed in Table 1. Included also in the mock-up tests was a fluid formulated to meet MIL-H-6083B using sulfonate D-1 (same as A-1 except that this sulfonate was treated to remove soluble inorganic salts by repeated washings with warm water at 60C saturated with secondary butyl alcohol).

It was found that fluids B to F and the hydraulic fluid made with sulfonate D-1 permitted satisfactory operation of the mock-ups after a storage period of nine weeks. After each test, all of the components were disassembled, and examinations indicated no rust or stains on any of the parts with fluid A, however, after nine weeks' storage the mock-ups would not start and the components were found to be badly rusted. Figure 5 shows typical rusted lock valve spools, guides, and balls. The results on the mock-ups using hydraulic fluids A and B corroborated field test data on M48A2 tanks using these same fluids after six to nine weeks of storage.

A study of the sulfonate analyses (Table 2) indicated that sulfonate A-1 used in hydraulic fluid A (which caused inoperability of the tank and mock-up) also contained a relatively high 0.12 per cent chloride salt, while the chloride salt content of sulfonate B-1 used in fluid B (which was satisfactory in the tank and mock-up) was found to be only 0.03 per cent. The galvanic corrosion data also correlated with the field test data: i.e., fluid A - tank inoperable; promoted galvanic corrosion; fluid B - tank operable; did not promote galvanic corrosion. The data (Tables 1, 2, 3) also showed that hydraulic fluids using sulfonates B-1 and D-1 containing 0.03 and 0.01 per cent chloride salt, respectively, were satisfactory in the mock-ups, showed no corrosion on hydraulic parts, and did not promote galvanic corrosion. The tests on hydraulic fluid C

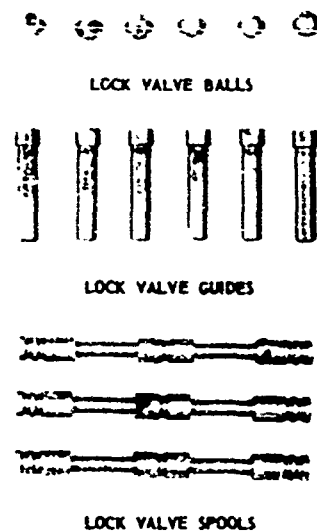


Fig. 5 — Hydraulic Components M48A2 Tank

which contained sulfonate C-1 gave satisfactory performance in the mock-up but, as noted previously, was considered borderline in the galvanic corrosion laboratory test.

An over-all evaluation of these data indicated that the inoperability of the tank systems was associated with the use of commercial mahogany sulfonates which contained a relatively high quantity of chloride salts. Since the inoperability of the tank and mock-up due to rusted components also, in general, correlated with the galvanic corrosion laboratory test (Federal Standard Test Method 5322 T), it would appear that the latter test can be used as a screening test in evaluating the ability of hydraulic fluids to permit galvanic corrosion due to the presence of soluble chloride salts.

Galvanic corrosion tests were also run on sulfonates B-1, C-1, and D-1 to which were added sufficient calcium chloride to vary the salt content from 0.01 per cent to 0.12 per cent. These tests were conducted in an attempt to establish the critical chloride salt content for initiation of corrosion. As seen from the data given in Table 4 that, in general, commercial sulfonates did not initiate galvanic corrosion when the chloride salt content was below 0.03 per cent.

Attempts were also made to determine the ability of sulfonates containing chloride salts to promote galvanic corrosion when added to petroleum lubricating oils of different viscosities. (Oils varying in viscosities from 2.2 centistokes to 38.0 centistokes at 37.8C were tested.) It was found that in all instances, 3.0% or more sulfonate (100 per cent active) containing 0.12% calcium chloride when added to the petroleum-base oil showed definite evidence of galvanic corrosion. The method of test was that described in Federal Standard Test Method 5322T. The results clearly indicate that sulfonates containing very small quantities of chloride salts are deleterious to petroleum lubricating oils having a wide range in viscosity.

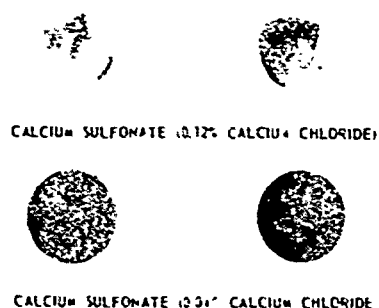


Fig. 4 — Galvanic Corrosion (Fed. Std. Test Method 5322T)

TABLE 4 - GALVANIC CORROSION DATA ON SULFONATES CONTAINING VARYING AMOUNTS OF CALCIUM CHLORIDE

| SULFONATE | CALCIUM CHLORIDE, % | RESULT |
|-----------|---------------------|-------------------------|
| A-1 | 0.12 ^a | Fail |
| B-1 | 0.12 | Fail |
| | 0.10 | Fail |
| | 0.08 | Fail |
| | 0.06 | Fail |
| | 0.04 | Fail |
| C-1 | 0.03 ^a | Pass |
| | 0.12 | Fail |
| | 0.10 | Fail |
| | 0.08 | Fail |
| | 0.06 | Fail |
| D-1 | 0.04 | Fail |
| | 0.03 ^a | Borderline ^b |
| | 0.12 | Fail |
| | 0.10 | Fail |
| | 0.08 | Fail |
| | 0.06 | Fail |
| | 0.04 | Fail |
| | 0.02 | Pass |
| | 0.01 ^a | Pass |

^aCommercial sulfonate as received.

^bOne or two small rust spots on steel disc.

SUMMARY AND DISCUSSION

The primary objective of this work was to determine the cause of the inoperability of the tank hydraulic system. In the light of the performance tests conducted on the tank and mock-up systems, it was apparent that the inoperability was due to the staining and rusting of the components of the hydraulic system.

The analytical data (Table 2) on the commercial mahogany sulfonates used as rust inhibitors in compounding fluids meeting specification MIL-H-6083B revealed that the combined inorganic chloride and sulfate content ranged from 0.02 per cent in D-1 to 0.19 per cent in A-1. It was demonstrated that the A-1 sulfonate containing 0.12 per cent chloride salt caused corrosion of steel in dissimilar metal couples in laboratory tests and, also, was found to cause staining and rusting of components with ensuing inoperability of both the mock-up and tank systems. On the other hand, the use of sulfonate B-1 (C-1 borderline) and D-1 with 0.01 to 0.03 per cent chloride salt did not initiate corrosion, permitted no rust on the components, and mock-ups and tanks were operable.

Further, it was also shown that the per cent sulfate salt found in B-1 sulfonate (Table 2) was the same as the per cent chloride salt found in the A-1 sulfonate (0.12 per cent), yet the B-1 sulfonate did not promote corrosion nor did it permit rusting or staining of the components of the hydraulic system. These results indicated that the deficiency of the hydraulic fluid meeting MIL-H-6083B was associated with the mahogany sulfonates used as rust inhibitors, and that the inoperability due to rusting and staining was caused primarily by the presence of inorganic

chloride salt. Since water (determinations on water content using ASTM D1744 showed that the qualified MIL-H-6083B hydraulic fluids referred to in Table 1 ranged from 0.08 to 0.18 per cent) is invariably present in hydraulic fluids, and dissimilar metals with differences in potential are present, the corrosion can be deduced to be associated with galvanic action.

It was of interest to note that in the absence of dissimilar metals, such as the tests mentioned previously (JAN-H-792 humidity cabinet tests and storage tests with the lock valves), no evidence of corrosion was apparent. This showed that the concentration of inorganic salts found in the commercial mahogany sulfonates studied did not significantly affect the rust prevention properties but their presence, particularly the chloride salt, did permit corrosion due to galvanic action.

The following is offered to describe the behavior of the inorganic salts. In the galvanic couple used in this work, the arrangement was such as to make the steel component anode. Under conditions where soluble inorganic salt is present, as in the commercial mahogany sulfonates described here, it was to be expected that the anion component of the salt would take place at the liquid-metal interface. This has been amply demonstrated, as is shown in Fig. 4. The steel surface in the galvanic cell will orient water molecules (due to the potential gradient of the system) so that the oxygen component is adjacent to the metal and the hydrogen portion farther away. It is quite possible for metal cations, therefore, to move into positions between the oxygen atoms, thereby displacing from the water molecules the appropriate number of hydrogen ions. This reaction would leave solid oxide or hydroxide on the metal surface (18). When a layer of oxide or hydroxide has been built up, water molecules will attach themselves to the oxide or hydroxide layer. The process then repeats itself until the film is thick enough to prevent further cations moving outward, thus providing a surface relatively passive to corrosion.

The sulfonates reported here (Table 2) were found to contain both chloride and sulfate salts. According to the adsorption theory (19), chloride ion adsorbs on the metal surface in competition with dissolved oxygen or hydroxide ion. Once in contact with the metal surface, chloride ion favors hydration of metal ions and increases the ease with which metal ions enter into solution, opposite to the effect of absorbed oxygen which decreases the rate of metal dissolution. From the point of view of the oxide film theory (20,21), chloride ion penetrates the oxide film through pores or defects easier than do other ions (e.g., sulfate), or chloride ion may colloiddally disperse the oxide film and increase its permeability (20). Sulfates not only do not penetrate oxide films readily, but form soluble corrosion products to a much lesser extent than the halide salts and are, therefore, not as corrosive as the halides (22). The data (Table 2) indicate this to be the case since the 0.12 per cent sulfate concentration of sulfonate B-1, while the same as the chloride concentration of sulfonate A-1, yet permitted no corrosive action to take place. Additional tests were run in which the sulfate content of sulfonate B-1 was further increased to a total 0.20 per cent, and again no corrosion was noted. On the other hand, the chloride salt concentration of sulfonate A-1 (0.12 per cent) effectively demonstrated that corrosion will occur.

Previously it was mentioned that Baker et al. (15) studied corrosion of steel balls and races in brass retainer ball bearings. It was concluded "that a hygroscopic impurity accumulated enough moisture at the oil-metal interface to set up localized galvanic corrosion. The effect was more marked in the presence of petroleum sulfonate rust inhibitors than in their absence, and was tentatively attributed to some constituent of these additives." In the light of the data presented in this paper, it is highly probable that the constituent of the sulfonate additives indicated in the above reference is inorganic chloride salt.

The findings reported in this paper are extremely important since mahogany sulfonates are at present extensively used as rust inhibitors by the military and industry. Recommendations are underway to permit the use of mahogany sulfonates as rust inhibitors for lubricants for military use only if the soluble inorganic chloride salt content (calcd. as calcium chloride) is not above 0.02 per cent and the sulfate content (calcd. as calcium sulfate) does not exceed 0.05 per cent. It is expected that other users and/or suppliers of these products will initiate similar recommendations.

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| 11 SUPPLEMENTARY NOTES | | 12 SPONSORING MILITARY ACTIVITY USA NUCOM | |
| 13 ABSTRACT A series of mahogany sulfonate rust inhibitors used in compounding qualified petroleum hydraulic fluids was studied in connection with the inoperability of tank hydraulic systems after short storage periods of six to nine weeks. Examination of the disassembled hydraulic components showed severe staining and rusting in valving areas having extremely critical tolerances. Analyses of the sulfonates indicated the presence of inorganic water soluble salts (calcd. as calcium chloride and calcium sulfate) ranging from 0.02 to 0.19 percent. It was found that the hydraulic fluids used in the tanks which did not perform satisfactorily contained sulfonates having a relatively high chloride salt content of 0.12 percent, while the use of hydraulic fluids with sulfonates containing less than 0.03 percent of the halide salt permitted satisfactory operation. Data are also presented which show that the concentrations of inorganic salts found in the commercial mahogany sulfonates studied do not appear to significantly affect the rust prevention properties of the sulfonates but their presence does promote corrosion due to galvanic action. A correlation was established between laboratory galvanic corrosion tests and performance tests in tank and mock-up systems. | | | |

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Security Classification

| KEY WORDS | LINK A | | LINK B | | LINK C | |
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| | ROLE | WT | ROLE | WT | ROLE | WT |
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