

# DEVELOPMENT OF ORGANIC COATINGS FOR USE AS LININGS OF BULK FUEL STORAGE TANKS

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CHEMIETRY DIVISION

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- Subj: Development of Organic Coatings for use as Linings of Bulk Fuel Storage Tanks; NRL Problem 32C03-17
- Ref: (a) BuDecks ltr L5-2 P334/3hd of 20 Dec 1948
  - (b) NRL Report 3685--"Organic Coatings Adaptable to Fuel Storage"--by Jack E. Cowling, Irving J. Eggert, and Allen L. Alexander, of 22 June 1950

#### INTRODUCTION

By reference (a) this laboratory was authorized to undertake a program of research and development on organic coatings suitable for use as linings in both concrete and steel bulk fuel storage tanks. Reference (b) was a formal interim report of the progress made on this project up until 22 June 1950, and also included a resume of the history and background experience on the use of organic linings in fuel storage tanks.

A second formal interim report is now in preparation and will include data and results of research through December 1951. In order that the principal results of this work may be brought to the attention of all interested activities with a minimum delay, this letter report has been prepared.

During the eighteen months since the preparation of NRL Report 3685 (Ref b), research on the subject problem has been intensified. The results of experiments have made it possible to narrow constantly the field of likely materials until now a relatively few remain which appear to hold promise of adaptability to use as components in organic linings for fuel storage tanks. However, the coatings industry is constantly developing new types of products, and the possibility of using some of these is not being overlooked, but it seems that several of the materials, which have behind them more than two years study and developmental work, will be the first fully satisfactory linings for bulk fuel storage tanks.

Research has progressed to a point where "Reverse Phase" type coatings i.e. emulsions based upon a polysulfide rubber (Thickol) and any of a number of vinyl copolymers, appear potentially satisfactory. As a result, a contract has been made between ONR and the Thickol Corporation for research to bring out the ultimate in the development of tank linings. One phase of this company's research is being devoted to developing the sptimum reverse-phase coating.

This laboratory has constructed a pilot-scale concrete tank installation (Figure 1) for the final evaluation of the better laboratory developments. It is contemplated that approximately ten good prospective lining systems may be evaluated on these tanks each of which will contain slightly less than a thousand gallons of fuel. Considerable experience on the practical aspects of the problem has been gained through visits by NRL scientists to field installations where bulk fuel tank reconditioning operations were to be undertaken. Field activities have often requested the advice of our personnel in specifying the requirements for the relining of bulk fuel tanks.

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#### FIELD EXPERIENCE

The purpose of this research program is to make available to the operating bases and stations of the naval establishment a more inert, longer lasting, semi-elastomeric coating for use in lining new and relining old bulk fuel storage tanks. The real test of the worth of any research development is its performance in normal service. Field experience has shown beyond doubt that the vinyl coatings permitted by BuDocks Specifications Lia (1944) have the desired inertness. However, they lack the elasticity of an "ideal" lining material, and in their application the large quantities of hazardous solvent vapors which must be coped with are objectionable. It has been shown that both Thiokol Latex and Thiokol FA-Sheet linings have the desired elasticity of an "ideal" material, but lack the inertness necessary for long service with some grades of fuel, particularly jet fuel JP-3 and diesel oil.

Within the past year the Naval Research Laboratory has been requested to recommend methods for relining concrete bulk fuel storage tanks at several locations. The new linings recommended for installation in these tanks represent the most advanced laboratory developments at this time. It is believed that they will give considerably longer satisfactory service than the thickol linings they replaced, but of course only time in actual service will prove beyond doubt the adequacy of laboratory developments and tests.

In order that the widest possible knowledge may be had of typical situations which may be met in service the following are resumes of tank lining problems actually encountered.

<u>Problem A:</u> An above ground concrete tank, unlined, had been used for diesel oil storage for about five years. It had commenced to leak rather badly and NRL was consulted concerning corrective measures. Inspection revealed that the great majority of leakage was occurring near the bottom of the tank, --probably through the ring seal though this fact could not be definitely established. It was obvious that the concrete was thoroughly oil soaked, and it was doubtful that a surface suitable for the application of an organic lining could be developed by any practical cleaning measures. In this connection steaming and sandblasting followed by "guniting" were considered. Therefore, it was recommended that the diesel oil be floated on a twelve to eighteen inch layer of water to be maintained in the tank. Water lost by leakage could be replaced at virtually no cest. The necessary piping modifications for handling the fuel and maintaining the proper water level were easily and economically accomplished. The Public Works Officer at the station concerned has reported that the remedial measures have been fully satisfactory after slightly more than a year of service. Figure 2 is a drawing showing the modifications installed by the Public Works Office concerned in order to carry out the recommendations for the solution of this problem.

Problem B: This laboratory was consulted concerning leakage from an above ground concrete tank used for diesel oil storage, and which recently (within the year) had been relined with a coating based upon a vinyl resin. Inspection revealed that in preparation for relining, the tank had been sandblasted, presumably to a depth below the penetration of oil, and a new concrete surface then gunited on the walls, after which the vinyl resin coating was applied. There were numerous hair-line cracks in the coating and gunite at the time of inspection by NRL's representatives. The gunite was not tightly bonded to the base concrete, for it gave perceptibly under blows from the fist. This condition was obviously the underlying cause of leakage. Vinyl resin coatings lack the elasticity necessary to bridge hair-line cracks such as occurred in this tank. A more flexible, elastic lining would have bridge these cracks and prevented leakage for some time, -- but this of course is no justification for a poor job of guniting. Other criticisms of the job were as follows:

(1) There was no single contract for the entire reconditioning operation, but rather three separate contracts, --one for cleaning and sandblasting, another for guniting, and a third for the lining application. The difficulties in fixing responsibility upon any single contractor for the conditions leading to early failure are obvious.

(2) The vinyl coating was very thin, and although this was in no way responsible for the early failure and resulting leakage, it is felt that the thinness of the coating would have eventually manifested itself as a cause of failure had the reconditioning job been fully satisfactory in other respects.

(3) The surface of the gunite was extremely coarse. The use of a fine sand is desirable to provide a fairly smooth texture surface for application of the vinyl coating. Some of the sharp, coarse sand particles standing out on the surface were virtually uncoated.

Problem C: Recommendations were requested pertinent to stopping leakage from a newly constructed, above ground, bolted steel tank. Inspection of the tank, and consultation with officials at the base concerned, revealed that leakage had occurred around the gaskets, which were neoprene, and was severest at the bottom of the tank. The Public Works Officer had decided to pour a three-inch concrete slab on the floor of the tank and coat it with Thiokol in order to effect a good seal. Since the intended contents of the tank were jet fuel JP-3, which was known to have a harmful effect upon Thiokol, it was planned to paint the Thickol with several coats of various vinyl resin base compositions.

At the time of inspection by NRL's representatives, the concrete was being prepared for application of the Thiokol. It was recommended that the Thickol be overcoated with at least three coats of BuShips Formula 113/49, a composition based upon a copolymer of vinylidine chloride (Saran), instead of the vinyl resin materials as originally planned. Tests by this laboratory have shown the Saran to maintain good adhesion to Thickol after months in contact with aviation fuels, while vinyl resin base materials lose their adhesion to Thiokol after six or eight months under similar conditions. It was also recommended that all bolted seams throughout the tank receive the same protective Saran coating to prevent leakage. The recommendations were carried out only as they pertained to the floor. Although this tank has not been reinspected by NRL's scientists, it has been verbally reported that leakage occurred through the seams above the bottom of the tank, and consideration was being given to switching the tank to water storage.

Problem D: It was requested that NRL inspect and make recommendations for the rehabilitation of two large underground concrete tanks which were being converted from the storage of aviation gasoline to storage of jet fuel JP-3. The linings of Thickol FA-Sheet had been installed in 1943-44, at the time of construction, and had contained only aviation gasoline until inspection by our personnel in September 1950. Inspection revealed varying degrees of deterioration of the lining in each tank as evidenced by a few blisters, cracking and checking, some loss of adhesion, and loss of elasticity in the lower three or four feet of wall lining. Moderate rubbing of the lining removed considerable loose carbon black, thus indicating some measure of disintegration of the binder. The condition of the better of these two linings was certainly "very good" while the poorer one could probably have been rated as "fair". Both linings could have continued in service with gasoline for several years, but in view of the known deleterious effects of JP-3 on Thickol, it was felt that the remaining useful life of these linings with JP-3 would be very short.

It was recommended that repair operations be immediately undertaken in one tank while the complete replacement of the second lining was recommended. In order to protect the repaired lining from the deleterious effects of jet fuel JP-3, the application over the FA-Sheet of an inert system comprised of several coats of reverse phase and Saran was recommended. The complete new lining recommended for installation in the other tank was a combination of Thickol Latex, Reverse Phase emulsions, and Saran solutions designed to give the balance of properties desired.

Approximately one year elapsed before the suggested repair measures could be undertaken. During this time the tanks were switched to service with JP-3. When repair operations were at last begun, it was found that the deterioration of the old linings had reached an advanced stage, and again NRL was consulted for recommendations on how to proceed.

The reinspection showed that JP-3 had caused so much additional deterioration of the FA-Sheet (a fact not surprising in view of previous experience) that complete relining of both tanks was now a necessity.

Accordingly, new recommendations for the job were prepared, and included the use of a thin Reverse Phase bending ceat to obtain better adhesion of the Thiokol to concrete. Such a procedure seemed advisable based upon laboratory experiments subsequent to the initial inspection of these tanks. However, when the relining operation was begun it proved impossible to obtain adhesion of the bonding coat to the cleaned surfaces, probably because of traces of moisture in the concrete. On the other hand, Thiokol Latex adhered tenaciously. Consequently, except for a small area where the adhesion of the Reverse Phase bonding coat was fair, the job was completed with the application of Thiokol Latex directly to the cleaned, lightly sandblasted concrete. The final coating system used consisted of the following:

- (1) Four coats of Thickel Latex, WD-7, with Osreburg cloth reinforcement imbedded in the second and third wet coats.
- (2) Two coats of Thiokol-VAGH Reverse Phase emulsion
- (3) Three coats of BuShips Formula 113/49 (Saran)

The system adheres well to concrete, possesses good flexibility and elasticity, and both the Reverse Rase coating and Saran have displayed outstanding inertness in laboratory tests.

Problem E: NRL was requested to inspect a large underground concrete tank lined with Thickol (applied as latex) which had contained aviation gasoline for about five years followed by two years service with jet fuel JP-1, and which was now about to be returned to service with aviation gasoline. Inspection revealed the lining to be in perfect condition with two or three negligible, minor exceptions. No repairs whatsoever were necessary, and the tank was immediately returned to service.

Problem F: A number of underground, rectangular concrete tanks of relatively small capacity were to be placed in service containing jet fuel. The tanks had once contained aviation gasoline but had been out of service for several years. NRL was requested to inspect them and recommend measures necessary to preserve their Thickol FA-Sheet linings. Inspection revealed the linings to be in generally good condition in spite of a few scattered blisters and minor cracking and checking. It was recommended that the blisters and other bad portions of the old lining be cut away and patches applied, in general following the procedure outlined in BuYards & Docks Specification LLa. Since it was not certain that harmful grades of jet fuel could always be excluded from the tanks, it was recommended that an inert coating be applied over the FA-Sheet. Four coats of a Thickol-Vinylite VAGH Reverse Phase were suggested; or four coats of BuShips Formula 113/L9 (Saran) as an equally satisfactory option. No reports have been received concerning the action taken by the station concerned.

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Problem G: NRL was requested to reinspect an underground concrete tank lined with Thiokol FA-Sheet, and which had been in service for fifteen months with jet fuel JP-3 preceded by approximately twelve months service with JP-1. Prior to its service with JP-1, the tank had contained aviation gasoline. This tank was initially inspected by NRL scientists in February 1949, and was at that time rated as being in excellent condition. By May 1951 (27 months later), when the second inspection was made, the lining had deteriorated to a condition of virtually complete failure. The lower half of the wall area was largely bare while the lining remaining on other areas of the tank was "lifeless" and exhibited severe cracking and alligatoring. It was evident that degradation of the lining had progressed at a greatly accelerated rate since the addition of jet fuel to the tank immediately following the initial inspection. It is regrettable that NRL was unable to inspect the tank after twelve months service in the storage of JP-1, and just prior to the introduction of JP-3. However, based upon previous experience and knowledge of the fuel characteristics, it is assumed that only a very minor amount of the lining degradation is attributable to JP-1, and it is believed that the fifteen months service with JP-3 is largely responsible. Complete relining of the structure was recommended. However, officials at the station concerned indicated that there were no immediate plans contemplated for this purpose, and the tank was withdrawn from service.

The above actual service experiences have been briefly reported in order to present a fair example of the kind of problems being encountered in the field of bulk fuel tank linings. (Other experiences were cited in the initial report on this subject, Ref b).

The conclusion should not be drawn that organic liners for bulk fuel storage tanks are in large measure rapidly disintegrating after relatively short service. In fact the opposite is true. Several hundred tanks built during World War II, and lined with materials based upon either Thickol or vinyl resins, have given and are continuing to give very satisfactory service in the storage of gasoline. In general their use has been a complete success, and the relatively few instances of severe lining degradation have been attributable to a fuel (JP-3 jet fuel) which did not exist at the time when most of the storage facilities were constructed.

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Other conclusions that can be reached, based upon actual experience, are as follows:

(a) Certain vinyl resin coatings possess the desired degree of inertness but are too brittle or inflexible to bridge hair-line cracks which may occur in the concrete. If normally plasticized, the plasticizer is rapidly leached from the coating by the fuel. The quantity of material so leached has no noticeable affect upon gum content or other fuel properties, but of course leaves the coating unplasticized. Hazardous solvents are necessary in the application of the usual vinyl-type coatings.

(b) Thiokol, although adequately inert for use with gasoline, is unstable to prolonged contact with some grades of jet fuel, diesel oil, and fuels containing appreciable quantities of mercaptans. Its advantages lie in its rubber-like properties, excellent adhesion to concrete and the fact that it may be applied in latex form, thus obviating the use of inflammable and hazardous solvents.

(c) Some evidence has been observed indicating that water, particularly that under a hydrostatic pressure which diffused through the concrete to the underside of the lining is equally as harmful as any fuel contained.

#### EXPERIMENTAL PROCEDURE

The methods for evaluating the inertness of experimental coatings are described in NRL Report 3685 (Ref b). Briefly, they consist of two sets of conditions. In one instance small concrete tanks to which the experimental linings have been applied are partially filled with a small amount of water and one of the following fuels:

(1) Blend: 60% 115/145 av. gas (Mil-F-5572) 20% Toluene 15% Xylene 5% Benzene

(2) 115/145 av. gas (Mil-F-5572)

(3) JP-3 jet fuel (Mil-F-5624)

(h) Diesel Oil (Mil-F-8961, class 3, 50-cetane)

The tanks with linings are sealed to prevent evaporation and exposed outdoors for 30 days; then emptied and allowed to dry for 5 days after which they are partially filled with water and another of the fuels, and the test repeated. Thus, a complete test cycle employing the above four fuels requires 1h0 days. Test linings are passed repeatedly through this cycle until failure. Some are still serving satisfactorily after two years under these conditions.

In the second experiment to evaluate inertness, steel panels are coated with the experimental lining materials, then immersed in a fuel-water blend at 130°F for 5 days. After removal and drying for 2 days, they are subjected to a second exposure to one of the afore-mentioned fuels. A complete cycle in this test requires 28 days. Panels with their test coatings are thus exposed until failure or for six months, whichever occurs first.

These tests provide an accelerated rate of degradation, but it is believed that the results give a fair indication of the comparative life of a lining under actual service conditions.

In addition to the above experiments, a test has been devised for evaluating the relative adhesion to concrete of the various linings. The experimental coatings are applied to concrete blocks, with a cloth reinforcement imbedded in the test coating to facilitate its removal by stripping. After the coating has thoroughly cured, a razor blade is used to cut two parallel slits one inch apart in order to make possible the measurement of the force required to pull back (strip-off)a one-inch width of coating. A Scott Tensile Tester is used for this purpose and makes a graph of the force required in pounds to remove the one inch wide strip of lining for the length of the block (6 inches). This procedure appears to be a fully satisfactory method of measuring an empirical value for adhesion. The method is practical and the results reproducible. Figures 3 and h illustrate the procedure and results respectively.

#### DATA AND RESULTS

Complete experimental data are reported in the forthcoming, formal, interim report, and because of their voluminous nature will not be included here. The experimental facts discussed here are taken either directly from the data or are conclusions reached after a study of those data.

Briefly data collected from the experiments thus far conducted show the following:

(1) A number of vinyl resin base coatings display adequate inertness but become brittle in service.

(2) Saran F-120, 1000 cps grade, forms a generally acceptable tank lining. It adheres well to Thiokol and possesses adequate inertness and elasticity. (3) Saran F-120, 1000 cps grade adheres satisfactorily to Thiokol even after many months of service with the test fuels. No other vinyl resin has shown this property.

(4) Certain Reverse Phase emulsions of (a) Thiokol and "Vinylite" VAGH, and (b) Thiokol and Saran F-120 display adequate inertness and physical characteristics for use as tank linings. Some are still satisfactory after more than a year in test.

(5) A 50/50 blend of the latex of Thiokol WD-6 with the aqueous resin emulsion of Saran F-122-A20, requiring no hazardous solvents, has passed twelve months test as a tank lining. Other blends of these latices are satisfactory after three months in test.

(6) Several vinyl resin base coatings appear satisfactory for use on steel. Saran F-120, 1000 cps grade, which is a resin meeting the requirements of Bureau of Ships Formula 113/49, is likewise satisfactory as a liner for steel tanks. The Reverse Phase products mentioned in subparagraph (4) above are also satisfactory. The use of suitable primers, specifically Pretreatment, Wash Frimer (MIL-P-15328) and Primer, Vinyl - Zinc Chromate (MIL-F-15930), markedly reduce the tendency of the steel to corrode.

(7) Both lead chromate and red lead improve the inertness of vinyl resin coatings based upon "Vinylite" VYHH. Reverse Phase coatings using "Vinylite" VYHH are likewise improved by these pigments.

(8) Certain furan, polyester, and epoxide resins possess the degree of inertness required of tank linings but lack the extensibility necessary for bridging fine cracks. However, the versatility of the expoxide resins is so great that it now seems probable that a copolymer using these materials will eventually be developed which will be suitable for use as a lining for bulk fuel storage tanks.

(9) Thickol Latex WD-6 is slightly superior to Thickol Latex WD-7 in inertness. On the other hand the WD-7 latex gives the better adhesion to concrete. However, since the adhesion of the WD-6 material is satisfactory on well cleaned concrete, the use of WD-6 in preference to WD-7 is normally desirable.

(10) Satisfactory adhesion is obtainable in most instances when cleaning and surface preparation prior to relining old tanks include either steam cleaning or sandblasting; and occasionally both are required.

#### DISCUSSION

The use of jet fuel is advancing rapidly and the need of satisfactory facilities for its storage in large quantities is increasing

accordingly. It is therefore essential that linings be found which are suitable for containing this fuel. Relining operations will become more prevalent as the existing structures age, and obviously it would be desirable to provide replacement linings inert to all petroleum fuels. When it becomes necessary to convert a Thiokol lined tank to the storage of jet fuel or diesel oil, it would be an economy to protect the Thiokol lining (assuming it still to be serviceable) from the deleterious action of the new contents.

Our experience indicates that it is possible to apply an inert coating over old Thiokol still in good condition so that its life will be materially and economically extended beyond that which otherwise could have been expected. Should the old lining be in poor condition, it should be replaced entirely.

In order to provide a complete solution to the problem efforts are directed toward the development of an "ideal" lining having the following properties:

- (1) Excellent adhesion to concrete
- (2) Excellent adhesion to old Thiokol linings which are still in fair or better condition
- (3) Elasticity and flexibility sufficient to bridge hairline cracks which may occur in concrete at any time during its life
- (4) Inertness to all foreseeable petroleum fuels except bunker oil
- (5) Inertness to fresh and salt water
- (6) Applicable by normal methods from a water emulsion
- (7) Good package stability (storage life)

Development has progressed in the laboratory to a point where a lining system has been devised which possesses a good measure of all the above seven properties, the main shortcoming being that only about half the coats can be applied in latex form, the remainder requiring the use of hazardous solvents. The following system of coats will provide a very good lining of this type:

Step 1. Apply 1st coat of Thickol Latex; allow to dry and cure.

Step 2. Apply 2nd coat of Thiokol Latex; imbed preferably glass cloth (Owens-Corning ECC-11-111) or Osnaburg fabric in the Thiokol Latex while still wet; then immediately apply a 3rd coat of Thiokol Latex; allow to dry and cure.

Steps 3 and 4. Apply 4th and 5th coat of Thickol Latex; allow to dry and cure. Note: Coverage per coat of Thickol Latex should be about 140 sq. ft. per gallon at 55% solids.

Step 5 thru 11. Apply seven brush coats of BuShips Formula 113/49 (pigmented Saran solution), alternating white and orange coats. Coverage should be approximately 150 sq. ft/gal/coat of material as supplied conforming to specifications. Allow twentyfour hours drying between coats. Gertain Reverse Phase coatings may be substituted for the Formula 113/49. They will provide slightly less toughness, flexibility and extensibility, but may provide better intercoat adhesion. Laboratory experiments have revealed no marked differences between the performance of the better Reverse Phase coatings and Saran F-120, 1000 centipoise grade, but a slight preference is indicated for the latter material because of its background of satisfactory service as a liner for the holds of tankers, and the additional fact that it is covered by a Navy Specification,-BuShips Formula 113/49. It is normally a stock item.

While the foregoing system of coatings comprises a good lining, it must be regarded as only an interim solution to tank lining problems. The immediate aims in the continuation of research are three; namely,

(1) Devising a coating system requiring the use of no inflammable or hazardous solvents;

(2) A study of the effect of various pigments, particularly those of lead, upon lining properties; and

(3) The development of more or less standard methods for reconditioning operations. It is probable that "standard" methods will suffice for approximately ninety per cent of all relining operations, but the remainder will require special technical services by this Laboratory or other experienced consultants.

Of no less emphasis, but somewhat longer range planning are the following additional phases in this research and development program:

(h) The development of the optimum Thiokol Reverse Phase coatings for use as bulk fuel tank linings;

(5) Greater inertness, particularly of the initial coats,i.e. the ones bonding the top, more inert coats to the concrete;

(6) Continued appraisal of new developments in the protective coatings field.

Good progress has been made toward the objectives of the above six phases of the research program. The first three have received the most emphasis as they are of the greatest immediate importance. The following six sub-sections summarize the progress made to date on each of these phases of the research program:

(1) Devising a coating requiring the use of no hazardous solvents: Exploratory experiments in which a vinylidine chloride copolymer in aqueous dispersion, and containing

a plasticizer, was blended with Thiokol Latex, WD-6, on a 50/50 solids basis produced a coating of good inertness, toughness, adhesion and elasticity based upon ten months exposure on a small concrete tank placed in the cycled fuel test. After six months in this test the results appeared so promising that four additional tanks were placed in test to evaluate other ratios of water dispersions of vinylidine chloride and Thickol Latex blends. Other grades of both materials are available and it may prove desirable to further expand this phase of the reserrch at an early date. The plasticizer is rapidly leached from the coating, but its proportion is so small compared to the volume of even a relatively small fuel storage tank that the quality of the fuel is unaffected. At the same time, the coating remains fully satisfactory. It is hoped that a reasonable amount of additional laboratory experimentation and testing will justify the use of one of these all latex coatings in lining a field installation in the near future.

(2) Study of effects of pigmentation: There are two reasons for pigmenting the component coatings making up the final tank lining. The first is that it can facilitate application and coverage. It is often customary to pigment the same fundamental formula in two contrasting colors, thus making it easier to obtain proper coverage with each successive coat (alternate colors) in the poorly lighted interior of a fuel tank. This practice is used in applying Bu Ships Formula 113/h9. A second reason for pigmenting is that proper pigmentation will often greatly improve the quality of a coating. (An improper choice of pigments may have an equally marked detrimental effect.) The inertness of both vinyl and Thiokol coatings can be increased by the use of lead pigments. However, as is well known, the package stability may be affected adversely at the same time.

Primarily, the pigment study phase of this laboratory's research is being directed toward improving the inertness of the coating. A wide choice of unreactive pigments are available for providing contrasting colors to aid in application. Froprietary products have been received containing various pigments, including lampblack, titanium dioxide, mica, zine oxide, zine dust, aluminum, and others, but so far none allegedly have contained any lead pigments. We have prepared and evaluated a number of Reverse Phase type coatings, both clear and pigmented with non-reactive types of pigments. The poprest of these reverse phase coatings was chosen for the purpose of evaluating the effect of various lead pigments. Steel panels coated with the experimental formulations were cycled through the four test fuels at 130°F. At the end of nine weeks the paints containing lead pigments (lead chromate, and red lead) are perfect while those containing no pigments failed in about five weeks. The tests are continuing and must be expanded to include other lead compounds. Storage test also must be made before definite recommendations can be given concerning pigmentation with lead compounds. It has been noted that formulas containing pigments such as mica, zinc oxide, lampblack, etc., give results intermediate between those obtained with unpigmented Reverse Phase coatings and those containing red lead or lead chromate. Fanels which were primed with a "Wash Primer" (MIL-P-15328) and Vinyl-Zinc Chromate Primer (MIL-P-15930) were superior to those which were unprimed. This latter fact is attributed to the corrosion inhibitive properties of the primers.

(3) Standardization of cleaning and other preparatory measures for lining or relining operations: More or less standard procedures must be developed for lining new, and relining old structures. New concrete meeting Bureau of Yards and Docks specifications for Prestressed Concrete Bulk Fuel Storage Tanks requires but little attention prior to applying organic coatings. For the application of vinyl type coatings it should be etched with a ten per cent solution of hydrochloric (muriatic) acid, washed, and allowed to dry thoroughly before commencing application. For the application of Thickol Latex the surface should be wetted down, and the initial coat applied while the concrete is very slightly moist, but not wet.

Field experience is rapidly emphasizing the need for standard and dependable measures of cleaning and preparing an old tank for the application of a new lining. Frequently the concrete will contain some fuel which has soaked in during years of service, or it may contain residues from the adhesive used with Thiokol FA-Sheet. It usually will have a relatively high moisture content. These are the conditions always to be anticipated, but no doubt still others will be encountered as the breadth of our experience increases.

It is not readily possible to carry out on a laboratory scale all the prospective cleaning measures which might be used with certainty that they will prove feasible, practical, and economical when applied in the field. For example, this laboratory has been able to steam clean quite adequately h inch by 6 inch concrete blocks preparatory to applying test linings, and the latter have adhered well. But, while in the laboratory this is a practical cleaning measure, the quantities of steam necessary to clean a large concrete tank may be excessive. Recently, a number of the field structures have been relined, and the preparatory measures included as the final step a light sandblasting to provide a relatively clean surface. The minute quantities of fuel remaining in the concrete did not interfere with the application of Thickol Latex. As much as possible of the old lining had been removed by hand scrapers, etc. prior to sandblasting.

At this stage in the research it is believed that the following cleaning measures should be considered in contracting any relining job.

- (1) Mechanically strip off the maximum amount of eld lining and adhesive, if present.
- (2) Steam clean, and/or
- (3) Lightly sandblast to remove all traces of old lining (and adhesive) and excessive quantities of absorbed fuel.

These preparatory measures should suffice for more than ninety per cent of the relining operations; and the remainder, when encountered, must be decided individually by experienced consultants.

Other cleaning methods are under consideration such as the use of detergents, acid etches, and etc., but these must be further proven before being recommended.

(4) The development of the "optimum" Thickol Reverse Phase type coating: The preparation and evaluation of emulsions of Thickol and vinyl resins, known as Reverse Phase materials, for use in the role of tank linings is receiving attention. In addition, the Thickol Corporation is attempting to develop the "optimum" in these coatings as one phase of their research contract. There is a complete exchange of ideas between laboratories and each checks and evaluates the results of the other.

(5) Improving the inertness of the lining,-particularly initial (bonding) coats: In the search for more inert bonding coats the following materials are under study:

- (a) "Base" Reverse Phase solutions which contain approximately 7% "Vinylite" VMCH with Thickol WD-6. These materials have outstanding adhesien to concrete.
- (b) Blends of Thickol and Saran latices
- (c) Polymer blends such as Thickol with epoxide resins.

(6) Continued appraisal of new developments: Already this phase of research has included exploratory evaluation of furan resins, polyesters, "Epons" and others. While most of these materials are sufficiently inert, the conditions necessary for their application preclude their consideration at present. It is conceivable, however, that further developments with any of these classes of materials may make them of practical value for tank lining use.

#### CONCLUSIONS

Research on this problem has attained an advanced stage making it possible to reach a number of conclusions based upon the combination of actual service experience and laboratory experimentation.

The most important conclusion, and Objective, reached thus far is that an essentially new lining system has been developed which is superior in its over all performance to those permitted by Bureau of Yards and Docks Specifications Lua. This development is referred to as the "Interim Lining" for it is superior to those used in the past, but not equal in all respects to the "Ideal Lining" which is sought. Briefly, it consists of five coats of Thiokol WD-6 containing imbedded glass or Osnaburg fabric reinforcement, preferably glass, topped with seven brush-coats of Bureau of Ships Formula 113/49, the latter being based upon a vinylidine chloride copolymer vehicle. The system has been fully described on page ten of this report.

Other conclusions which have been reached are as follows:

(1) Certain Reverse Phase type coatings combining Thickol and vinyl type resins possess adequate inertness for use as tank linings as well as sufficient adhesion, elasticity and toughness. NRL has found proprietary materials of this class containing

- (a) Approximately 60% Thickol WD-6 to 40% "Vinylite" VAGH or
- (b) Appreximately 50% Thiokol WD-6 to 50% Saran F-120 (1000 cps grade), to be satisfactory. While the solids content and viscosity of these proprietary products were lower than desirable these properties can be adjusted upward. Reverse Phase type materials are best used as about five or more coats over a Thiokol WD-6 base in the same manner as that followed in the application of Saran F-120 solutions.

(2) With the exception of one property, that of bridging hairline cracks which may occur in the substrate, certain vinyl base coatings are fully satisfactory for use as tank linings. Where this property can be discounted satisfactory service can be expected from these materials. For application to steel tanks it is believed that vinyl base coatings are on a par with Bureau of Ships Formula 113/49 now extensively used in lining the holds of Navy tankers. The vinyl base materials permitted by Bureau of Yards and Docks Specifications Lia are satisfactory with the single exception of bridging hair-line cracks which may occur in concrete. (3) Good progress is being made in the development of a 100% latex coating system. As yet insufficient data have been accumulated to recommend a material for immediate use, but initial experiments are very encouraging. Greatest success along this line has been obtained by blending the latex of Thiokol WD-6 with an aqueous dispersion of Saran F-122, A20. Various proportions are now under study in order to determine the optimum.

(h) Preparatory measures for reconditioning operations can be standardized for about ninety per cent of the jobs arising. The remainder will require special technical services by experienced consultants, or scientists of the Naval Research Laboratory. Conditions which may indicate a requirement for special attention are (a). excessive amounts of absorbed fuel in the substrate, (b). excessive leakage, (c) the presence of large quantities of water within the tank, (d) unusually large cracks, and (e). inability to obtain good adhesion with the initial coat. The "standard" cleaning and preparatory measures are covered in the final section of this report, -Recommendations.

(5) Proper pigmentation, and particularly the use of lead pigments, tends to greatly improve the quality of linings containing Thickel and certain vinyl resins. Experiments along this line are not yet far removed from the exploratory phase, but results have been sufficiently attractive to warrant continuation and expansion of this phase of research. Package stability must at the same time be investigated.

(6) A test for determining an empirical value of adhesion has been developed. Results are reproducible, and are of great value in comparing this property of the various prospective coatings. Briefly, it involves the measurement of the pounds of pull required to strip off a one inch wide strip of lining from a concrete block. A Scott Tensile Tester is the instrument required, and the rate of separation of the jaws should be ten inches per minute or less.

#### RECOMMENDATIONS

It is recommended that, pending the development of a more or less "Ideal Lining," the following procedures be followed in lining new, or relining old bulk fuel storage tanks.

### 1. Cleaning:

- (a) Steel should be scale and rust-free. This condition is best obtained by wire brushing or sandblasting.
- (b) Concrete should be as clean and dry as obtainable by practical measures. For new concrete, wash away loose dust and dirt, and allow to dry. In old tanks it is usually impractical to remove the last traces of

absorbed fuel and gum, therefore the following cleaning measures should be carried out:

- (1) Mechanically remove the maximum amount of old lining and adhesive deemed practical.
- (2) Steam clean, and/or
- (3) Lightly sandblast to remove all traces of old lining and adhesive, and excessive quantities of absorbed fuel and gum.
- (4) Make several adhesion tests with test patches, and repeat cleaning measures if necessary.

#### 2. Lining

- (a) Steel: Apply five brush coats of Bureau of Ships Formula 113/49, alternating white and orange colors. Coverage should be at least 150 sq. ft. per gallon per coat based upon the unthinned material as received conforming to specifications. Occasionally it is difficult to obtain good adhesion to steel with this material. This can be remedied by the use of primers, the following being recommended:
  - 1 coat Pretreatment Wash Primer (MIL-P-15328) 2 coats Primer, Vinyl-Zinc chromate (MIL-P-15930)

The use of these primers is also recommended to reduce corrosion.

- (b) Concrete: Apply the following system of coats to build up the lining.
  - Apply first coat of Thickol Latex, Type WD-6. The concrete surface should be slightly damp, but not wet, just prior to applying this initial coat. Allow first coat to cure.
  - (2) Apply second coat of Thickol Latex, WD-6; then imbed in the wet latex a reinforcing fabric of Osnaburg (26 x 26 thread count, 3.5 to 1.0 ounces per sq. yd.), or preferably glass cloth similar to Owens-Corning ECC-11-111. If Osnaburg is used it should be wetted, but wrung free of excess water. Then, immediately apply the third coat of Thickol Latex over the fabric while it and the second coat are still wet. Allow to dry and cure.
  - (3) Apply fourth and fifth coats of Thiokol-Latex, WD-6 and allow to cure.
  - (4) Apply seven brush coats of Bureau of Ships Formula 113/49, alternating the white and orange colors. Coverage should be a maximum of 150 sq. ft. per gal. per coat of the unthinned material as received conforming to specifications. Twenty-four hours drying should be allowed between coats.

Caution: The latex coats should cure within twenty-four hours. Sometimes, because of low temperatures, or high humidity and rain, this material sets so slowly that runs and sags may occur. Under these conditions resort must be made to artificial drying by warming the interior of the tank; otherwise the lining is likely to prove faulty resulting in early failure. If such a condition does occur, extra coats of latex must be applied under satisfactory drying conditions to remedy the situation.

It is further recommended that Thiokol Latex WD-7 (also sometimes known as SP434) not be used in future lining operations. Thiokol Latex WD-6 is recommended instead because of its slightly superior inertness. It is felt that extra care in surface preparation should compensate for the slightly less adhesion as compared to WD-7 and the greater degree of inertness is very desirable.

3. Contracts: All phases of a lining, or relining operation, including cleaning and surface preparation should be covered by a single contract, thus clearly fixing responsibility.

It is also recommended that technical supervision and assistance be provided wherever lining operations are undertaken. The manufacturers of the lining materials can usually provide qualified technical assistance.

4. <u>Consultation</u>: Whenever the officials of a Naval Activity <u>concerned</u> with a fuel tank lining operation believe the foregoing recommendations are inapplicable, or for some reason cannot be followed, the services of the Naval Research Laboratory or other experienced consultants should be sought to assist in the solution of the problem. The primary tests of the suitability of these recommendations is the adhesion test. If good adhesion is obtainable, the recommended linings should prove satisfactory; otherwise special attention must be given to the lining problem. It is usually feasible to apply several small patches for adhesion tests either prior to, or at an early stage in the preparatory cleaning operations.

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# ILLUSTRATIONS

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Figure 1

Filot-Scale Tank Farm for studying the effects of petroleum fuels on organic coatings used as linings. Each tank contains slightly less than 1000 gal. of fuel.





Figure 3





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Pounds of pull required to remove a 1-inch wide strip of coating\* from steam cleaned concrete block previously soaked in diesel oil. (\*coating= 3 coats of Thickol-Latex topped with 3 coats of Saran F-120).