AZI-LIA EXPANDED RESEARCH PROGRAM

AMERICAN ZINC INSTITUTE, INC.

LEAD INDUSTRIES ASSOCIATION

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Director of Research

August 18, 1960
Ref.: LP-34-60

To All Members of the Technical Steering Committee,
the Chemical Subcommittee and the Lead Pigments
Technical Committee of the Lead Industries Association

Gentlemen:

Herewith is a copy of the Initial Quarterly Report by the Eagle-Picher Company covering the period 1 April 1960 to 30 June 1960 entitled "Anti-Corrosive Properties of Lead Pigments in Water Reducible Ferrous Metal Primers."

It is, of course, too early to expect practical data from this assignment, but I feel sure that you will find the technical discussion to be of particular interest, underlining as it does the complexity of the problem which is being tackled.

Yours sincerely,

/s/ Cook

A. R. Cook

ARC:pk
Enc.

SINCE 1843

EAGLE-PICHER

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Anti-Corrosive Properties of Lead Pigments in Water Reducible Ferrous Metal Primers

Initial Quarterly Report
1 April 1960 to 30 June 1960

to the
Lead Industries Association
Project LP-34-60

Eagle-Picher Company
Chemicals and Metals Division
Research Laboratory
Joplin, Missouri

I SUMMARY

Purpose of LP-34-60

The purpose of this project is to develop information on the use of lead pigments in water reducible ferrous metal primers. The initial phase of the program is a screening study to select for further study those lead compounds that exhibit definite corrosion inhibitive properties under the test conditions. The second phase of the project will study the more effective lead compounds in a wider variety of vehicle and formulation variables.

This Initial Quarterly Report presents a discussion of the background of the project and the reasoning that has led to the experimental program herein discussed. The experimental detail are discussed as they are currently being followed. No experimental results are reported as a complete set of data is not yet available on a unit of the study.

II INTRODUCTION

The traditional drying oil and oleoresinous varnish types of paint and coatings vehicles that have been used for several hundred years have been replaced in many applications in recent years by a large number of widely different vehicles. Many of the coatings formulations based on these new vehicles do not depend upon chemical reactions between pigment and vehicle components to develop the protective film. The new vehicles and their use has necessitated a critical re-examination of all aspects of paint technology and has shifted the emphasis in much research in this field from the pigmentation to the vehicle.

One area in which the pigmentation of the coating remains very important is that of anti-corrosive ferrous metal primers. The modern vehicles do enable effective protection to be achieved with thinner films and smaller concentrations of the rust inhibitive pigments. In some instances the concentration of rust inhibitive pigment is reduced to a level where it may more accurately be described as a chemical additive rather than a pigment in the usual sense.

Project LP-34-60 is intended to cover the study of lead anti-corrosive pigments or addition agents in both automotive and maintenance type finishing systems. The automotive aspects of the project are being attacked first, primarily because of the large potential volume requirements existing for a small number of

products and the opportunity presented for the Lead Industries to develop a market in an industry in which they heretofore have had no significant amount of business.

The economic factors justifying this project as a study of water reducible systems arise from the technical problems associated with the use of organic solvent thinned systems. The traditional paint or coating formulation contains a considerable volume percentage of volatile organic solvent or diluent which play no part in the final film, and is important only during manufacture, storage and application of the paint. Once the paint has been applied the organic thinners present only problems, being responsible for a large variety of film defects as they evaporate from the film, slowing production schedules by their slow rate of evaporation, presenting hazards by their toxic and flammable properties, and making a large contribution to the modern problem of atmospheric pollution. Many efforts have been directed at the replacement of the organic solvents, at least in part, with that cheapest of all solvents or dispersing media--water. The problems involved are many and although much remains to be done sufficient success has been achieved to develop formulation systems that are being used in large scale industrial operations.

A factor which introduces a sense of urgency to this project is the change in design of the engineering of automobile frames and bodies. The recent trend to the "unit body" type of construction has led to an acute corrosion problem. The corrosion of the "unit body" is not simply an appearance problem, as it is with a separate body mounted on a separate frame of relatively heavy members and more open construction, but is a possible source of structural failure with potentially disastrous results. The problem of corrosion here is really a complex involving not only the properties of the protective materials used but their effective application to the interior of essentially blind or closed shapes that furnish the structural strength for the car. The problem is made more severe by the use of calcium chloride, for the removal of ice from city streets and for the control of dust on urban roads, which finds its way into internal cavities and pockets of the "unit body".

The long term success of the "unit body" style of construction for large scale manufacture is somewhat dependent upon the effectiveness with which the corrosion problems can be solved. The possible automotive use of paints containing even a small amount of an inhibitive lead pigment per gallon represents a large volume of business that amply justifies a considerable amount of research.

III EXPERIMENTAL

A. Technical Organization of the Project

Numerous questions arise in the design of a research program that require definite answers and the election of a specific course of action. These questions are of two types; those having to do with the basic reasoning involved and which have a large influence on the value of the results obtained, and those having to do with the experimental detail and which are somewhat less important as long as they are consistently applied and intelligently interpreted. In the present phase of the project the choice of the paint formulation, and the selection of the lead compounds to be tested are of major importance, and their choice is here discussed.

1. The Paint Formulation for the Screening Tests.

a. Introduction.

The modern water-reducible industrial finishes as complex multiphase dispersions, owe their existence in part to the development of very effective emulsifying agents and the technology of their use, which has resulted in a large family of water dispersions of synthetic polymers, and copolymers, and modifications, which are termed "latexes". After application removal of the water phase usually by high temperature (350°F.) drying or baking results in a fusion of the polymer phase and the formation of a continuous film. Equivalent results are being approached by recent developments in air dry maintenance type products. The pigments that are also present in suitably dispersed form will become an integral part of the continuous polymer film during the fusion or drying process.

The possibilities for variation in formulations of this type are almost infinite: The choice of polymer and the method of preparation of the latex. The possibility of modifying the latex with emulsions of alkyds, epoxy resins, or waxes, or various other resins. The use of a large variety of additives to promote pigment wetting and dispersion; protective colloids to prevent the several coexisting disperse phases from reacting either chemically or physically; anti-foaming agents; thickening agents to control consistency; driers and catalysts to promote the desired type of film; preservatives to prevent bacterial growth; and chemical agents to control pH. A successful formulation represents a nice balance of many components and the happy marriage of many component properties.

The possible choices of a suitable latex vehicle and formulation for the screening part of the study were discussed by representatives of the contractor with a number of leading manufacturers of vehicles, of water-dispersible finishes, and several users of water-dispersible finishes. It had been recognized of course that only general information on product formulation would be available to us, but our chief concern was that our screening test formulation would be of such a type that the data obtained by its use would be generally acceptable as being indicative of the rust inhibitive potentialities of the lead pigment tested.

For our test purposes it was desired that the corrosion resistance provided by the film should be related to the content of rust inhibitive pigment, and that in the absence of such pigments the rusting of the test panel should occur rather quickly. The top coats used in some of the actual application systems are therefore omitted to reduce the time required to develop information on the pigmentation of the primer.

b. The Primer Formulation.

The automotive type underbody primer formulation finally adopted after some preliminary experimental work is based on Arlon 304 (Archer-Daniels-Midland) and Dow 566 Latex (Dow Chemical Co.). A first unit of tests made at a 35PVR did not yield results as satisfactory to us as did a lower PVR which is being used in all tests. Because of the sensitivity of these formulations to many factors the actual brand designations of the materials used are given. This is not to imply that other brands giving equivalent results are not available, or that these materials were selected by us on the basis of tests made by us.

Test Formulation

Pigmentation	Lbs./100 Gal.
Iron oxide (C.K. Williams & Co., VVF-B-2093-F)	188.3
Barytes (C.K. Williams & Co., Spermite)	322.8
China Clay (Minerals & Chemicals Corp., ASP-400)	26.8

Each lead test pigment is added in an amount sufficient to introduce 0.3 pounds of lead per gallon, and to compensate for the volume of the added lead compound, the volume of the three pigments of the base formulation is reduced by an amount equal to the volume of the lead compound added. This maintains a constant pigment volume and a constant ratio of the three base pigments.

Ball Mill Vehicle

Ben-A-Gel (National Lead Co.)	4.0
Arlon 304 (Archer-Daniels-Midland, 43% N.V.)	137.7
Manganese Naphthenate, 6% Metal (Nuodex)	8.01
Zirco Drier Catalyst, 6% Metal(Advance Solvents)	8.0
Water, deionized	102.1
Surfynol 104, 20% in ethanol (Air Reduction Sales Co.)	1.2

The slurry of the above components is adjusted before ball milling to a pH of 9.5 with monoethanolamine. It is ball milled for 18 hours or to a Hegman scale minimum reading of 6. Twelve pounds of steel balls are used with a laboratory charge of 798.9 grams or .1875 gals. for the base formulation without the lead replacement.

Let Down

The pigment dispersion from the ball mill is reduced by the addition of the following components to form the basic paint upon which the storage stability tests are made. Some variations of the base formula may require slight additions of water to permit laying down a film of the desired dry film thickness.

	Lbs./Gal.
Dow 566 Latex, 46% N.V.(Dow Chemical Co.)	386.0
Immediately prior to use the latex is adjusted to a pH of 8.5 with NH_4OH.	
Triton X-100, 25% N.V.(Rohm & Haas)	6.6
Carboxymethylcellulose 7HSP, 0.75% (Hercules Powder Co.)	65.0
Total formulation	1256.5

2. Selection of the Lead Pigments and Compounds for Test.

a. Introduction.

The original proposal for this project listed a number of inorganic lead compounds to be tested. This list had been compiled on the basis of various handbooks, chemical reagent catalogs, and reference books which indicated that such compounds existed and could be obtained. The granting of the contract started the operation of accumulating these materials for the test formulations. Several of the items are no longer available, and considerable question exists

as to the identity of the chemical species of the materials available under certain names. A critical review of the inorganic lead compounds to be included in the project is thus very important, if the entire field is to be adequately studied. It was not the original intention that this project would involve the laboratory preparation of any products to be tested. However, it appears that several materials whose testing is very desirable, are not commercially available and as they do not present any special preparation problems they will be prepared.

As far as possible each compound tested will be representative of a class of compounds, or some combination of properties, and together all the compounds will form series in which there is some systematic gradation of properties.

The primary interest is in the prevention of corrosion of a ferrous metal surface by the presence of a coating containing a lead compound which by some mechanism inhibits, prevents, or reduces the corrosion of the metal. It is possible that the lead compound used in the paint formulation is important only as its physical and chemical properties operate to control the availability of lead in the proper concentration and ionic state to prevent corrosion. It is very important to the interpretation of the results of this project that the compounds be so chosen that any beneficial effect due to the lead portion of the compound can be evaluated in the presence of possible interfering, perhaps beneficial, effects of the associated anion.

The lead compounds in the paint film will be present as solids or as organo-metallic complexes with the vehicle. Some of the reactions within the paint film are probably solid state reactions and the ions involved are probably not those that would be expected from the formula as the chemist traditionally writes it. The problem is complicated by the tendency of lead compounds to ionize in several modes dependent on the pH, temperature, concentration, and ionic environment. Ions in solution may give rise to a whole series of hydrolysis products. To be effective as a corrosion inhibitor the ionic products of the lead compounds with water, and the ions that accelerate corrosion that are normally present, must be of such nature as to minimize further attack.

For the purposes of a broad screening study many considerations become of secondary importance, although such items as cost, toxicity, color, color stability, and availability may be deciding factors where several analogous compounds permit a choice. The properties of the selected compounds, however, must fall within

practical limits from the view of the paint maker and user: this eliminates only a few compounds. Chemical handbooks list in the order of 100 inorganic lead compounds of which perhaps 30 may be of interest to test. A literature search reveals a paucity of specific data that can be accepted at face value.

b. Types of Lead Compounds.

It is possible to write chemical formulas for many series of analogous compounds whose properties show dissimilarities not easily explained on the basis of such chemical formulas. Physical studies of many types have revealed complex structures for many compounds whose composition by chemical analysis is in agreement with an apparently simple chemical formula. From the chemical point of view the common lead compounds may be divided into five classes, as follows:

(A) "Normal" divalent lead compounds, as $PbCl_2$ or PbO.

(B) "Normal" tetravalent lead compounds, as $PbCl_4$ or PbO_2.

(C) "Normal" divalent lead compounds of the oxy-acid type of which there are many examples: $Pb(OH)_2$; $Pb(AsO_2)_2$; $Pb(NO_3)_2$; $Pb(ClO_4)_2$; $PbCO_3$; $PbSO_4$; $Pb_3(AsO_3)_2$; $Pb_3(PO_4)_3$ and $Pb_2(PbO_4)$. A few "normal" tetravalent lead compounds of oxy-acids are known but in most cases they exist only as transition compounds.

(D) "Basic" divalent lead compounds which are actually compounds of PbO with the normal compounds of the above types, as in $PbO.PbSO_4$.

(E) "Basic" lead compounds of the Werner coordination ol-type, which are usually written as $2Pb(OH)_2.PbX_2$ where the X may be a variety of anions.

This does not exhaust the possibilities of lead compounds since many known double, and triple salts of lead exist and the field has not been studied thoroughly. Of particular interest are the very large number of insoluble polyanion complex compounds of lead in which one of the anions is the chloride ion. Compounds exist in many percentage compositions in which the chloride ion is associated with one or more of these anions; hydroxyl, carbonate, sulfate, phosphate, arsenite, chromate, molybdate, silicate, antimonate, and others. Many of these occur in nature as minerals.

The lead cation may also be partially replaced with alkali metals, alkali earths, iron, aluminum, zinc and other metal ions One might theorize that under some conditions a chloride ion entering the paint film, either by migration or through a surface scratch, might be immobilized or rendered innocuous by the formation of an insoluble lead-iron-chloro-complex.

c. The Inorganic Lead Compounds.

There are in the order of 100 possible inorganic lead compounds from which the selection of a group for test must be made. Of these 32 have been selected on the basis of their prior use as pigments or their interesting physical, chemical, or structural properties. The available data compiled from various sources with respect to their properties are listed in Tables IA, B, C and D. The actual material used for test may in some instances differ from the formula and structure as given here. Where reasonable doubt exists as to the chemical or physical species present it is planned to use X-ray diffraction to identify those phases present

A few comments on each compound with some discussion of the reasons for their inclusion in the test appears desirable. The order of treatment follows that of the table of properties.

1. Lead Antimonate (Naples Yellow)
Historically this is a very old pigment which has been most used in ceramics.

2. Lead Arsenate
The compound desired here is either that form which is isomorphous with lead ortho-phosphate, or that form comparable to the vanadate

3,4. Lead Carbonates
The normal carbonate (the mineral cerrusite) probably represents the end point to which the basic lead carbonate (the mineral hydrocerrusite) or "White Lead" is converted upon long exposure to air by the absorption of additional carbon dioxide. Normal carbonate upon heating forms a series of anhydrous basic carbonates that are quite different in physical properties from "White Lead" which contains a hydroxy compound of the Werner ol-type coordination The drying temperatures used in this study are probably not high enough to cause any significant thermal decomposition of normal carbonate.

5,6. Lead Chlorides

These compounds are of interest because of the very large number of complexes in which they may enter. The chloride ion is very active in promoting corrosion under many conditions and it might be possible to develop some protection by providing suitable complexing compounds. The normal chloride is the mineral cotunnite, and a number of anhydrous basic chloride minerals of varying basicity are known and include Penfieldite, Matlockite, Mendipite, and Lorettside. The basic compound $7PbO.PbCl_2$ is a yellow pigment that has been known variously as Cassel's Yellow, Turner's Yellow, Veronese Yellow, and Mineral Yellow. Hydrated basic chlorides also exist as minerals and include Fiedlerite and Laurionite. Related to the hydrated forms, and perhaps also to the basic nitrate, is Pattinson's White Lead which has the formula Pb(OH)Cl. Of these it is planned to test only the normal and the most basic compound.

More complex chloride compounds exist as minerals in which a part of the lead cation is replaced by iron, zinc, alkali earths, etc., and the anion portion may contain arsenates, phosphates, silicates, titanates, carbonates, sulfates, etc., in addition to the chloride ion. Some of these may be formed in the paint film from the products of corrosion. Whether or not they inhibit further corrosion will depend entirely upon their properties.

7,8. Lead Chromates

The known corrosion inhibitive properties of the chromate ion make it important to test these in both normal and basic forms. There is perhaps some relation between their behavior and that of such salts as lead molybdate and tungstate.

9. Lead Cyanamid

This compound has been highly touted in Europe as an excellent rust inhibitive pigment.

10. Lead Cyanate

This compound may have anti-corrosive properties.

11. Lead Dioxide.

This compound is of interest because it is one of the few tetravalent lead compounds reasonably stable in water, and because of its oxidizing properties

12. Lead Hydroxide

The hydroxide does not exist in the dry form but loses some water to form a hydrated oxide, the "lead hydrate" of commerce which has

a composition approximately represented by the formula 5PbO. $2H_2O$. If basicity is an important aspect of rust inhibition this compound represents the practical limit to which basicity can be developed by the use of a lead compound. Part of the potential basicity of lead hydrate may be lost by reactions taking place during the preperation and application of the paint and paint film.

13. Lead Molybdate

The behavior of this compound is to be compared with lead chromate.

14,15. Lead Nitrates

In alkaline solutions the very soluble lead nitrate is converted to the insoluble basic lead nitrate. The ease of reduction of the nitrate ion offers interesting possibilities for reactions.

16. Basic lead nitrite

It appears from a review of the literature that the existence of the normal salt is doubtful. However, many complexes of the nitrite ion with the nitrate and hydroxyl ion are described. For our purposes a preparation yielding the relatively simple dibasic nitrite $2PbO.PbNO_2$ has been selected. Compounds containing the nitrite ion are of special interest because of their use in many anti-corrosive compounds.

17. Lead Oxide

Lead monoxide offers the highest lead content and the highest density of available compounds. It should contribute some basicity, is reactive with many compounds and although relatively insoluble alone it is quite soluble in the presence of many other compounds. It exists in two crystalline forms with the orthorhombic form coverting to the tetragonal form upon ball milling in the presence of water.

18. Lead Ortho-Phosphate. (Pyromorphite)

The phosphate coatings applied to iron surfaces for rust inhibitive purposes make tests of the phosphates as pigments very desireble.

19. Lead Phosphite, Dibasic. (Diphos*)

As a phosphorous compound of interesting chemical properties, and a useful polyvinyl chloride stabilizer this compound is included.

20,21. Lead, and Calcium, Plumbates, (Red Lead and analogue) These compounds represent the salts of the hypothetical tetrabasic plumbic acid, H_4PbO_4 . These two compounds by comparison may throw light on whether the rust inhibition of red lead is

*Trademark name of the National Lead Company

related to the properties of the divalent lead cation or the tetravalent plumbate anion.

22,23. Lead Silicates
Previous work has demonstrated rust inhibitive properties of the basic silicate. Silicates are of interest on the basis of their formation of many complex minerals, and their use as polyvinyl chloride stabilizers. Both the normal and basic compounds are found as minerals, Alamosite, and Barysilite.

24. Lead Silicochromate
This pigment combines lead with both the silicate and chromate ions in a single complex.

25. Lead Sulfamate
A very soluble salt of lead used in some lead plating baths. The presence of the amine group in the sulfamate ion may permit some interesting reactions.

26,27. Lead Sulfates (Anglesite, and Lanarkite)
The basic sulfate type of white lead has been used as a pigment for many years.

28. Lead Sulfide (Galena)
Naturally occuring lead sulfide, as galena, has been deposited in many places from sea (salt) water, as evidenced by the droplets of salt water to be observed in many freshly fractured galena crystals. It will be of interest to observe the behavior of very finely divided (precipitated) lead sulfide in a paint film in a salt spray atmosphere. Lead sulfide has been a minor component of "Basic Sulfate Blue Lead" pigment for many years.

29. Lead Sulfite
Lead sulfite should show about the same relation to lead sulfate as lead nitrite does to lead nitrate, but at a much lower level of solubility. Its behavior with respect to its environment should be more sensitive than the sulfate.

30. Lead meta-Titanate
This cream colored pigment has very interesting physical properties but has not been widely used as a paint pigment.

31. Lead Tungstate. (Two forms-Stolzite, Raspite)
This is of interest in comparison with the chromate and molybdate.

32. Lead meta-Vanadate
This compound of lead with a VB element is the analogue of lead nitrate which is a VA element compound.

d. The Organic Lead Compounds.

The lead organic compounds to be tested will be discussed in a later report on the basis of those compounds that are available for test.

3. The Testing Program.

a. Paint Properties.

The properties of the paint, as distinguished from the properties of the paint films that they produce, that are of the most interest in this project are those having to do with stability during use and storage. Industrial products are seldom called upon to have a long shelf life and a six month storage period would probably be a reasonable limit. However, during use paint products may be subjected to conditions such as temperature or aeration that could cause trouble in a paint with a short storage life. Reactions occuring during storage, or during use, that result in gelling, loss of dispersion, or large changes in viscosity could prevent the use of an otherwise effective product.

For the screening program can stability tests will be run at both room temperature and under accelerated conditions (120°F.) and will be examined after 1, and 2 weeks for settling and at 3 weeks for settling, pH change, and viscosity or consistency changes. The time for the room temperature tests will be extended as necessary. Viscosity determinations are made using a Ford cup with #4 orifice at 78°F.

b. Film Properties.

(A) Preparation of the Test Films.

Test Panels. Phosphate treated steel panels (Parker Rust Proof Bonderite #100) were adopted as suitable test surfaces. The surfaces of these panels have not been polished. The use of 4" x 12" panels makes it possible to shear each panel into 3 test areas and thus to obtain several tests from each individual film application. The diagram below shows how the tests are correlated on each panel, and their abbreviated code:

Reference	Physical Tests PA	Physical Tests PB
Salt Spray SA	Salt Spray SB	Humidity Cabinet HB
Humidity Cabinet HA	Acc. Weathering WA	Acc. Weathering WB

Identification of the test pieces is done by use of a marking brush on their rear.

Protection of edges and rear of the test pieces in the salt spray and humidity cabinet is obtained by dipping the edges, and brushing the backs with a coating of wax. The wax used is a mixture of

40% Refined Crystalline Paraffine Wax, M.P. 135-140°F., and
60% Refined Amorphous Paraffine Wax, Mobil #2305,

both obtained from the Socony-Mobil Oil Co. The test pieces for the accelerated weathering machine were coated with a lacquer. The reference and physical testing test pieces were uncoated.

Application of Test Paints. Water reducible industrial finishes are applied by dipping or spraying but neither method is very satisfactory for the precise application of thin films of uniform and known thickness from paints of variable composition. Satisfactory results have been obtained by using a wire wound rod type of doctor blade*. Dry film thickness of 0.55 mils is desired and the films obtained are within a plus or minus 10%.

The control of the dry film thickness is obtained by adjusting the consistency of the paint with water until measurements of a dry film show the proper value. Reproducible film thicknesses can then be laid down. The checking of the dry film thickness is done with a magnetic thickness guage (American Instrument Co., Magne-Gage). Checks made on film thickness using a standard machinists

*We used a #20 RDS Laboratory Coating Rod obtained from R. D. Specialities, P.O. Box 397, Webster, N. Y.

micrometer indicate that the phosphate coating has an appreciable thickness for which a correction must be applied to the magnetic guage reading.

Test paint age at the time of initial application is approximately 1 week, which is sufficient to eliminate any whose storage life is very short. After the panels are coated they are permitted to air dry until the surface appears dry, and are then baked 25 minutes at 350°F. (177°C.). In most instances the test panels will be at least a week old before exposure tests are started.

(B) Testing of the Test Films

Many of the physical properties of a paint film may be significantly altered by slight variations in the percentage composition with respect to some component, or by replacement of one item by some modification of it. In this project we are interested in the general paint film properties primarily to assure that the paint being tested is in fact a useful paint. With this assured the anti-corrosive properties exhibited by the paint are meaningful when related to the lead pigment being tested. The tests fall into two categories. Physical Tests, and Corrosion Resistance Tests.

(1) Physical Tests. These tests are applied to the test pieces immediately after their preparation, and after 500 hours of exposure in the accelerated weathering test. It is not expected that the accelerated weathering treatment will cause any apparent film failure, but it is expected to produce enough film aging to show up perhaps as poorer adhesion, or increased brittleness with poorer impact resistance. The accelerated weathering is provided by an X-1A National Carbon Co. machine whose light source uses Sunshine cored carbons (13mm. below and 22mm. above).

Hardness is measured by a Sward Rocker. Impact Resistance is measured by a Gardner. Variable Impact Tester using a 5/8" spherical die. Bend Flexibility is determined by use of a cylindrical mandrels of several sizes.

(2) Corrosion Resistance Tests.

Salt Spray. The primary testing tool in this project is the salt spray which is operated as described in ASTM Specification B117-57T for Salt Spray (Fog) Testing. The test pieces are mounted at 15° and are exposed in the cabinet for 250 hours with inspections at intervals. The central portions of all test pieces contain an X-scratch to the bare metal to enable rust inhibition, and rust creepage under the film to be observed and evaluated.

Positive Condensation Humidity. In this test the test pieces are supported with their backs against cold plates producing positive condensation on their faces. The atmosphere approaches 100% humidity at 100°F ± 2°. These conditions promote blistering as well as rusting. The test pieces are subjected to these conditions for 750 hours.

IV WORK IN PROGRESS

The next report will be based on the data available from the completion of several series of tests.

Table I
The Inorganic Lead Compounds
A. Names and Sources

No.	Chemical Name	Mineral or Pigment Name	Source	Brand Name Code or Grade
	Lead			
1.	Antimonate	Naples Yellow	Amend	
2.	Arsenate		Amend	
3.	Carbonate,n	Cerrusite		
4.	Carbonate,b	White Lead	E-P	A
5.	Chloride,n	Cotunnite		
6.	Chloride,b	Cassel's Yellow	Lab.	
7.	Chromate,n	Chrome Yellow	Reichhold	RCI 1298
8.	Chromate,b	Chrome Orange	Reichhold	RCI 2020
9.	Cyanamid			
10.	Cyanate		Lab.	
11.	Dioxide	Plattnerite	E-P	
12.	Hydroxide	Lead hydrate	Amend	
13.	Molybdate	Wulfenite	Lab	
14.	Nitrate,n			
15.	Nitrate,b		Lab	
16.	Nitrite,b		Lab	
17.	Oxide	Litharge	E-P	
18.	Phosphate,o	Pyromorphite	Amend	
19.	Phosphite,b		National Lead "Diphos"*	
20.	Plumbate	Red Lead	E-P	97%
21.	Calcium Plumbate		Gamichon	
22.	Lead Silicate,n	Alamosite	Amend	
23.	Silicate,b	Barysilite	E-P	E-P #202
24.	Silico-chromate		E-P	E-P M-143 "Permox"**
25.	Sulfamate		Lab	
26.	Sulfate,n	Anglesite	Bikers	AR
27.	Sulfate,b	Lanarkite	E-P	E-P #41
28.	Sulfide	Galena	Amend	
29.	Sulfite,n		Amend	
30.	Titanate,n		TMA	
31.	Tungstate	Stolzite	Lab	
32.	Vanadate,n	Dechenite	Lab	

* Trademark name of the National Lead Co.
**Trademark name of the Eagle-Picher Co.

I The Inorganic Lead Compounds

B. Chemical Formula Data

No.	Chemical Formula	Formula Weight	Per Cent Pb	Sp.Gr.	Bulking Gal./lb.
1.	$Pb_3(SbO_4)_2$	993.15	62.59		
2.	$PbHAsO_4$	347.13	60.70	5.79	
3.	$PbCO_3$	267.22	77.54	6.6	0.01817
4.	$2Pb(OH)_2.PbCO_3$	775.67	80.3	6.7	.01790
5.	$PbCl_2$	278.12	74.50	5.85	.02050
6.	$7PbO.PbCl_2$	1840.59	90.07		
7.	$PbCrO_4$	323.22	64.11	6.3	.01904
8.	$PbO.PbCrO_4$	546.43	75.84	6.87	.01747
9.	$PbCH_2$	247.23	83.81		
10.	$Pb(CNO)_2$	291.25	71.14		
11.	PbO_2	239.21	86.62		
12.	$3PbO.2H_2O$	1152.08	89.93		
13.	$PbMoO_4$	367.16	56.43	6.8	.01764
14.	$Pb(NO_3)_2$	331.23	62.56	4.53	.02648
15.	$Pb(OH)NO_3$	286.22	72.40		
16.	$2PbO.Pb(NO_2)_2$	745.64	83.37		
17.	PbO	223.21	92.83	9.35	.01283
18.	$Pb_3(PO_4)_2$	811.67	76.59	7.3	.01643
19.	$2PbO.PbHPO_3.\frac{1}{2}H_2O$	742.63	83.71	6.94	.01728
20.	$Pb_2(PbO_4)$	685.63	90.66	9.1	.01318
21.	$Ca_2(PbO_4)$	351.37	58.97	5.71	.02101
22.	$PbSiO_3$	283.27	73.15	6.49	.01848
23.	$PbO.2PbSiO_3$	789.81	78.7	6.45	.01860
24.	---	---	42.7	4.09	.0294
25.	$Pb(NH_2SO_3)_2$	399.39	51.88		
26.	$PbSO_4$	303.27	68.32	6.2	.01935
27.	$PbO.PbSO_4$	526.48	78.71	6.4	.01874
28.	PbS	239.27	80.60	7.5	.01599
29.	$PbSO_3$	287.27	72.13		
30.	$PbTiO_3$	303.11	68.36	7.52	.01395
31.	$PbWO_4$	455.13	45.53	8.23	.01457
32.	$Pb(VO_3)2$	405.11	51.15	5.8	.02068

Table I
The Inorganic Lead Compounds
C. Crystal Structure Data

No.	Crystal Class	Unit Cell Dimensions a_o	b_o	c_o		Symmetry Symbol
1.						
2.						
3.	Orthorhombic	8.468	6.146	5.166	-	V_h^{16}
4.	Hexagonal					
5.	Orthorhombic	4.496	7.667	9.153	-	V_h^{16}
6.						
7.	Monoclinic	7.10	7.40	6.80	102.45°	C_{2h}^5
8.						
9.						
10.						
11.						
12.						
13.	Tetragonal	5.41	-	12.08	-	C_{4h}^6
14.	Cubic	7.84	-	-	-	T_h^6
15.						
16.						
17.	Orthorhombic	5.50	4.72	5.88	-	V_h^{19}
18.	Hexagonal	5.98	-	20.50	-	D_{3d}^5
19.						
20.	Tetragonal	8.788	-	6.351	-	D_{4h}^{13}
21.						
22.	Monoclinic	11.28	7.03	13.06	120.00°	
23.						
24.						
25.						
26.	Orthorhombic	8.480	5.398	6.958	-	V_h^{16}
27.	Monoclinic	13.73	5.68	7.07	116.217	
28.	Cubic					
29.						
30.	Tetragonal	3.894	-	4.140	-	D_{2h}^1 (tr to cubic @about 500°C)
31.	Tetragonal	5.44	-	12.01	-	C_{4h}^6
32.						

Table I
The Inorganic Lead Compounds
D. Solubility and Other Data

No.	Solubility in grams/ 100 ml			Melting Point C°.
	Cold Water	Hot Water	Ammonia	
1.	i	-	-	
2.	i			d 280
3.	0.0001	d	i	d 315
4.	i	i	-	d 400
5.	0.99	3.3%	s	501
6.	i	-	-	
7.	0.000 006	i	i	844
8.				920
9.				
10.	s	-	-	d
11.	i	i	-	d 290
12.	0.015	sl.s	-	d 145
13.	i	-	-	
14.	56.5	127.	-	d 470
15.				
16.				
17.	0.0017	-	-	tr. 495
18.	0.000 014	i	-	1014
19.				
20.	i	i	-	d 500
21.	i	d	-	d
22.	i	-	-	766
23.				977
24.				
25.				
26.	0.0042	0.0056	s salts	d 1000
27.	0.0044	v sl s	-	
28.	0.000 09	-	-	1114
29.	i	i	-	
30.	i	i	-	
31.	0.03	-	-	1123
32.	sl s	-	-	

Abbreviations used in Tables

Amend - - - Amend Drug & Chemical Co., Inc. 117-119 E. 24th St., New York 10, N. Y.

AR- - - - - Analytical Reagent grade

b - - - - - basic

d - - - - - decomposes

Comichon- - Comichon Corretto et Cie, Rieux (Oise), France

i - - - - - essentially insoluble

Lab - - - - compound prepared in laboratory of contractor

m - - - - - meta

n - - - - - normal

o - - - - - ortho

s - - - - - soluble

sl- - - - - slightly

TMA - - - - Titanium Metal Alloys Div. of National Lead Company

tr- - - - - transition

v - - - - - very