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Acerbic Approach to Problem Of Old Lead-Base Paints — Feasibility Study

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Painted surfaces can be rendered unappealing to the taste by treatment with solutions of acerbic compounds such as sucrose octa-acetate and piperine. The wide-scale application of such solutions to reduce the hazard of lead poisoning of children in poorly-maintained dwellings is shown to be feasible from both technical and economic viewpoints.

KEY WORDS: Ingestion; Acerbic substances; Sucrose octa-acetate; Piperine; Lead-base paints.

INTRODUCTION

IT is a matter of record that repeated ingestion of old lead-base paints by children may result in lead poisoning. In many older residences currently utilized as multi-family units, surfaces carry heavy accumulations of such old paints. The condition of these surfaces is such that loose flakes or chips of paint are readily available to children. These old lead-base paints, moreover, seem to have a pleasant or sweet taste to children, which further increases the chances of copious ingestion.††

While restriction of the lead content of new coatings can minimize the hazard of future lead poisoning, such measures do nothing to alleviate the danger presented by lead-base paints applied earlier. Neither can one expect prompt elimination of the hazard by removal of the old paint or by isolation of these surfaces from children, since currently available methods of removal or isolation are expensive and usually require vacating the residential area, at least temporarily.

If it were possible, however, to render the old paint unpalatable, the probability of ingestion by children could be greatly reduced. The trustees of the Paint Research Institute, desiring to define more precisely the potential value of this approach to a remedial program, sponsored the study reported here.

GENERAL SPECIFICATIONS

As a point of departure, we determined that if successful large-scale treatment of deteriorating painted surfaces were to be possible the following properties were desirable in the applied acerbic material:

(1) Fragments or chips of the treated surfaces introduced into the mouth should produce an intensely acerbic (unpalatable) sensation in a high percentage of the population.

(2) The acerbic potential of the treated surfaces should be retained for extended periods of normal use and cleaning.

(3) No substances employed should present appreciable toxicological or flammability hazard to applicators or residents.

(4) The treatment should not require the removal of furniture or fixtures; reoccupancy should be possible within a short time after the treatment is completed.

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†† A recently reported incident describes a child taking a bag of lead base paint chips to school and passing them around to his friends as he would candy.

(5) Application of the material should be possible by either trained crews or by unskilled residents.

(6) Treatment should not interfere with or unduly complicate subsequent refinishing or redecorating.

(7) The cost of materials and application labor should be small.

SURVEY OF ACERBIC SUBSTANCES

Distasteful, oral sensations may be classified for present purposes as: bitter, peppery (hot, burning), sour (acid), or astringent (puckering).

Sensations involving primarily olfactory responses have not been included here because continued exposure in residential areas would tend to render occupants insensitive to the repelling agent.

Bitter substances generally produce the most intense acerbic reactions. Some chemicals produce a bitter taste at concentrations near one part per million.[1] Unfortunately, however, the sensitivity to bitter oral sensations produced by synthetic chemicals is weak or entirely absent in up to 30% of humans for some compounds.[2] Sensitivity to bitterness is thought to be an inherited trait. Bitterness alone, therefore, should not be depended upon as the repelling force for all persons.

Peppery taste, if intense, may be acerbic, but continued exposure to heavily seasoned foods may increase tolerance or even produce a desire for the sensation. Some children of Spanish-American or East Indian extraction, for example, enjoy eating hot peppers which are repugnant to others.

Sour and astringent principles may not be promising as acerbic agents for this use inasmuch as most are highly water-soluble (e.g., zinc chloride and alum). Furthermore, many (e.g., tannic and gallic acid derivatives) decompose rather rapidly by chemical or photochemical reaction.

We believe that based on the factors detailed above, the primary materials to consider as acerbating agents in treating paint films should be some combination of bitter and peppery principles.

It appears, therefore, that we should utilize a bitter principle as the primary source of repugnance and add a peppery principle to repel those children who are immune to bitterness. *Table* 1 lists some of the candidate substances in these two categories, together with some of their pertinent properties.

Sucrose octa-acetate possesses an intensely bitter taste at low concentrations. It exhibits relatively low toxicity[3] and is commercially available at a reasonable price. For these reasons, sucrose octa-acetate was chosen as the bitter principle for our initial experimentation.

The choice of a peppery principle was more difficult. Crude extracts from red peppers are highly colored (red to orange) and hence treatment may cause the appearance of treated surfaces and surfaces inadvertently contacted to change. Extracts from black pepper or from white peppers, on the other hand, are lighter in color or colorless. Piperine, a constituent of black pepper available as a purified, crystalline chemical, was chosen for the initial experiments. Extraction of ground black and white pepper indicated that less expensive peppery principles may be obtained from these sources.

Table 1

Bitter Principles	
Bacitracin	Very bitter taste; virtually nontoxic except by parenteral administration and in presence of preexisting renal disease. Soluble in water.[a]
Brucine sulfate	Bitter. Moderately soluble in water. Used for denaturing alcohol. Physiological reactions, less than strychnine.[a]
Quassin	Intensely bitter. Colorless. Slightly soluble in water. Used for denaturing alcohol.[a]
Sucrose octa acetate	Intensely bitter. Colorless. One part soluble in 1100 parts water. Very soluble in acetone, methyl acetate, toluene and chlorinated hydrocarbons. Used as denaturant for alcohol.[a,b]
Diethyl phthalate	Intensely bitter principle approved for denaturing alcohol. A liquid; must be stabilized in a resin to form a solid film.[a]
Peppery Principles	
Capsaicine	Burning taste detectable at one part in 100 000. Insoluble in cold water. Freely soluble in some organic solvents.[a]
Capsicum	Cayenne pepper extract. Bright red.
Piperine	Constituent of black pepper. Tasteless at first but burning after taste. Almost insoluble in water. Soluble in some organic solvents. Used to impart pungent taste to brandy.[c]

(a) The Merck Index.
(b) Handbook of Chemistry and Physics.
(c) Private communications.

EXPERIMENTAL

Samples of sucrose octa-acetate,* and piperine† were dissolved in methylene chloride. Thin films of these solutions were applied and dried on the surface of varnish-coated paper board, prepared to simulate the surface of old paint. Small (1 sq cm) wafers of treated board produced pronounced acerbic sensations in the mouth of test individuals at solution concentrations of less than 1% for piperine and about 2% for sucrose octa-acetate.

More extensive testing was performed with the following solution:

Acerbic Solvent Test Solution
10% (weight) sucrose octa-acetate,
1% piperine
89% methylene chloride

* Custom Haviland Products Co., Grand Rapids, Mich.
† Custom Aldrich Chemical Co., Milwaukee, Wis.

Of the over 75 adults who were given 1 sq cm wafers of board treated with this solution to chew, all but one reported a pronounced acerbic reaction within 15-25 seconds. The offensive taste generally increased in intensity during the first minute and persisted for 10-30 minutes after gustation. Smoking or eating highly-flavored foods prior to testing appeared to partially mask the reported acerbic response, but in no case was it eliminated. The presence in the mouth of monosodium glutamate during testing did not appear to affect the acerbic sensations of the few persons tested.

It has been reported that children possess more gustatory receptors on cheeks and tongue surfaces than adults.[4] It may be possible, therefore, to obtain the desired repugnancy to children with lower concentrations of acerbic principles than those used in these experiments.

To test the durability of the acerbic films, treated, varnished surfaces were washed repeatedly with water, and household detergent in water with no apparent diminution of the acerbic property. Moreover, films exposed to bright sunlight for 11 days did not change in appearance or become more palatable. Repeated scouring with abrasive household cleansers did reduce acerbic reaction, but resistance to removal by scrubbing appeared to increase with aging of the treated surface.

The persistence of the acerbic treatment described above can be explained by the fact that sucrose octa-acetate solutions, on drying, form clear, flexible films possessing surprisingly low water sensitivity. It was felt that the physical properties of the acerbating films, such as abrasion resistance, should be improved by the addition of a small amount of an inexpensive, compatible resin. This was confirmed experimentally by adding 5% (by weight) of a petroleum resin[5] to a test solution. Careful adjustment of the type and content resin would be necessary to optimize physical properties without appreciably diminishing the acerbic property.

Sucrose octa-acetate has been known and used as a plasticizer for many polymer systems. It should, therefore, interact strongly with the binders of old paint films. The methylene chloride solvent is also readily absorbed by paint films, so it is likely that appreciable amounts of acerbic principles can be absorbed into the old paint surfaces during the drying of the treating solution. The degree of absorption may be increased by the use of some less volatile solvent, such as trichloroethylene. This would, however, increase the potential toxicological hazard during application and drying (TLV) of methylene chloride = 500 ppm; trichloroethylene = 100 ppm).[6]

The test solution (without fortifying resin) was sprayed so that the fringe of the spray pattern (the "overspray") fell on typical, nonpainted household surfaces. No damage or appreciable change in appearance was evident on wallpaper, fabric, leather, or brick surfaces exposed to the light "overspray." A slight increase in gloss was noted when impingement approached a full treatment film. Adhesion of the acerbic film to glass or porcelain surfaces is poor and overspray could be removed from windows and porcelain fixtures by normal washing procedures.

Application of the acerbic materials as an emulsion in water may have economic and other advantages over simple solvent systems. The general feasibility of emulsion preparation was checked by applying an emulsion of the following formula:

Emulsifiable Acerbic Solution

20%, (by weight) sucrose octa-acetate
2% methylene chloride extract from ground black pepper
0.5% emulsifying agent
77.5% methylene chloride

This solution was stirred vigorously into an equal volume of water (by volume). The resulting emulsion applied by spraying or by brushing dried in a few minutes, leaving the treated surface strongly acerbic. Addition of the emulsifier increased the water sensitivity of the dried treatment film slightly.

The raw materials employed in the emulsified treatment are estimated to cost less than $0.01 per square foot of treated surface.

METHODS OF APPLICATION

The acerbic solutions described above could probably be applied most efficiently and economically by spraying, from either portable spray equipment (for trained application crews) or from aerosol spray cans (for limited, topical application by householders).

Spray tests were first run utilizing portable air-atomizing spray equipment. Proper choice of spraying tip and air pressure reduced the "overspray" to a satisfactory level while surfaces to be treated were covered with an adequate wetting film. Excessive application, resulting in "runs" was avoided by applying the material slightly less than that required for a fully continuous coating. No problems of equipment operation or maintenance were observed. Equipment cleanup was accomplished satisfactorily with a mixture of chlorinated hydrocarbon (e.g., trichloroethylene) and mineral spirits.

Airborne particles of acerbic material were moderately offensive to the applicator and nearby observers, causing a very unpleasant taste in the mouth and some sneezing. Use of a respirator designed to trap particulate matter eliminated this nuisance for the operator. For large scale operations, applicators will apparently have to wear respirators which would

[5] Piccopale 8 (the Pennsylvania Industrial Chemical Corp., Clairton, Pa.

[6] Amsco® SHT-75, Armour Industrial Chemicals Co., Chicago, Ill.

screen out both solvent vapor and particulates, and other persons will have to be kept from the area until the air is free from acerbic material and solvent vapors.

Spraying of the emulsified acerbic formulation produced less effect upon the operator than did the solvent solution. The use of respirators for large-scale work still seems advisable.

A portion of the acerbic solvent solution was packaged in aerosol cans using propane isobutane as the propellant.* Contents of the aerosol can was 50% (by weight) acerbic solvent solution and 50% propellant. No problems of filling or use were evident except that, during spraying, inhalation of airborne droplets of the solution produced very intense acerbic reaction almost instantaneously in the mouth and nasal passages of the applicator and persons in adjacent rooms. Even brief exposure caused coughing, sneezing and gagging. Detectable amounts of acerbic material in the air persisted for about 20 minutes. It was concluded, therefore, that aerosol application of this type of solution without adequate respiratory protection would not be practical.

Application by airless spraying equipment was not attempted, because the experience reported above showed that formation of very finely atomized droplets is undesirable.

Application by swabbing, brushing or rolling would eliminate the problem of lingering airborne droplets, but applying the methylene chloride solvent solution by these methods proved to be difficult due to the very high rate of evaporation and the resulting inability of laying down a reasonably even coating.

Replacing part of the methylene chloride with a less volatile solvent blend might reduce the evaporation rate sufficiently to permit application with reasonable control by swabbing or brushing. For example, sucrose octa-acetate dissolved in a blend of 20% (by weight) mineral spirits, 13% dipentene, and 65% methylene chloride was easily applied by swabbing with a cellulose sponge.

TOXICOLOGICAL AND LIABILITY HAZARDS

No effort was made during this study to evaluate the toxicological hazard of ingredients employed in the experimental formulations except to determine from standard sources that none of them was cited as being acutely toxic. When a final formulation is proposed for large-scale testing, the toxicity of both the volatile solvent fraction and the deposited acerbic film must be determined precisely.

Similarly, legal questions, such as patent position and possible liability for either personal or property damage, will have to be considered. It is our understanding that a British patent has been issued dealing with some aspects of this work and that additional patent applications have been made in the U. S.

CONCLUSIONS

The investigations reported here confirm the general feasibility of treating old lead-base painted surfaces to render them unpalatable to a high percentage of children. Such treatment should appreciably reduce the probability of ingestion of such materials and the accompanying danger of lead poisoning. A strongly acerbic solution can be applied economically to the potentially hazardous surfaces of a residence without stripping the living area of furniture or fixtures. Reoccupancy will be possible within a short time following the treatment. Bitter and peppery principles in combination appear to be best suited for this use. Specifically, a combination of sucrose octa-acetate and black pepper extractions are proposed. When dissolved in organic solvents, these substances may be applied directly or as an emulsion of a more concentrated solvent solution and water.

Application by spraying is attractive from the economic standpoint, but formation of very fine, airborne particles of the acerbic solution must be minimized by careful selection of spraying equipment and operating conditions.

Application by swabbing, rolling or brushing may also be feasible when treatment of small areas by non-trained personnel is desired.

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(1) Mancrieff, R. W., "The Chemical Senses," 3rd Ed., p. 287 (1967).
(2) *Ibid.*, p. 273.
(3) Sax, "Dangerous Properties of Industrial Materials."
(4) Mancrieff, R. W., *op cit.*, p. 245.
(5) "Threshold Limit Values of Airborne Contaminants," American Congress of Industrial Hygienists.

* Courtesy The Sherwin-Williams Co., Cleveland, Ohio.

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TITANOX, ANTIMONY OXIDE, AND LITHOPONE

A paper presented at the request of the International Association of Master House Painters and Decorators of the United States and Canada, at the February, 1924, Atlantic City Convention.

NEW EXPOSURE TESTS ON TITANOX PAINTS AND ON LITHOPONE PAINTS

NEW EXPOSURE TESTS TO DETERMINE THE EFFECT OF ALUMINUM POWDER IN WHITE PAINTS AND THE EFFECT OF ALUMINUM POWDER PAINTS AS WOOD PRIMERS

By Henry A. Gardner

TITANOX, ANTIMONY OXIDE, AND LITHOPONE*

By H. A. Gardner

White lead, both basic carbonate and basic sulphate, with zinc oxide, have constituted the white base of paints for many years. Lithopone was then developed, and its commercial production in America dates back to 1903, when 100 tons were produced. Other substances have been proposed as supplementary pigments, and among these are oxides of bismuth, tin, antimony, and titanium. The former two have not been used commercially in paints because of their high price. The latter two, however, have recently come into quite wide usage.

Titanox.—Titanox pigment is made from titanium ores known as ilmenite and rutile, a large quantity of iron being present in the former. Ilmenite is found throughout the world, having a wider distribution than any other metallic ore from which white opaque pigments are made. Surface mining of the sands of Florida beaches yields much of the ore that is used in America, over a million tons probably being available for this purpose. The ilmenite concentrates are mixed with fluxes and acid and put through an electric furnace to separate the constituents. The crude concentrate of titanium oxide is further purified by acid treatment, then dissolved in stronger acid and the solution precipitated upon a base of finely divided blanc fixe. The composite pigment thus produced is dried, calcined, and ground; very marked increase in hiding power resulting from proper calcination. The pigment thus produced is remarkably fine in subdivision for a precipitated pigment. Its color is much whiter than any white lead the writer has examined, but not as dazzlingly brilliant as French process zinc oxide or the better grades of lithopone. Its hiding power is much greater than that of any other known white pigment, being twice that of white leads of good brightness. Its remarkable hiding power has probably been responsible for its rapid introduction, inasmuch as its application will often hide dark surfaces in one coat, thus saving the large labor charge of applying two coats of paint.

Its inertness toward varnish liquids of high acidity and colloidal properties has also made it of value in the production of compositions that can be kept in packages without danger of livering or thickening.

* This subject was assigned to the writer for presentation before the International Association of Master Painters and Decorators, at the Convention at Atlantic City, February 5-9, 1924.

Its oil absorption is slightly lower than that of lithopone, a mixture of good brushing qualities consisting of 50 parts of pigment and 10 parts of liquid. Such a mixture has the appearance of being somewhat heavy in body. As a matter of fact, however, it brushes out easily to a smooth film. This tendency to appear thick has probably misled some users to thin such paints so that they will pour easily like ordinary types of mixed paint. When thus thinned, however, it produces a film, because of its great spreading properties, that is rather thin and less durable than one of the consistency referred to above. The correct proportions for most prepared Titanox paints (60-40) might also be obtained by thinning 100 pounds of certain paste Titanox paints with a total of about 3 gallons of oil and thinners for final coat work. For first coat work, a slightly larger quantity of liquid could be used. These proportions will, however, vary with the formula of the paste.

Titanox (100 per cent) in oil without drier dries very slowly to a soft film. The fairly liberal use of drier containing lead, manganese, and cobalt, overcomes this lack of drying in the pigment, while the use for exterior work of from 30 to 50 per cent of zinc oxide causes the formation of a firm, dust-resisting film. The use of a small amount of boiled oil may also be considered advantageous.

When exposed to sunlight, Titanox appears to be entirely stable. In atmospheres of hydrogen sulphide it remains unchanged, and for this reason forms the base of paints that are used in many factory districts where hydrogen sulphide is evolved from industrial processes, or in mining regions and oil districts where hydrogen sulphide is encountered in quantity. Its use as a light colored coating upon oil storage tanks in the oil regions has been very successful. Tests made by the writer at Passaic, N. J., and in Texas oil regions, where abnormally high percentages of hydrogen sulphide are present, showed very white surfaces for over two years under these most trying conditions.

The first sample of Titanox pigment that ever came to the attention of the writer was delivered to him early in July, 1921. It was contained in a small vial and appeared to be of a pronounced cream color. Laboratory tests showed such interesting properties that a request was made that a few hundred pounds be made up and submitted for practical test. This was done by Mr. L. E. Barton, the inventor, with a small experimental furnace equipment. Within three months (October, 1921) the writer had prepared paints with it on

over thirty different formulas, and exposed the panels at Miami, Fla., Pittsburgh, and in the Great Lakes region. Subsequently a few tests were also exposed at Washington and at Passaic, N. J. The general results of the tests are given below.

When Titanox is exposed outside in raw linseed oil liquids, it chalks rapidly. When it is exposed in light tints, this chalking causes the tints to be observed to some extent, and they appear to be faded. The soft nature of the straight Titanox reduces the retention of plant pollen or wood and dust that may be present in the air. This gives a darkened appearance to the film. Similar conditions are observed when Titanox and inert pigments are used together without any other additions.

When Titanox is combined with white lead, the same softness of film and dust retention is noted for the first exposure period of probably two or three months. Subsequent chalking causes the removal of the surface dirt and a white surface of great durability is disclosed. Such paints do not check or alligator, even after many years of exposure, but the initial defects of softness and dust retention make their use inadvisable, especially in tints. It is possible that a suitable liquid might be devised to prevent the defect referred to, so that white lead could be used suitably with Titanox.

When Titanox is mixed with from 25 to 50 per cent of zinc oxide, a paint is produced, that dries rapidly to a firm, sound, clean film that does not check or crack even after many years of wear. Such a paint remains clean and white under the most trying atmospheric conditions, especially when the air is highly polluted with industrial gases. It will, however, show mild chalking of a desirable nature after about two years' exposure. For tints, the use of the formula with the higher concentration of zinc maintains the clarity and color for long periods.

It was assumed by the writer when he first made the points referred to in the above tests that Titanox would be a thoroughly non-toxic substance. In order, however, to have experimental proof, a series of experiments were made during 1915, in which a number of healthy guinea pigs were utilized. These were placed in individual cages. The rabbits were each given the same amount of food every morning and their temperatures noted and body weights taken. For a period of two months the diet of each pig was exactly the same, and included one-half gram of Titanox per day. There was apparently no disturbance on the part of the subjects in taking the treated diet. No abnormal changes in body temperatures were observed during these experiments. There was, however, a normal gain in weight. In a period of two months the pigs, which weighed from 550 to 600 grams each, gained from 20 to 25 per cent in weight. No ill-furtherance or ill results of any kind were noted. These tests demonstrated that Titanox is to be regarded as thoroughly non-toxic. It is probable that the pigment is so insoluble that it cannot be taken up in the system but simply passes through with the food, being eliminated in almost quantitative amounts. Examinations of the feces collected from two of the animals showed the presence of over two-thirds the amount of pigment that had been administered. Collections for a longer period would probably have shown the full amount.

ANTIMONY OXIDE:- Antimony oxide as now produced comes as a result of roasting antimony ores, the vapors being condensed to form a white powder. The method is somewhat analogous to the production of zinc oxide, with the exception that there is said to be a difference in the vapor state of the two substances.

The oxide of antimony produced about twenty years ago was said to be somewhat coarse and of unsatisfactory properties for use in paint. One early patent has referred to the production of a pigment from lead ores containing antimony, by a roasting process at high temperatures, followed by sublimation. Clark* also refers to an early French process in which the vapors from roasted antimony ore were placed in contact with liquids containing alkaline earth metals, a combination pigment being produced. One of these pigments thus produced contained barium sulphate and might be termed an "antimony lithopone." Apparently, however, all of these early pigments met with no great success. It is possible that the chief fault was their color, which was often yellow, due to antimony sulphide being present. With further reference to processes, it would be of interest to know the extent of antimony ore which may in the future be available for the production of such a pigment, since some investigations have claimed that the world's supply of antimony is comparatively small. If this is true, its use will not be as extended as some might desire.

The writer's first experiments with antimony oxide date back to 1908, when a quantity of this product as made by an American manufacturer was submitted. A few preliminary laboratory tests were made with the product, but no exposures. It was not, however, until 1921 that any quantity of product was received for actual exposure tests, and this was in the form of a very finely divided white powder obtained abroad, where it has recently been introduced in a commercial manner. The writer's investigations disclosed the following properties,

In the table below are given the specific gravity, bulking value, and fineness of antimony oxide as compared with lithopone and Titanox.

*Development of Antimony Oxide. H. E. Clarke; Proc. Paint and Varnish Soc. of Great Britain, April, 1921.

Evaluation of White Pigments. H. E. Clarke: Jour. of Oil and Color Chem. Assn. of London. Jan., 1921.

	Specific gravity.	Weight per solid gal.	100 lbs. Pigment will bulk gallons of paint.	Fineness: per cent through 325 mesh screen.
Antimony Oxide.....	5.73	47.73	2.09	99.8
Lithopone	4.30	35.82	2.79	99.8
Titanox	4.30	35.82	2.79	99.8

Oil Absorption.—The oil absorption of a pigment depends to some extent upon the fineness, surface area of the particles, and other physical properties. Antimony oxide is rapidly wetted with linseed oil, 100 grams requiring 23.2 cc. of oil in the Gardner-Coleman test.* In an ordinary grinding test, the pigment forms a smooth paste, with 88 parts of pigment to 12 parts of linseed oil. For spreading, this can be reduced with further quantities of oil, drier and thinner to make a satisfactory paint if there is present from 68 to 75 per cent of pigment, the balance being liquids.

Approximately Correct Percentages of Pigment and Oil for Paint of Good Brushing Consistency

	Per cent.		Per cent.		Per cent.
Antimony Oxide.....	70	Lithopone.....	55	Titanox.....	60
Oil Liquids.....	30	Oil Liquids.....	45	Oil Liquids.....	40

Hiding Power and Whiteness.—The hiding power of a white pigment depends upon its refractive index, fineness, and other physical properties. Although brushing tests over black spotted areas are often made to determine the hiding power of a paint, the most reliable method is through the use of the Pfund cryptometer, a simple piece of apparatus that gives quick results in the laboratory. Whiteness or brightness and hiding power are, however, very closely associated, and all tests should be based upon pigments of comparatively the same color. Thus, for instance, most all white opaque pigments of good brightness are lower in hiding power than Titanox, lithopone, or antimony oxide, but their hiding power may be equal to some of the above if they are "dirty" or off-color, due to impurities in the process of manufacture.

*See Circular No. 95.

The writer's results with a number of samples of the white pigments referred to show approximately *the following order of hiding power:*

Titanox	Greatest hiding power
Lithopone	Next best hiding power
Antimony Oxide	Lowest hiding power

As referred to above, however, all of the above pigments are of greater hiding power than practically any other white pigment of equal brightness.

Other Physical Properties.—When antimony oxide in linseed oil is spread, it forms a film that is soft and tacky for many days, thus indicating a lack of drying properties. Even when the usual amount of drier is added, the same condition may be shown. This result has been attributed to the acidic nature of the pigment as contrasted to the basic nature of many other white pigments that dry up rapidly when exposed in oil with a small amount of drier. The use, therefore, of an extra amount of drier with antimony oxide is warranted, and *especially the use of a drier that contains some cobalt.* Another characteristic of antimony oxide paints is the tendency to "flat." In the writer's opinion, this condition is interwoven with the drying-retarding characteristics of the pigment, and could be largely overcome with suitable drier additions. Clarke* has advocated that one per cent of litharge be ground with antimony oxide as a *drier* accelerant. In many cases this would doubtless be an advantage. The writer has found that zinc oxide is also satisfactory for this purpose, and its use tends to produce a hardening action that holds back excessive chalking. In exposure tests at Washington,† antimony oxide weathered for several months without any marked defects except a curious form of erosion and surface chalking. No cracking or flaking was observed, but very marked surface discoloration was apparent, probably due to the retention of smoke and dust particles by a film that had not dried hard. Moreover, tints of the white faded out very rapidly, possibly due to over-chalking. In the composite pigment tests, the use of barium sulphate or of lithopone in moderate amounts resulted in much cleaner films, probably due to the added chalking tendency of these pigments. Quite good films were thus obtained in white, but in light tints, fading, due to chalking, was noted. It is too early to predict the proper formulas for exterior work, but it would

*See references above.
†Scientific Section Circular No. 187

be expected that a considerable amount of zinc oxide mixed with the inert pigment added to the antimony oxide would be an advantage. Sound films might thus be obtained.

On exposure to hydrogen sulphide fumes, antimony oxide paints turned yellow, due to the formation of antimony sulphide. This yellow color, however, disappeared entirely upon removal of the paints to clean air, oxidation of the yellow sulphide to a white antimony compound having occurred in less than 18 hours.

FIG. 1

In referring very extensively to the possible toxic effect of certain *antimony compounds*, Clark points out that antimony oxide is probably non-toxic, and ascribes certain cases of poisoning attributed to antimony as really due to arsenic. The claims that have been made regarding the non-toxic nature of antimony oxide led the writer to conduct a series of tests in which were included lithopone and Titanox, although previous evidence *already had classed these two latter pig-*

ments as non-toxic. The tests were made in co-operation with Dr. Oscar B. Hunter, of George Washington University, upon a number of rabbits that were first kept for several weeks under observation to determine their suitability for the tests. The rabbits were fed with an emulsion of pigment and water, the emulsion in each instance being passed through a rubber tube down the animals' throats into the stomach. By this method it was a simple matter to insure delivery of exact amounts. Body weights and temperature were taken occasionally. Starting with one gram of pigment, the doses were slowly increased to 5 grams daily. The latter amount represents what might be contained in a sewing thimble and is comparable to an amount based on the body weight of an average human, to 4½ ounces (an amount sufficient to fill a demitasse cup). Even such heroic doses administered over several weeks' time failed to have any marked effect, as is indicated in the following table:

Report on Clinical and Pathological Effects of Paint Pigments

Rabbit No. 2. TITANOX.

Material emulsified in 25 cc. of water and given as follows:

July	17, 1923	1 gram	(weight of animal, 3,100 grams)
"	20, 1923	3 grams	
"	30, 1923	5 "	(weight of animal, 3,055 grams)
"	31, 1923	5 "	
Aug.	1, 1923	5 "	
"	3, 1923	5 "	
"	10, 1923	5 "	
"	14, 1923	5 "	
"	16, 1923	5 "	
"	18, 1923	5 "	
"	20, 1923	5 "	
"	22, 1923	5 "	
"	24, 1923	5 "	
"	26, 1923	5 "	
Sept.	4, 1923	5 "	
"	6, 1923	5 "	
"	7, 1923	Killed and autopsied (weight, 3,200 grams)	

CLINICAL OBSERVATIONS: Animal exhibited no symptoms, except the usual indisposition to eat after administration.

AUTOPSY: Findings are negative, except for the presence of coccidiosis and the digestive disturbances noted in Rabbit No. 1. No definite pathological findings traceable to preparation.

IN LATER TESTS, THE RABBITS GIVEN TITANOX AND CITRIC ACID WERE UNAFFECTED.

Report on Clinical and Pathological Effects of Paint Pigments

Rabbit No. 1. LITHOPONE.

Material emulsified in 25 cc. of water and given as follows:

July	17, 1923	1 gram	(weight, 3,250 grams)
"	20, 1923	3 grams	
"	30, 1923	5 "	(weight, 3,300 grams)
"	31, 1923	5 "	
Aug.	1, 1923	5 "	
"	3, 1923	5 "	
"	10, 1923	5 "	(weight, 3,320 grams)
"	14, 1923	5 "	
"	16, 1923	5 "	
"	18, 1923	5 "	
"	20, 1923	5 "	
"	22, 1923	5 "	
"	24, 1923	5 "	
"	26, 1923	5 "	
Sept.	4, 1923	5 "	
"	6, 1923	5 "	
"	7, 1923	Killed and autopsied (weight, 3,310 grams)	

CLINICAL OBSERVATIONS: Animal exhibited no symptoms of importance, except some indisposition to eat after administration of preparation.

PATHOLOGICAL: Animal exhibited no pathological manifestations.

AUTOPSY: Autopsy shows no gross pathological changes, except some congestive disturbance of the intestines and mesentery. Coccidiosis present. Abdominal and thoracic viscera in good condition. Large intestines are somewhat distended, but show no pathological defects.

IN LATER TESTS, THE RABBITS GIVEN LITHOPONE AND CITRIC ACID WERE UNAFFECTED.

Report on Clinical and Pathological Effects of Paint Pigments

Rabbit No. 3. ANTIMONY OXIDE.

Material emulsified in 25 cc. of water and given as follows:

July	17, 1923	1 gram	(weight, 3,000 grams)
"	20, 1923	3 grams	
"	30, 1923	5 "	(weight, 3,025 grams)
"	31, 1923	5 "	

Aug.	1, 1923	5 grams
"	3, 1923	5 "
"	10, 1923	5 " (weight, 3,680 grams)
"	14, 1923	5 "
"	16, 1923	5 "
"	18, 1923	5 "
"	20, 1923	5 "
"	22, 1923	5 "
"	24, 1923	5 "
"	26, 1923	5 "
Sept.	1, 1923	5 "
"	6, 1923	5 "
"	7, 1923	Killed and autopsied (weight, 3,815 grams)

CLINICAL OBSERVATIONS: No symptoms observed, except the usual indisposition to eat after administration.

AUTOPSY: On superficial examination the thoracic and abdominal viscera appeared to be in good condition. Slight chronic gastritis, with some thickening of the stomach walls. Liver exhibited some coccidiosis. Intestines show some slight enteritis, with moderate congestion, but otherwise normal. Large intestines are distended with feces matter, brownish-black in color, but no specific pathology found. Heart and lungs normal.

IN LATER TESTS, THE RABBITS GIVEN ANTIMONY OXIDE AND CITRIC ACID DIED IN 24 HOURS.

The first results were rather startling in relation to the antimony oxide, since it was the writer's opinion that this substance was very apt to be toxic. It is apparent, however, that the antimony oxide was insoluble in the stomach juices of the animals and passed out in the feces without effect. In fact, examination by furnacing of the feces from all the animals resulted in round white pills giving every test for the pigment as originally administered. In view, however, of the fact that workmen might drink acidulated beverages during the application of paint, it was thought desirable to repeat the tests with occasional administration of the diluted juice of lemons. This was done and no effect was noted on the rabbits treated with Titanox or with lithopone. Those treated with antimony oxide died in 24 hours. Subsequent repeat tests checked this finding. After an autopsy, the stomach and intestines were examined and soluble antimony compounds found. Apparently the citric acid in the lemon juice had exerted a very marked solvent power upon the antimony oxide.

Tests were then made with lemon juice and different strengths of C. P. citric acid upon antimony oxide. The tests indicated that these substances, even in dilution, are great solvents for antimony oxide. Shaking 1 gram in a liter of 0.25 per cent citric acid showed about 40 per cent soluble.

LITHOPONE.—The writer's first experience with lithopone dates back to 1907, when this pigment was being introduced gradually in the paint trade, one of its chief uses at that time being in linoleum, rubber, and similar industries.

The production of exterior paints with it had not then been found satisfactory, due to the occasional darkening effect of sunlight and moisture, but its use in interior paints was well started. Combined with Chinese wood oil varnishes carrying a considerable amount of thinner, manufacturers were able to place on the market quick drying flat coatings of great hiding power and whiteness. Probably no type of interior paint ever gained as rapid headway or became as popular in a short period of time. A comparison, for instance, for the year 1907, when two or three plants made a total of a thousand tons, with 1922, when eight American plants produced a total of over one hundred thousand tons (two hundred million pounds), shows the strides made in its production and use. Some recent figures on lithopone production, as presented before the American Zinc Institute in 1923, are of interest to record:

	Tons.		Tons.
1906	1,200	1914	32,819
1907	10,275	1915	66,494
1908	8,282	1916	51,290
1909	14,947	1917	63,713
1910	12,675	1918	62,923
1911	16,898	1919	80,000
1912	24,250	1920	90,375*
1913	29,965	1921	55,000*
		1922†	100,000 to 115,000

The chief cause of this rapid development was the use of lithopone in flat and gloss interior whites. Previous to 1910 the use of paints was very largely confined to exterior surfaces, the interior walls and ceiling surfaces being coated with paper in the case of houses or offices, or left bare in the case of factories. When people began to appreciate the ease with which surfaces could be hidden with the new type of paint, and the increase of light and sanitary conditions following its use, the development became rapid and interior painting popular.‡

*Domestic production marketed, as given by U. S. Geological Survey.
†Estimated.
‡For information regarding the value of interior wall paints, see Scientific Section Circulars, Nos. 7, 10, 28, 33, 43, 34, 107, etc., etc.

It is most probable that the amount of electric current saved through the introduction of these paints in factories, together with the increased efficiency of the workers, has more than balanced the entire cost of the paints and their application. What this has meant to the contractor as well as to the economic situation in the employment of labor is difficult to estimate.

During 1909 the writer exposed in Atlantic City and in Pittsburgh a series of lithopone paint tests in white and in colors to ascertain what combination was best suited for exterior work. Mixtures with various forms of white lead, zinc oxide, and with inert pigments were tried. Without referring to the darkening action that may have occurred on some of these as a result of the unsatisfactory quality of the lithopone then produced, the main defect noted in the tests was a form of rapid chalking that seemed in some way related to washing, some investigators claiming that the zinc sulphide present in lithopone undergoes oxidation to zinc sulphate, which is, of course, water soluble. The writer found, however, that mixtures with about 50 per cent of zinc oxide gave better results than with the other formulas, and the use of some inert pigment seemed justifiable. Thus, for instance, one of the best lithopone formulas tried out was composed of 40 per cent lithopone, 10 per cent zinc, and 20 per cent whiting. These tests probably afforded the first information regarding the proper design of lithopone paints for exterior use. Since that period, further tests have been made by other observers, and recommendations have been made that experimental paints should be made up with about 50 per cent zinc oxide. One formula that has been widely recommended for test is as follows:

Pigment	55
Liquid	45

Pigment.

Lithopone	45
Zinc Oxide	45
Barytes	10

The liquid to be raw linseed oil, with not over 15 per cent combined thinner and drier.

Many tests have also been proposed with liquids containing a percentage of varnish or boiled oil in an effort to retard the chalking properties of the pigment.

A formula recently recommended to the writer for exposure i
follows:

Pigment	65%
Liquid	35%

Pigment Composition

Zinc Oxide	40%
Lithopone	30%
Asbestine	20%
Silex	10%

Liquid Composition

Raw Linseed Oil	40%
Boiled Linseed Oil	45%
Blown Linseed Oil	5%
Combined Turpentine and Drier	10%

NEW EXPOSURE TESTS ON TITANOX PAINTS AND ON LITHOPONE PAINTS

In these tests it is hoped to determine whether boiled oil or varnish will increase the durability of some single pigments and decrease the chalking tendency of others. It is also believed that the tests will indicate the relative value of the various pigment formulas.

One complete set of the new test panels are being exposed at the University of Florida at Gainesville, Fla., where climatic conditions are extremely severe upon paint coatings. The intense humidity throughout the majority of the year, combined with the high rainfall and very bright sunlight, form conditions that cause accelerated wearing of paints. Another complete set of these panels are exposed on the roof of the writer's laboratory in Washington, D. C. All panels were of white pine and the date of exposure January 20, 1921.

In the first series of tests, Titanox (100 per cent) and then lithopone (100 per cent) were each tried out in four different liquids, the formulas and formula numbers being given below. Each paint was applied in white and also in a blue tint, Prussian blue being used for tinting. The pigment concentration in the finished paints was usually 60 per cent for the Titanox combinations and 50 per cent for the lithopone combinations in this first set. The liquids used are described in the formulas.

Formulas

	Per cent by weight.		Per cent by weight.
11		**12**	
Titanox	60	Lithopone	50
Raw Linseed Oil	34	Raw Linseed Oil	43
Drier	6	Drier	7
13		**14**	
Titanox	60	Lithopone	50
Liquid T	40	Liquid T	50
15		**16**	
Titanox	50	Lithopone	50
Raw Linseed Oil	25	Raw Linseed Oil	31
Spar Varnish	11	Spar Varnish	19
Turpentine	7		

Formulas—Continued

	Per cent by weight.		Per cent by weight.
17		**18**	
Titanox	60	Lithopone	50
Aluminum Stearate	2	Aluminum Stearate	2
Liquid T	30	Liquid T	48
Turpentine	8		

The liquid referred to as "Liquid T" has the following composition:

	Per cent.
Raw Linseed Oil	62.5
*Boiled Linseed Oil	17.5
Turpentine	7.5
†Drier	12.5

The panel numbers with formulas of the paints used are given below:

Panel No.,	Painted with Formula (white and blue) of Formula No.
76	11
76	12
77	13
78	14
79	15
80	16
81	17
82	18

In the next series of tests, different mixtures of zinc, lead, and inert pigments were tried with Titanox and again with lithopone. The pigment concentration with the Titanox was 60 per cent, and 55 per cent with lithopone. All paints were applied in white and again in a blue tint. It will be noted that the boiled oil liquid formula was also used in these tests. The pigment ratio in each is shown graphically below:

Formula No.	21	22	25	27	29	31	33	35	
Titanox	18	30	30	24	24	30	30	24	% by weight.
Zinc Oxide	12	24	30	24	12	30	..	24	" " "
Barytes	..	..	..	12	24	30	..	..	" " "
White Lead	..	..	..	..	..	..	30	12	" " "
Liquid T	40	40	40	40	40	40	40	40	" " "

* The boiled oil used was boiled linseed of such viscosity that 80 parts by volume with 20 parts of turpentine, checks Tube I in the Gardner-Holdt viscosity standards.

† Drier used throughout tests was a well thinned turpentine drier containing lead, manganese, and cobalt.

Formula No.	22	24	26	28	30	32	34	36	
Lithopone	44	33	27	22	22	18	27	22	% by weight.
Zinc Oxide	11	22	25	22	11	18	..	22	" " "
Barytes	..	..	..	11	22	18	..	..	" " "
White Lead	..	..	..	..	..	..	27	11	" " "
Liquid T	45	45	45	45	45	45	46	45	" " "

The actual formulas, showing pigment and liquid, are given below:

21

Titanox	48
Zinc Oxide	12
Liquid T	40

22

Lithopone	44
Zinc Oxide	11
Liquid T	45

23

Titanox	36
Zinc Oxide	24
Liquid T	40

24

Lithopone	33
Zinc Oxide	22
Liquid T	45

25

Titanox	30
Zinc Oxide	30
Liquid T	40

26

Lithopone	27.5
Zinc Oxide	27.5
Liquid T	45.0

27

Titanox	24
Zinc Oxide	24
Barytes	12
Liquid T	40

28

Lithopone	22
Zinc Oxide	22
Barytes	11
Liquid T	45

29

Titanox	24
Zinc Oxide	12
Barytes	24
Liquid T	40

30

Lithopone	22
Zinc Oxide	11
Barytes	22
Liquid T	45

31

Titanox	20
Zinc Oxide	20
Barytes	20
Liquid T	40

32

Lithopone	18
Zinc Oxide	18
Barytes	18
Liquid T	45

33

Titanox	30
White Lead Carbonate	30
Liquid T	40

34

Lithopone	27.5
White Lead Carbonate	27.5
Liquid T	45.0

35

Titanox	24
Zinc Oxide	24
White Lead Carbonate	12
Liquid T	40

36

Lithopone	22
Zinc Oxide	22
White Lead Carbonate	11
Liquid T	45

The panel numbers with formulas of paints used are shown below:

Panel No.	Painted with 3 coats (white and blue) of Formula No.
83	21
84	22
85	23
86	24
87	25
88	26
89	27
90	28
91	29
92	30
93	31
94	32

Panel No.	Painted with 3 coats (white and blue) of Formula No.
95	33
96	34
97	35
98	36

Reference Panels.

99	White Lead.
100	Lead-Zinc.

For control tests to judge the durability of these paints, panels No. 99 were given 3 coats of white-lead paint in white and in blue, and panels No. 100 were given 3 coats of lead-zinc prepared paint.

NEW EXPOSURE TESTS TO DETERMINE THE EFFECT OF ALUMINUM POWDER IN WHITE PAINTS AND THE EFFECT OF ALUMINUM POWDER PAINTS AS WOOD PRIMERS

In a series of tests with aluminum powder, made during 1921 to 1923,* a few panels of wood were included, and to these a priming coat of aluminum paint was applied with two subsequent coats of lead-zinc paint. Weathering indicated on some panels less checking and warping of the wood near the edges of the panels, and consequently fewer defects in the white paint than when the primer was omitted. In order to make a more exhaustive study of the possibilities of aluminum primers for wood, a number of panels have been painted, including many woods that are supposed to be "refractory" to painting.

Aluminum powder, when used as a paint pigment, effectually shuts off the ultra-violet rays of light that destroy linseed oil. For this reason, aluminum paints, when made with a good liquid, have proved very durable. With the idea that even a small amount of aluminum powder might have a similar effect upon white paints, many of which are not thoroughly opaque to ultraviolet rays, the writer has included a series of paints to which 1½ per cent of aluminum powder was added. This was sufficient to throw off the apparent color of the paints, and actually made them of greater apparent whiteness and of greater hiding power. If this small amount of aluminum powder effectually prevents the rapid chalking of certain single pigments by stopping the action of light upon the liquids, the results will be most interesting.

Outline of Tests

Exposed at Washington, D. C., January 15, 1924

Woods:	White Pine	Douglas Fir	Cypress	Yellow Pine	Redwood
	W	F	C	Y	R

Paints: Liquid G used in several paints is composed of 85 per cent Raw Linseed Oil and 15 per cent Combined Drier and Turpentine.

* See Scientific Section Circulars Nos. 130, 153, and 187.

Paint No. 1

	Per cent by weight.	
Aluminum Powder	25	A rapid drying aluminum paint.
Spar Varnish	50	
Mineral Spirits	25	
	100	

Paint No. 2

	Per cent.	
Zinc Oxide	31	A high grade prepared paint formula of
Basic Carbonate - White Lead	31	the 50-50 type.
Liquid G	38	
	100	

Paint No. 3

	Per cent.	
Zinc Oxide	24	A correctly proportioned Titanox paint.
Titanox	38	
Liquid G	40	
	100	

Paint No. 4

	Per cent.	
Aluminum Powder	25	An aluminum paint made with a vehicle of
Heavy Bodied Linseed Oil	45	good suspension and dispersion proper-
Turpentine	30	ties. To get satisfactory drying proper-
	100	ties, a gill of L. & M. Liquid Drier was added to a gallon of paint.

Paint No. 5

	Per cent.	
Aluminum Powder	25	An aluminum paint that has proved dur-
Boiled Linseed Oil	50	able in previous tests. To get satisfac-
Mineral Spirits	25	tory drying properties, two-thirds of a
	100	gill of L. & M. Liquid Drier was added to a gallon of paint.

Paint No. 6

	Per cent.
Basic Carbonate-White Lead	72
Liquid G	28
	100

Paint No. 6 A

Same as No. 6, but tinted with Prussian blue.

Paint No. 6 B

Same as No. 6, but with 1½ per cent added Aluminum Powder.

Paint No. 7

	Per cent.
Basic Sulphate White Lead	70
Liquid G	30
	100

Paint No. 7 A

Same as No. 7, but tinted blue.

Paint No 7 B

Same as No. 7, but with 1½ per cent added Aluminum Powder.

Paint No. 8

	Per cent.
Zinc Oxide	50
Liquid G	50
	100

Paint No 8 A

Same as No. 8, but tinted blue.

Paint No. 8 B

Same as No. 8, but with 1½ per cent added Aluminum Powder.

Paint No. 9

	Per cent.
Lithopone	50
Liquid G	50
	100

Paint No. 9 A

Same as No. 9, but tinted blue.

Paint No. 9 B

Same as No. 9, but with 1½ per cent added Aluminum Powder.

Paint No. 10

	Per cent.
Titanox	62
Liquid G	38
	100

Paint No. 10 A

Same as No. 10, but tinted blue.

Paint No. 10 B

Same as No. 10, but with 1½ per cent added Aluminum Powder.

Order of Tests

Letters indicate wood and figures indicate paint formula and number of coats used. Thus W 1-6-6 means white-pine wood with 1 coat of Paint No. 1 and 2 coats of Paint No. 6.

In this series three white paints are tested out with and without a priming coat of aluminum powder in spar varnish.

Panel No.	Wood and Paint Numbers.	Panel No.	Wood and Paint Numbers.
1	W 6 6-6	16	C 1-5-5
2	W 1-6 6	17	[illegible]
3	P 6-6-6	18	[illegible]
4	P 1-6-6	19	[illegible]
5	C 6-6-6	20	[illegible]
6	C 1-6 6	21	[illegible]
7	T 6-6 6	22	[illegible]
8	T 1 6 6	23	[illegible]
9	R 6 6 6	24	[illegible]
10	R 1-6-6	25	[illegible]
11	W 5-5 5	26	[illegible]
12	W 1-5 5	27	[illegible]
13	P 5 5 5	28	[illegible]
14	P 1 5-5	29	[illegible]
15	C 5 5 5	30	[illegible]

In this series, aluminum powder in heavy boiled linseed oil *versus* aluminum powder in boiled linseed oil are tested out as a primer for a lead-zinc paint.

Panel No.	Wood and Paint Numbers.	Panel No.	Wood and Paint Numbers.
31	W 4-2 2	36	C 3-2-2
32	W 3 2 2	37	[illegible]
33	P 4 2 2	38	[illegible]
34	P 3 2 2	39	[illegible]
35	C 4 2 2	40	[illegible]

In this series, two coats of different types of aluminum powder primers, namely, spar varnish *versus* heavy boiled linseed oil, are tested out with two coats of a lead-zinc paint.

Panel No.	Wood and Paint Numbers.	Panel No.	Wood and Paint Numbers.
41	W 1-1-2-2	46	C 4-4-2-2
42	W 4-4-2-2	47	Y 1-1-2-2
43	F 1-1-2-2	48	Y 4-4-2-2
44	F 4-4-2-2	49	R 1-1-2-2
45	C 1-1-2-2	50	R 4-4-2-2

In this series, three coats of the heavy bodied oil-aluminum powder primer are applied to a panel without any subsequent paints.

Panel No.	Wood and Paint Number.
51	W 4-4-4
52	F 4-4-4
53	C 4-4-4
54	Y 4-4-4
55	R 4-4-4

In this series, five different white paints are tried out alone, again tinted blue, and again in white, but with a small percentage of added aluminum powder to cut off the ultraviolet rays of light that destroy oil films. The amount added was small (1¼ per cent) and served to make the paints of greater apparent whiteness.

Panel No.	Wood and Paint Number.
56	W 5
57	W 6 A
58	W 6 B
59	W 7
60	W 7 A
61	W 7 B
62	W 8
63	W 8 A
64	W 8 B
65	W 9
66	W 9 A
67	W 9 B
68	W 10
69	W 10 A
70	W 10 B