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EFFECT OF GAMMA RADIATION
ON SELECTED ORGANIC PROTECTIVE COATINGS

by

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Missouri School of Mines and Metallurgy
in partial fulfillment of the requirements for the
Degree of

MASTER OF SCIENCE
in
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I. INTRODUCTION

The development of atomic energy with the accompanying production of waste fission products has produced a large number of problems. The fission products are radioactive and emit large quantities of beta and gamma radiation that are able to cause atoms or molecules to become ionized or electronically excited, either directly or indirectly. These effects may lead to chemical changes, especially if the radiations interact with compounds containing covalent bonds, such as organic materials. Plastics and non-pigmented protective coatings are essentially pure organic compounds that will usually be used in areas where radioactive products are being handled.

Organic solids undergo rather complicated chemical reactions as a result of the absorption of this high energy radiation. These reactions are not yet fully understood in even the most simple polymers. Two general classes of reactions have been recognized, however, which can account for most of the observed changes in the physical properties of these polymers. The reactions are: (1) Cleavage or scission, and (2) crosslinking. The cleavage reaction breaks the polymer chain into smaller fragments while the crosslinking reaction causes the polymer chains to be bound together in a network whose extent is dependent on the amount of crosslinking that has taken place.

It has been found in the normal field of use that pigments may impart many desirable properties to organic protective coatings. Pigments are used primarily to impart color and opacity, but they also affect other physical properties of coatings. In general, inorganic pigments contribute to the hardness and firmness of coatings. Plate-like pigments decrease permeability, but bulky pigments tend to make the coating spongy or porous. Many pigments are nodular in shape, but some are needle-like and tend to physically reinforce the coating. Basic pigments will react with free fatty acids, found in many film formers or formed during oxidation, to form soaps that may tend to harden or soften the film, depending on the pigment. Other pigments may impart such special properties as protection against ultra-violet light, mildew, and certain corrosive atmospheres; give the film self-cleaning properties; or simply add bulk to more expensive pigments.

The purpose of this investigation was to prepare pigmented and non-pigmented organic protective coatings and to irradiate them with gamma rays. The coatings received a constant radiation dose of eight million rads. Seven tests were performed on the various coatings. The data from the tests were tabulated so that a direct comparison could be made as to the effect of the radiation on the coatings. The data from the tests also enabled comparisons to be made to determine if the various pigments had imparted any specific properties that would be desirable in coatings being exposed to radiations.

II. LITERATURE REVIEW

This review is presented in three parts: (1) The interaction of gamma rays with matter, (2) the reactions of polymers resulting from absorption of high energy radiations, and (3) the effect of gamma radiation on organic protective coatings.

Gamma Rays

Gamma rays(11) are electromagnetic radiations similar to x-rays. They are highly penetrating and have short wavelengths, usually 10^{-9} to 10^{-11} centimeters. They include all electromagnetic radiation of nuclear orgin. The energy of gamma rays is emitted as photons. These photons may be regarded as particles of radiation. The energy of the photon is equal to the difference in energy between the two energy states (excited and ground) involved in the transition of a nucleus.

$$\text{Radioactive Parent} \xrightarrow[\text{or } \beta]{-\alpha} \text{Excited State} \xrightarrow{-\gamma} \text{Ground State}$$

Gamma radiation is described in terms of its photon energy, that is, one Mev (million electron-volts) gamma rays refer to gamma photons with one Mev of energy.

<u>Interaction of Gamma Rays with Matter.</u>(12) All electromagnetic radiations of high energy (0.01 to 100 Mev) interact with matter in a similar manner. This includes gamma rays, x-rays, bremsstrahlung, and annihilation radiation. Gamma rays interact in several different

ways with an absorbing medium. The ways to be considered in this review are: (1) Photoelectric effect, (2) Compton effect, and (3) pair production.

Photoelectric Effect.(12) A gamma photon which has energy greater than the binding energy of an orbital electron of an atom will interact with an electron in such a manner that the entire photon energy is transferred to the electron. The ejected electron is called a photoelectron, and has a kinetic energy equal to the difference between the photon energy and the electron binding energy. The photoelectron then behaves as a beta particle in its passage through matter. Photoelectric interaction is dependent on the energy of the gamma radiation (E), and the number of electrons present, which is the same as the atomic number (Z) of the absorbing material.

$$\left.\begin{array}{l}\text{Probability of Photo-}\\ \text{electric Interaction}\end{array}\right\} = \text{constant} \times \frac{Z^n}{E^3} \qquad (1)$$

This equation is a rough approximation. The "n" varies from three for low-energy gamma photons to five for high-energy gamma photons. In actual practice, photoelectric absorption is important for gamma rays with energies less than one Mev and then only for absorbers of high atomic number. When the photoelectrons are ejected from an inner orbit, an outer electron will take its place, with emission of characteristic x-rays. These x-ray photons frequently eject an outer electron (Auger electron) with the photon losing all its energy. In this manner, the photoelectric effect may cause complete absorption of gamma photons.

<u>Compton Effect.</u>(13) In this process, a gamma photon makes an elastic collision with an outer electron of an atom of the absorbing material. The electron is so loosely bound that it acts as if it were completely free. Both momentum and kinetic energy are conserved. Part of the photon energy is transferred to the electron. The photon is deflected or scattered from its original path. The relations can be expressed as:

$$\frac{E'}{E} = \frac{0.51}{E(1 - \cos \theta) + 0.51} \qquad (2)$$

where: E = energy of incident photon, Mev.

E' = energy of deflected photon, Mev.

θ = scattering angle.

As an interaction between a photon and an electron is involved in the Compton effect, the probability of its occurance is dependent on the number of orbital electrons, or the atomic number (Z) of the absorber. An approximation of the probability of a Compton interaction is given by the following equation.

$$\left.\begin{array}{l}\text{Probability of}\\ \text{Compton Interaction}\end{array}\right\} = \text{constant} \times \frac{Z}{E} \qquad (3)$$

In this interaction, the energy of the photon is merely decreased and the photon will ultimately escape from the medium if it is not absorbed by a photoelectric interaction, which becomes more probable as the photon energy is decreased.

Pair Production.(14) When a gamma-ray photon passes through the strong electrical field near a nucleus, the photon can be annihilated with the formation of an electron-positron pair. The energy equivalent of the mass of the electron-positron pair is 1.02 Mev, therefore, this is the minimum energy necessary for pair production. Photon energy in excess of 1.02 Mev appears mainly as kinetic energy of the pair, with a small part transferred to the atomic nucleus. The probability of pair production is dependent on the energy (E) of the photon and the atomic number (Z) of the absorbing nucleus.

$$\left.\begin{array}{l}\text{Probability of}\\ \text{Pair Production}\end{array}\right\} = \text{constant x } Z^2(E - 1.02) \qquad (4)$$

Pair production becomes the dominate type of interaction for gamma rays at high energies (above five Mev). The probability of this interaction increases with increasing photon energy while the probability of both the photoelectric effect and the Compton effect decreases with increasing photon energy. Pair production results in the complete absorption of the gamma-ray photon similar to the photoelectric effect. Although some of the electrons and positrons may come together to produce annihilation radiations (two 0.51 Mev photons), the proportion continuing in the forward direction is very samll because the radiation has an isotropic distribution. Therefore, for many practical purposes, it is assumed that photons are completely absorbed in pair production.

Absorption of Gamma Rays.(15) When a collimated beam of gamma-ray photons pass through matter, it has been found that a fairly straight line is obtained if the logarithm of the intensity of the gamma-ray beam is plotted versus the thickness of the absorber traversed. Thus it is shown that:

$$d(\ln I) \propto - dx \qquad (5)$$

$$= - \mu \, dx \qquad (6)$$

where: I = intensity, photons (or Mev) per square centimeter per second.

μ = linear absorption coefficient, per centimeter.

x = thickness of absorber traversed by the gamma rays, centimeter.

Equation (6) may be integrated, to give:

$$I_x = I_o \, e^{-\mu x} \qquad (7)$$

where: I_x = intensity of beam emerging from absorber.

I_o = intensity of beam impinging upon the absorber.

This shows that the intensity of gamma rays falls off in an exponential manner in their passage through matter, and that, although the amount of radiation absorbed by a specified material is proportional to the intensity, the fraction of the radiation absorbed is independent of the intensity. Also, the equation shows that, theoretically, to absorb gamma radiation completely, an infinite thickness of absorber must be used.

Absorption Coefficients.(16) The linear absorption coefficient in equation (7) may be evaluated by measuring the intensity of a collimated beam of monoenergetic gamma rays before and after passing through a known thickness of absorber. This will be the total absorption coefficient and is the summation of the contributions made by the photoelectric effect, the Compton effect, and pair production. As seen previously, the mode of interaction of gamma rays is energy dependent, so that the linear absorption coefficient will vary with the energy of the gamma rays. Also, it will be observed that the value of the linear absorption coefficient varies over a wide range for different materials. However, if we define a mass absorption coefficient of an absorber as its linear absorption coefficient divided by its density, it will be seen that for gamma rays of a specified energy this ratio is approximately constant for most materials. This fact may be used to estimate absorption coefficients when appropriate data are unavailable.

Dosage Units.(4, 7, 17) As we have seen in the preceeding sections, the end result of the absorption of gamma rays is the production of energetic electrons in matter, with a net effect of ionization and excitation of molecules. The amount of ionization that is caused in a substance is used to express the dose of radiation that the substance has received. One such unit of dosage is the roentgen which is defined as "that quantity of X- or gamma radiation such that the associated corpuscular emission per cubic centimeter of dry air at standard temperature and pressure, produces, in air, ions carrying one electrostatic unit of quantity of electricity of either sign." This is equivalent to an energy absorption of eighty-three ergs per gram of air. The unit used in this study was the rad, which represents one hundred ergs per gram, imparted by ionizing particles to the irradiated material at the point of interest.

Reactions of Polymers Caused by Absorption of High Energy Radiation

Reactions (2, 3, 38, 40) which have been observed as a result of the absorption of high energy radiation by organic compounds are crosslinking, degradation, discoloration due to F-centers, gas liberation, double bond formation, free radical formation, oxidation, and polymerization. Two from this group, cleavage and crosslinking, have been recognized that can explain most of the observed changes in the physical properties of plastics and elastomers.

It is a well established fact that when long chain polymers are irradiated by high energy radiation, new chemical bonds between the molecules and molecular scissions are produced. Saito (35) has classified the effects on the polymers as crosslinking, degradation, endlinking, and cyclization. Crosslinking is the chemical binding by which two molecules are combined at their side bonds, with the molecules ultimately being bound together in a network similar to a gel. Scission or cleavage of molecular main chains results in degradation. Endlinking is the combination of molecular ends, newly produced by scission, with other molecules at the side bonds. Links formed between monomers of the same molecule are called cyclization. Upon being irradiated, polymers may suffer all of these effects at the same time, with the overall effect being the one that predominates.

Wall (40) has reported that crosslinking and scission can be related with the heats of polymerization. In general, crosslinking with low monomer yield was found to be associated with polymers that have high heats of polymerization, and degradation to be associated with polymers with the lower heats of polymerization. The results of this work is shown in Table I, page 11. Low heats of polymerization are usually associated with high steric repulsion between side groups on the polymer chain. This effect would favor scission and would also operate against recombination in case of a main chain rupture. This is in agreement with Wall's report.

Sisman and Bopp (38) have pictured the crosslinking, unsaturation, and gas production reactions with a very simple molecule as shown in Figure 1, page 12. The creation of free radicals by the release of hydrogen from the polymer chains by the radiation is the start of the reaction. If the life time of the free radicals is sufficiently long, some may combine with each other to form crosslinked molecules. The

TABLE I

Correlation of Polymer Properties with Irradiation Effects

Direction of Irradiation Effect	Polymer	Heat of Polymerization, kcal/mole Monomer	Monomer Yield, Wt. %
Crosslink	Ethylene	22	0.025
do.	Propylene	16.5	2
do.	Methyl acrylate	19	2
do.	Acrylic acid	18.5	----
do.	Styrene	17	40
Degrade	Methacrylic acid	15.8	----
do.	Isobutylene	13	20
do.	Methyl methacrylate	13	100
do.	α-Methyl styrene	9	100

Wall, Leo A.: Degradation of Polymers by Radiation, Papers Presented at the New York Meeting, The American Chemical Society, Division of Paint, Plastics, and Printing Ink Chemistry, 17, p. 300 (Sept. 1957).

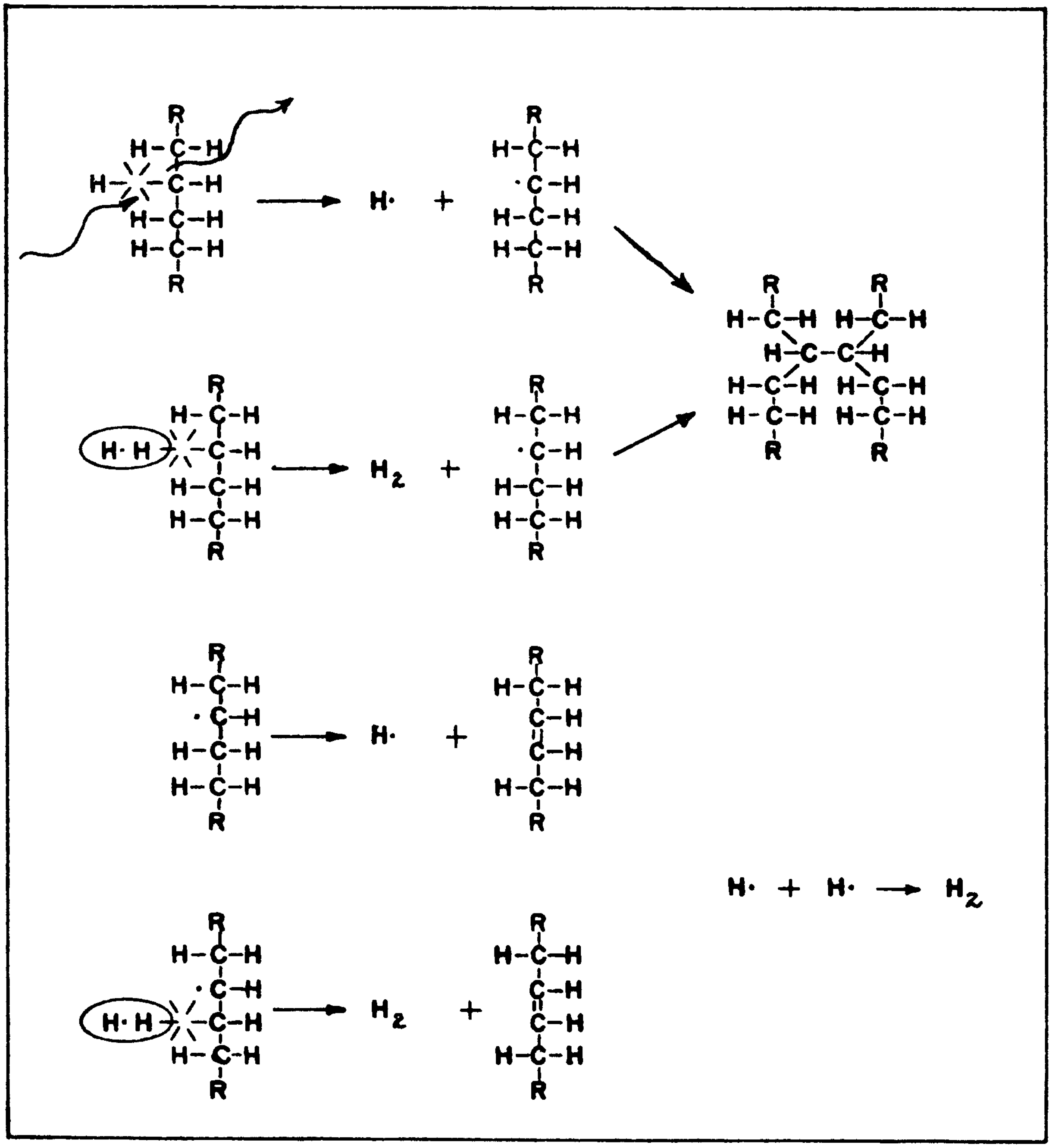

Figure I. Illustration of Crosslinking, Unsaturation, and Gas Production.

Sisman, O. and C. D. Bopp: A Summary of the Effect of Radiation on Some Plastics and Elastomers, ASTM Special Technical Publication No. 208, <u>1</u>, 120 (1957).

crosslinking is considerably enhanced by the fact that the hydrogen atom may abstract a hydrogen atom from a nearby molecule leaving two free radicals close together and in a good position for crosslinking. The release of hydrogen gas accompanies this reaction.

It is not so easy to picture the cleavage reaction, and this mechanism has been the subject of many conflicting theories. Most agree that crosslinking is the logical reaction and that cleavage will occur only when conditions are right. One such theory (38) for the reaction is pictured in Figure 2, page 14. This reaction involves a molecule that will tend to absorb a hydrogen atom at the site of a strained bond and cause the molecule to break into two smaller fragments. One of the fragments is a free radical and may join with another radical, or become unsaturated with the release of a hydrogen atom. Less gas is produced by materials that cleave than those which crosslink, though both may suffer the formation of a considerable amount of unsaturation.

The stability against cleavage of the basic chemical structures of many of the plastics and elastomers have been studied. A few are listed in order of stability in Figure 3, page 15. The order is an approximate one and the resistance to cleavage may not be much different for structures lying close together, but for structures ranked far apart, there is always a large difference.

The basic unit of polystyrene is the highest ranking structure. It is predominantly crosslinked by irradiation as its resistance to cleavage is very high, although its rate of crosslinking is very low.

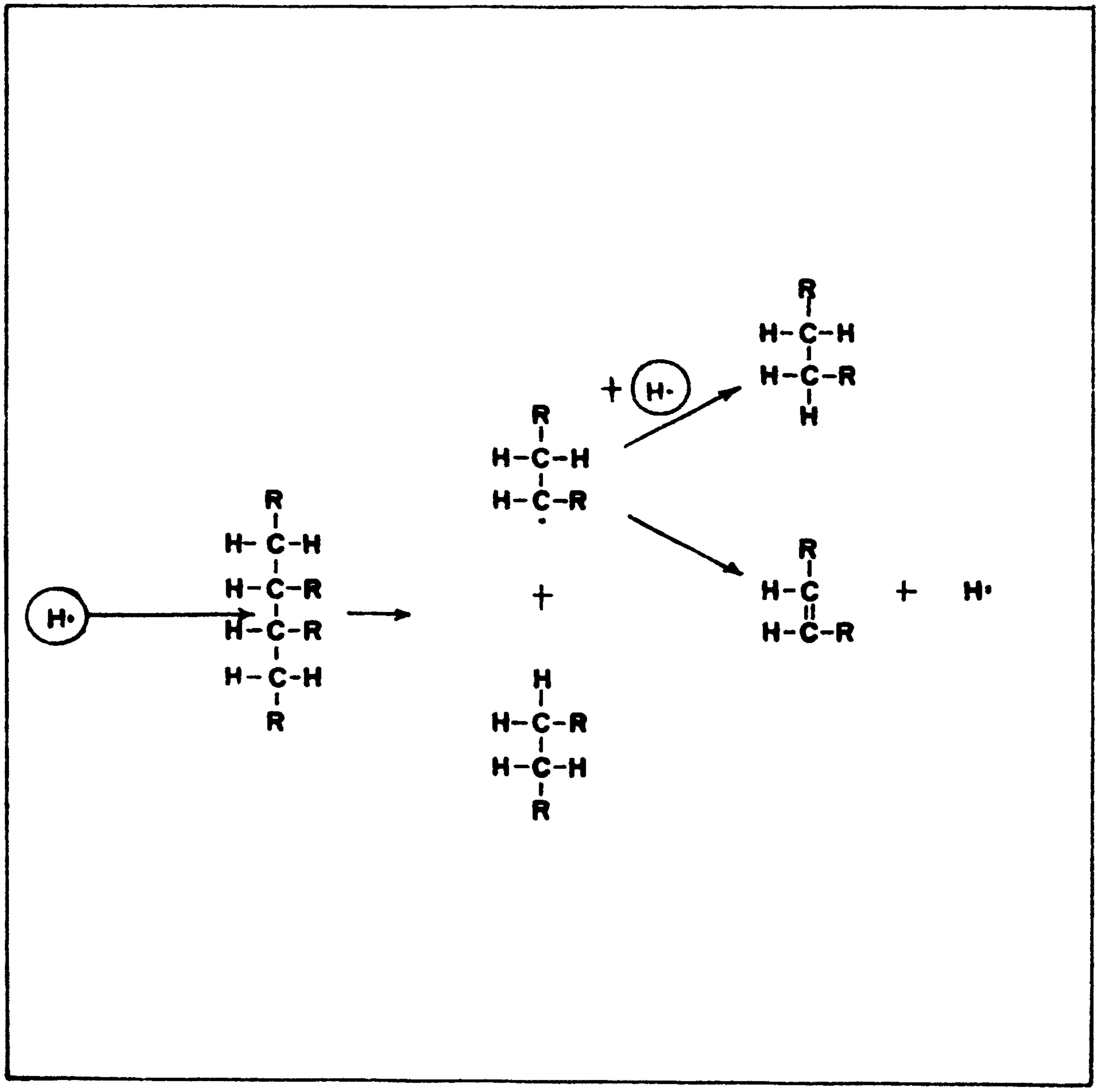

Figure 2. Illustration of Cleavage.

Sisman, O. and C.D. Bopp: A Summary of the Effect of Radiation on Some Plastics and Elastomers, ASTM Special Technical Publication No. 208, 1, 121 (1957).

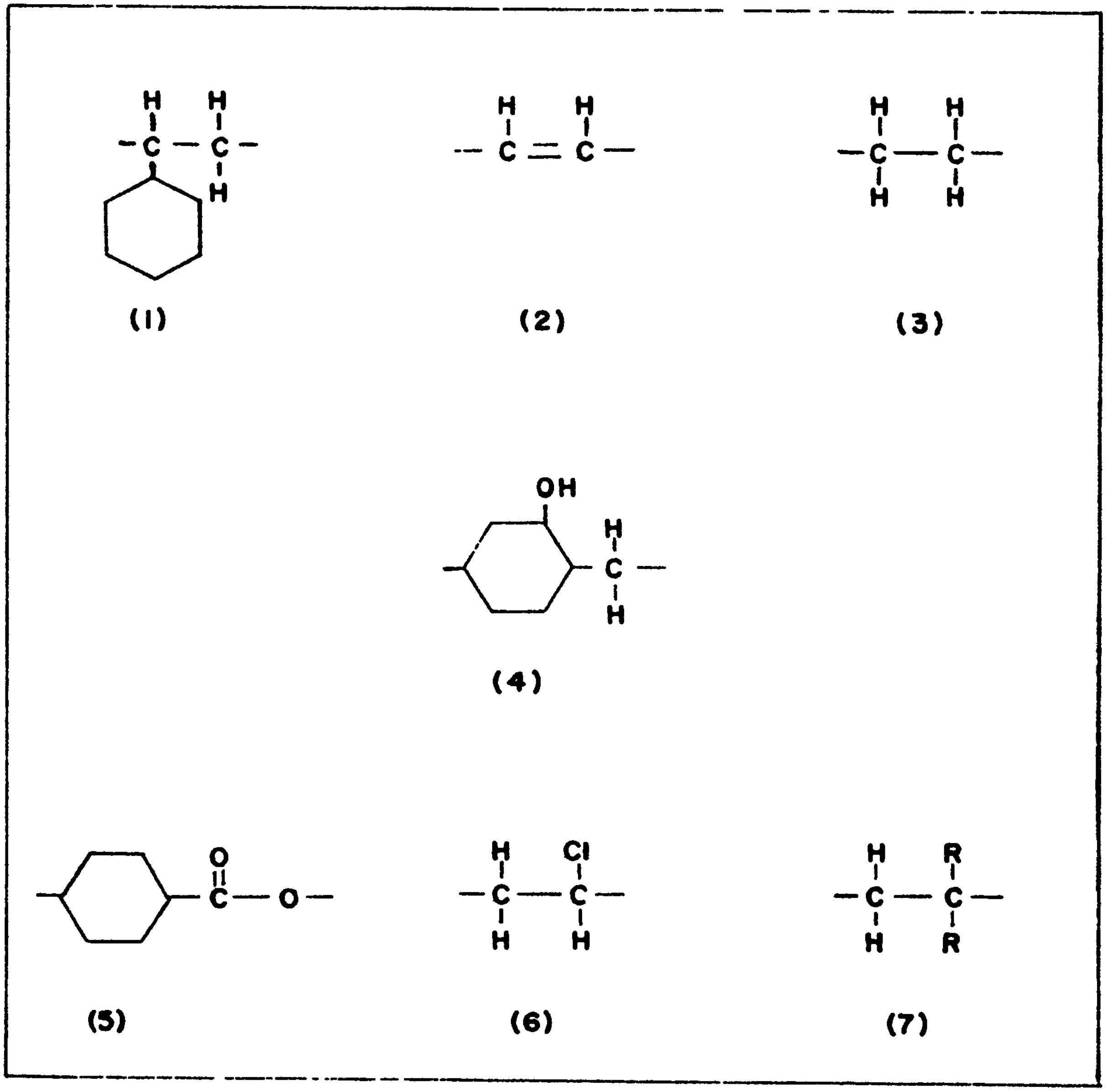

Figure 3. Chemical Groupings Ranked in Order of Radiation Stability.

Sisman, O. and C. D. Bopp: A Summary of the Effect of Radiation on Some Plastics and Elastomers, ASTM Special Technical Publication No. 208, 1, 123 (1957).

Crosslinking proceeds at a relatively fast rate in the repeating unit of polyethylene. There is no evidence of cleavage. The ethylene structure has about the same resistance to cleavage, but is ranked below the polyethylene unit because it was thought the unsaturation would impart some added stability.

The next structure is that of the phenol formaldehyde polymer. It is thought that the presence of the benzene ring in the main chain increases cleavage since unfilled phenolic crumbles for exposures which do not decrease the strength of polyethylene. This is in contrast with the polystyrene structure which has the benzene ring as a side group.

The remaining structures suffer from increasing susceptibility to cleavage. Some polyester materials have been softened by radiation with a few being subsequently hardened after an initial softening. Greatly increased tensile strength is found in some of these materials for moderate exposures.

Polyvinyl chloride is predominantly cleaved. It is softened and hydrogen chloride is produced. Very marked changes are observed at radiation exposures that do not cause much change in any of the previous structures.

Polymers having quaternary carbon atoms as shown in the last structure of Figure 3, page 15, are depicted as being the most easily cleaved. However, very rigid materials of this nature have high radiation stability that is attributed as an inherent function of their rigidity.

Effect of Gamma Radiation on Organic Protective Coatings

A very limited amount of information is available in the literature about the effect of radiation on organic protective coatings. However, some of the information available as pertains to plastics should also be applicable to protective coatings as both may be formed from similar polymers.

Sisman and Bopp (38) have reported that phenolic polymers may be protected from radiation effects by the addition of inorganic fillers. A paper and an asbestos filler was used in their work. The paper-filled plastic was not appreciably better than the unfilled material but the asbestos-filled material had greatly improved radiation stability.

Sisman (37) reports that materials which harden under irradiation often show an increase in tensile strength and a decrease in impact strength. Materials which show an initial increase in tensile strength will show a decrease under prolonged irradiation. The decrease is due to cleavage that becomes predominant with longer irradiation. Materials that soften under irradiation will decrease in tensile strength but may retain their impact strength.

Sisman (37) also states that the atmosphere in which a material is irradiated is very important as there may be some radiation-induced reaction in the atmosphere. If oxygen is present, oxidation is usually increased. Oxygen is converted to ozone by radiation, so ozone damage may be very severe. Nitric acid may be formed by nitrogen and oxygen in the air combining with water vapor that is present. Water absorption for many materials is increased when they are irradiated, causing them to suffer a larger effect when moisture is present. Most of these effects are surface effects and may be the controlling factor for thin specimens.

Horrocks (18) reports that as a result of gamma irradiation, polymers usually exhibit an increase or decrease in molecular weight and either become embrittled or more fluid. He also reports that the gamma radiation resistance of polymers is increased by increasing electron mobility and/or oxidation stability; that in general, the molecules in an aromatic system are comparatively stable because of high electron mobility; and that in some cases a pigment may protect a polymer by increasing its oxidation stability.

III. EXPERIMENTAL

Purpose of Investigation

The purpose of this investigation was to prepare pigmented and non-pigmented organic protective coatings and to irradiate them with gamma rays. The coatings received a constant irradiation dose of eight million rads. Seven tests were performed on the various coatings. The data from the tests were tabulated so that a direct comparison could be made as to the effect of the radiation on the coatings. The data from the tests also enabled comparisons to be made to determine if the various pigments had imparted any specific properties that would be desirable in coatings being exposed to radiations.

Plan of Experimentation

The resins used for this investigation were film formers that are commonly used in the paint industry. They were: (1) A vinyl chloride copolymer resin, (2) a phenolic resin modified with tung and linseed oil, and (3) an alkyd resin modified with soya oil.

Vinyl Chloride Copolymer Resin. (28, 34) This resin is a patented invention in which vinyl chloride, 55 to 75 per cent, is copolymerized with (1) a dihydrogen maleate, chloromaleate, or fumarate, or mixture of esters of this type, 14 to 35 per cent, (2) a monohydrogen monoalkyl maleate, chloromaleate, furmarate, or mixture of esters of this type, 5 to 10 per cent, and (3) trichloroethylene, 1.5 to 6.5 per cent. These resins have good solubility in hydrocarbon solvents, are compatible with alkyd resins, and have good adhesion to metals. They also have good hardness, good resistance to aging, and are particularly applicable to coatings such as paints for outdoor-exposed metal equipment.

Modified Phenolic Resin. This resin was obtained in a spar varnish. It was a trade sales product that was ready for use. The film former consisted of a phenolic resin, 11.0 per cent; modified with linseed oil, 14.7 per cent; and tung oil, 29.3 per cent. A drier was present and mineral spirits were used as the solvent. The spar varnish is recommended (6) for use to protect woodwork exposed to weather, excessive moisture, or strong, direct sunlight; or interior surfaces subjected to abnormal heat, light, or moisture.

Alkyd Resin. (27) Soya oil, 63 parts, and glycerine, 15 parts, were first reacted to form a monoglyceride in the manufacture of this resin. The monoglyceride was reacted with phthalic acid, 25 parts, and maleic acid, 1.5 parts, until an acid value of 6 to 10 was reached. This is a general purpose resin and can be used for baking or air-drying enamels.

The pigments used in conjunction with the above resins were: (1) Titanium dioxide, (2) zinc oxide, and (3) lead carbonate. Aluminum paste and carbon black were also used as pigments with the spar varnish to see if they might be of any special significance to a film that is being irradiated. Titanium dioxide, zinc oxide, and lead carbonate as pigments are commonly used in coatings, but are usually used as a part of a combination of pigments. However, in this investigation, they were used separately so as to isolate any special property that they might give the film.

<u>Titanium Dioxide</u>.(21) This is a brilliant white pigment that is acid and alkali resistant, and is inert to all paint, varnish, and lacquer ingredients. It is extensively used in paints, finishes, coatings, lacquers, and enamels.

<u>Zinc Oxide</u>.(20) This pigment consists of very fine particles with a wide range of shapes. It is one of the whitest pigments, will not yellow, is opaque to ultraviolet light, is not discolored by sulphur gases, and is used as a preventive for mildewing. It is also used to harden paint films, and to control chalking, checking, and dirt retention.

<u>Lead Carbonate</u>.(19) This is a reactive pigment that forms an opaque white film having excellent durability and water resistance. It reacts with the vehicle breakdown

components in the film to form lead soaps that stabilize the film.

Aluminum Paste. (5, 22) Aluminum pigments consist of extremely small, thin flakes of highly polished metal. They are made by ball milling atomized aluminum in such a way as to forge or hammer the grains of the powder into minute leaflike particles.

A combination of several properties make aluminum pigments valuable. Their lamellar structure and coating of an adherent organic film due to the method of manufacture causes the pigment particles to lie horizontally and concentrate in the outer part of the film. This "leafing" action lengthens the path that moisture must traverse to get through the film. In addition, aluminum pigments excludes ultra-violet light. Aluminum is usually used as a pigment in paints that are to be applied over a primer coat.

Carbon Black. (5) This pigment has a laminar structure that is valuable for increasing moisture impermeability and to ease local mechanical stresses. It is usually made from petroleum products and is known as "lampblack". One important property of lampblack is its fine particle size which is responsible for its intense blackness. Special properties may be given the lampblack by varying its volatile content (combined oxygen and hydrogen) to control its pH.

Each of the previously mentioned resins was used as the film former for a test series of protective coatings. Each series consisted of a clear varnish, an enamel pigmented with titanium dioxide, an enamel pigmented with zinc oxide, and an enamel pigmented with lead carbonate. The coatings were brushed on tin-plated sheet-steel test panels.

A portion of each series of coatings was irradiated with gamma rays, then tests performed on both the irradiated and non-irradiated panels. The non-irradiated panels were used for control, as a basis of comparison for the irradiated panels.

Seven tests were performed on the irradiated and non-irradiated coatings. The tests were: (1) Light reflectivity before and after irradiation, (2) alkali resistance, (3) acid resistance, (4) flexibility, (5) abrasion resistance, (6) scratch hardness, and (7) impact resistance. The panels used for the impact test were also used to rate the adhesion of the coating to the panel. These tests are all standard performance tests that are used to evaluate protective coatings.

The results of these tests were compared to determine the effect of the irradiation on the coatings and the effect of the different pigments on the radiation resistance of the coatings.

Materials

The following materials were used in this investigation. The specifications for use or purchase, the manufacturer or supplier, and the use of the material is listed.

Acetate, n-Butyl. Commercial grade, 90 to 92%; boiling range, 118°C (initial), 128°C (dry); median specific gravity at 20/20°C, 0.875. Manufactured by Carbide and Carbon Chemicals Co., New York, N. Y. Used as a solvent for Exon 470.

Acetone. Commercial grade; boiling range, 56°C (initial), 57°C (dry); median specific gravity at 20/20°C, 0.792. Manufactured by Carbide and Carbon Chemicals Co., New York, N. Y. Used as a solvent for Exon 470 and to clean paint brushes.

Aluminum Pigment. Standard Paste, No. 205. Specifications: Nonvolatile content, ave. 65.5%, max. 66%; average mesh size, 99.5% through 325; water covering value, max. 18,000 sq cm/gm; moisture content, normal 0.07%, max. 0.10%; approximate specific gravity, 1.47; bulking value, 0.0820 gallons per pound; average leafing value, 80% (3.5 gm sample), 65% (1.5 gm sample). Manufactured by Aluminum Co. of America, Pittsburg, Pa. Used as a pigment for one group of the test panels in the spar varnish series.

Carbonate of Magnesia. USP, blocks, 2 x 4 x 0.5 in. Manufactured for Wheeler and Huisking, Ltd., London, England. Used as color standards for the GE Recording Spectrometer.

Drier. Oronite, cobalt naphthenate, 6% cobalt. Manufactured by Naftone, Inc., New York, N. Y. Used as a drier for the alkyd resin.

Hydrochloric Acid. Meets ACS specifications, lot No. 90065; assay 36.5 to 38.0% HCl; specific gravity at 60/60°F, 1.185 to 1.192; residue after ignition, 0.0004%; sulfate (SO_4), 0.0001%; sulfite (SO_3), 0.0001%; free chlorine (Cl), 0.0001%; ammonium (NH_4), 0.0003%; arsenic (As), 0.000001%; heavy metals (as Pb), 0.0001%; iron (Fe), 0.00001%. Manufactured and distributed by J. T. Baker Chemical Co., Phillipsburg, N. J. Used for the acid resistance test on the coated test panels.

Ketone, Methyl Isobutyl. Commercial grade; boiling range, 114°C (initial), 117°C (dry); median specific gravity at 20/20°C, 0.802. Manufactured by Carbide and Carbon Chemicals Co., New York, N. Y. Used as a solvent for Exon 470.

Lampblack. Germantown, Eagle Brand. Manufactured by L. Martin Co., Inc., New York, N. Y. Used as a pigment for one group of the test panels in the spar varnish series.

Lead Carbonate. Powder, analytical reagent, meets ACS specifications. Maximum limits of impurities: Chloride (Cl), 0.002%; insoluble in acetic acid, 0.02%; iron (Fe), 0.005%; nitrate (NO_3), 0.005%; substances not ppted by H_2S (as sulfate), 0.20%; sulfate (SO_4), 0.005%; zinc and cadmium (as Zn), 0.005%; catalogue No. 5709. Obtained from Mallinckrodt Chemical Works, St. Louis, Mo. Used as a pigment in one group of each of the resin series.

Mineral Spirits. Bronoco Mineral Spirits--WR, boiling range, 315 to 400°F. Manufactured by R. J. Brown Co., St. Louis, Mo. Used as a solvent for the alkyd resin and to clean paint brushes.

Resin, Alkyd. No. 1155. Specifications: Viscosity, Y to Z; weight per gallon, 7.95 to 8.05 lbs.; color, 7 to 9; solids, 69 to 71%, (63 parts soya oil, 15 parts glycerine, 25 parts phthalic acid, 1.5 parts maleic acid); acid value of solids, 6 to 10; solvent, mineral spirits. Manufactured by Price Varnish Co., Valley Park, Mo. Used as the film former for one series of test panels.

Resin, Vinyl Chloride Copolymer. Exon 470. Specifications: White granular powder; specific gravity, 1.31; bulk density, 0.8 gms/cc (dry), 0.091 gal/lb (solution); relative viscosity, 1.35 (1% in cyclohexane). Manufactured by Firestone Tire and Rubber Co., Akron, Ohio. Used as a film former for two series of test panels.

Sodium Hydroxide. Certified Reagent, electrolytic pellets, lot No. 771325; assay 98.3% NaOH; iron (Fe), 0.0002%; sodium carbonate (Na_2CO_3), 0.3%; chloride (Cl), 0.0001%; sulfate (SO_4), 0.000%; phosphate (PO_4), 0.0000%; ammonium hydroxide ppt, 0.01%; heavy metals (as Ag), 0.0000%; Potassium (K), 0.000%; nitrogen compounds (N), 0.002%. Distributed by Fisher Scientific Co., Fair Lawn, N. J. Used to make 5% solution for the alkali resistance test on coated test panels.

Tape. Scotch, 3/4 in., transparent cellophane. Manufactured by Minnesota Mining and Manufacturing Co., Saint Paul, Minn. Used to secure bundles of test panels during shipment to and from, and during irradiation by Argonne National Laboratory.

Titanium Dioxide. Titanox-RA, rutile. Specifications: Titanium dioxide, 97% (min.); specific gravity, 4.2; bulking value, 0.0286 gallon per pound; particle size, 0.4 micron (ave. dia.); tinting strength, 1550; oil absorption, 19-20. Manufactured by Titanium Pigment Corp., New York, N. Y. Used as a pigment in one group of each of the resin series.

Toothpicks. Flat, white birch. Manufactured by Diamond Match Co., New York, N. Y. Used as spacers between the wrapped test panels during shipment to and from, and during irradiation by Argonne National Laboratory.

Varnish. Spar, "61". Composition: Phenolic Resin, 11.0%; linseed oil, 14.7%; tung oil, 29.3%; drier and mineral spirits, 45.0%. Manufactured by Pratt and Lambert, Inc., Buffalo, N. Y. Used as the film former for one series of test panels.

Water, Distilled. Obtained from distilled water tap, Organic Laboratory, Room 106, Chemical Engineering Building, Missouri School of Mines and Metallurgy, Rolla, Missouri. Used to make 5% sodium hydroxide solutions for alkali resistance tests.

Wrap. Saran, 1-1/2 in. x 25 ft. Manufactured by Dow Chemical Co., Midland, Michigan. Used as a wrap for individual test panels during shipment to and from, and during irradiation by Argonne National Laboratory.

Zinc Oxide. Powder, CP. Specifications: Insoluble in HCl; Mn, "nil"; Fe, 0.0005%; Cd and Pb, present; Cu, "nil"; Cl, 0.0230%; Loss on ignition, 0.175%. Manufactured by Baker and Adamson Chemical Co., Easton, Pa. Used as a pigment in one group of each of the resin series.

Apparatus

The following apparatus was used in this investigation. The specification for use or purchase, the manufacturer or supplier, and the use of the apparatus is listed.

Abrasion Tester. Available in MSM paint laboratory. Consists of a platform on which test panels can be mounted horizontally and a driving mechanism that causes a brush to be scrubbed back and forth across the coated surface of the panels. The brush had a face area of 1.25 x 3.25 in., contained 60 tufts, and weighed 62 grams. A counter is incorporated with the drive mechanism that counts each cycle of the brush (two trips across the panels by the brush). The driving mechanism is powered by an electric motor, type NS1-33 R, 110 v, ac, 60 cy, single phase, 0.010 hp, 37.5 rpm. Manufactured by Bodine Electric Co., Chicago, Ill. Used in performing an abrasion test on the coated test panels.

Balance. Analytical, chainomatic, MSM property No. 12656, weighing range, 0 to 100 gm, to nearest 0.1 mg. Manufactured by Christian Becker, Inc., New York, N. Y. Used to weigh test panels before and after application of the protective coatings, and before and after the abrasion test.

Balance. Triple beam, MSM property No. 13990, weighing range, 0 to 111 gm to nearest 0.01 gm. Obtained from Welch Scientific Co., Chicago, Ill. Used for weighing resin, pigment, and solvent.

Brush. Paint, 1 in., K-V 185 flagged tynex nylon bristles. Obtained from Ben Franklin Variety Store, Rolla, Mo. Used to brush coatings on test panels.

Can. Paint, 1/2 pt, D. T., code 51, No. 146. Manufactured by American Can Co., North Kansas City, Mo. Used for storage of varnish and enamel.

Can. Paint, 1/8 gal, D. T., code 51, No. 167. Manufactured by American Can Co., North Kansas City, Mo. Used for storage of varnish and enamel.

Can. Paint, 1 qt, D. T., code 51, No. 170. Manufactured by American Can Co., North Kansas City, Mo. Used for storage of resin.

Dissolver. Cowles, type 1VT, serial No. 0329553, 115 v, ac, 10.8 amp, 60 cy, single phase, 3450 rpm. Manufactured by Cowles Co., Cayuga, N. Y. Used to put the Exon 470 resin into solution.

Impact Tester. Variable impact, range of 2 to 30 in.-lb, 2 in.-lb graduations, catalogue No. 1660. Obtained from Gardner Laboratory, Inc., Bethesda, Md. Used to determine the impact resistance of the dry protective coatings on the metal test panels.

Mandrel Set. Set of nine steel mandrels, diameters of 1, 3/4, 1/2, 7/16, 3/8, 5/16, 1/4, 3/16, and 1/8 inch, with rigid frame, catalogue No. 1610. Obtained from Gardner Laboratory, Inc., Bethesda, Md. Used to determine the flexibility of the dry protective coatings on the metal test panels.

Mill. Morehouse, model A-200, serial No. 125, 115 v, ac, 14 amp, 60 cy, single phase, 3600 rpm. Manufactured by Morehouse Industries, Los Angeles, Calif. Used to disperse the pigments in the varnish or resin solutions.

Oven. Forced air drying, model OV530, serial No. JN 943, regulated temperature range, 100 to 650°F, 230 v, ac, 50/60 cy, 3.6 kw, single phase. Manufactured by Blue M Electric Co., Blue Island, Ill. Used to dry the coated test panels after an initial period of drying at room temperature.

Scratch Tester. Hoffman, range of 0 to 2400 gm, 25 gm graduations, catalogue No. 1750. Obtained from Gardner Laboratory, Inc., Bethesda, Md. Used to determine the scratch hardness of the dry protective coatings on the metal test panels.

Spectrometer. Recording, No. 2649773, 115 v, ac, 60 cy. Manufactured by General Electric Co., Schenectady, N. Y. Used to measure the light reflectance of the protective coatings on the test panels before and after irradiation.

Test Panels. Bright dry finish, coke tin plate, 30 to 31 gage, 2-3/8 x 8 in., catalogue No. 6345. Obtained from Gardner Laboratory, Inc., Bethesda, Md. Used as a substrate on which the protective coatings were applied for irradiation and testing.

Vials. Shell, 15 x 45 mm, short style. Manufactured by Kimble Glass Co., Toledo, Ohio. Used as covers on drops of reagent placed on test panels during the alkali and acid test.

Irradiation Facility

The coatings prepared for this investigation were sent to the High Level Gamma Irradiation Facility (1), Argonne National Laboratory, Lemont, Illinois, for irradiation.

The source of the gamma radiation for the facility is spent reactor fuel elements from the Materials Testing Reactor. Twelve of these elements are placed in an irradiation rack located on the floor of a canal and covered by sufficient water to protect personnel from the radiation. The water is maintained at a temperature of seventy-five degrees Fahrenheit. The rack is designed to provide irradiation sites in the spaces between the elements. Samples located within the rack are rotated slowly at two revolutions per minute to insure symmetrical exposure to the gamma rays.

Samples are sealed in thin-walled aluminum sample urns to prevent their contact with the canal water.

A minimum gamma flux of one million roentgens per hour is maintained at the interior irradiation sites of the facility. The important gamma energies range from 0.22 to 2.5 Mev, with an average energy of about 0.75 Mev. (39) The intensity of the source and the dosage received by samples are measured with a Fricke (ferrous sulfate) dosimeter. A change in the spectral characteristics of the chemical indicates the dosage it has received. The dosimeter is enclosed in a polystyrene cell when it is used in the facility.

Method of Procedure

The method of procedure for this investigation has been divided into eight general steps. They are: (1) Preparation of varnish, (2) preparation of enamels, (3) preparation of test panels for coating, (4) coating the test panels, (5) preparation of test panels for shipment and irradiation, (6) irradiation of test panels, (7) unwrapping the irradiated test panels, and (8) testing the panels. Each general step has been subdivided as necessary for complete presentation of the procedure. Unless otherwise specified, the steps of the procedure are applicable for each of the three test series of coatings.

Preparation of Varnish. Each of the varnishes used in this investigation was prepared in a different manner, so they will be discussed separately.

Vinyl Chloride Copolymer Varnish. The vinyl chloride copolymer resin was put into solution using the following amounts of solvent and resin:

Vinyl chloride copolymer resin	200	grams
Acetone	100	grams
Methyl isobutyl ketone	100	grams
Butyl acetate	200	grams

This proportion was found by reference to a sales service bulletin (8) and experimentation with different solutions for one with desirable brushability, leveling characteristics, and drying rate.

The resin was put into solution with the aid of the Cowles' dissolver. The methyl isobutyl ketone and butyl acetate were first transferred to a one-quart paint can. The mixer was lowered into the can and the dissolver started. The resin was added slowly over a period of about five minutes. The acetone was then added with mixing continuing for about three to five minutes until the resin was dissolved. The acetone was added last to cut down on its loss during mixing by evaporation. The resulting solution or varnish was allowed to set overnight before it was used so that the air bubbles caused by the mixing could escape.

This solution was reactive with the paint cans and would form dark spots on the inside of the cans after several days contact. For this reason, the solution was not used if it had been stored in the can for over ten or twelve days.

Spar Varnish. The spar varnish was a trade-sales product that came ready for use. It was used exactly as it was obtained in one-gallon cans. A portion of the varnish was usually transferred to a one-pint paint can for use as it skinned readily after being exposed to the air several times.

This prevented small jelled particles from being formed in the entire supply of the varnish.

Alkyd Varnish. The alkyd resin was available in a concentrated, viscous solution. A varnish was made by using the following amounts of concentrated resin solution, solvent, and drier:

Alkyd resin solution	300	milliliters
Mineral spirits	150	milliliters
Cobalt drier	2.2	milliliters

This proportion was found by experimentation with different solutions for one with desirable brushability, leveling characteristics, and drying rate.

The varnish was made by transferring the concentrated alkyd solution into a one-quart paint can. Two-thirds of the mineral spirits were mixed with the resin solution. A solution was made with the remaining one-third of the mineral spirits and the drier, and was added slowly to the resin solution with constant stirring with a stirring rod.

No skinning was noticed in this varnish during its use.

Preparation of Enamels. Some of the enamels used in this investigation were prepared in a slightly different manner or with a different resin solids-pigment ratio, so the preparation of each enamel is presented separately.

Vinyl Chloride Copolymer Enamel with Titanium Dioxide Pigment. This enamel was the vinyl chloride copolymer varnish pigmented with titanium dioxide. The weight ratio of resin solids to the pigment was nineteen to twelve. The following amounts of varnish and pigment were used to make the enamel.

Vinyl chloride copolymer varnish	253.9 grams
Titanium dioxide pigment	53.5 grams

The pigment was dispersed in the varnish by placing the varnish in a beaker and slowly adding the titanium dioxide with constant stirring. The pigment was mixed thoroughly with the varnish with a stirring rod, then the mixture was run once through the Morehouse mill. This dispersed the pigment adequately so that a smooth textured enamel was obtained.

This enamel was stored in a paint can for two or three weeks during the time of its use. No reaction with the can or skinning was noticed. The pigment stayed fairly well dispersed and the enamel could be made ready for use with a small amount of stirring.

Vinyl Chloride Copolymer Enamel with Zinc Oxide Pigment. This enamel was the vinyl chloride copolymer varnish pigmented with zinc oxide. The weight ratio of resin solids to pigment was nineteen to twelve. The following amounts of varnish and pigment were used to make the enamel.

Vinyl chloride copolymer varnish	125	grams
Zinc oxide pigment	26.3	grams

The pigment was dispersed in the varnish in the same manner as in the vinyl chloride copolymer enamel pigmented with titanium dioxide, page 35.

The zinc oxide pigment reacted with the resin, causing a firm, jelly-like substance to be formed after a few hours. It was necessary to apply the enamel immediately to the panels as the reaction made it unfit for use within a period of two hours after the time of mixing.

Vinyl Chloride Copolymer Enamel with Lead Carbonate Pigment. This enamel was the vinyl chloride copolymer varnish pigmented with lead carbonate. The weight ratio of resin solids to pigment was nineteen to twelve. The following amounts of varnish and pigment were used to make the enamel.

Vinyl chloride copolymer varnish	125	grams
Lead carbonate	26.3	grams

The pigment was dispersed in the varnish in the same manner as the vinyl chloride copolymer enamel pigmented with titanium dioxide, page 35.

The lead carbonate pigment also reacted with the resin, causing a jelly-like substance to be formed, although not to the same extent as the zinc oxide pigment. However, it was still necessary to apply the enamel immediately to the panels as it became unfit for use in a few hours after it was mixed.

Spar Enamel with Titanium Dioxide Pigment. This enamel was the spar varnish pigmented with titanium dioxide. The weight ratio of resin solids to pigment was nineteen to twelve. The following amounts of varnish and pigment were used to make the enamel.

Spar varnish	500 grams
Titanium dioxide	173.7 grams

The pigment was dispersed in the varnish by placing the varnish in a beaker, then adding the titanium dioxide slowly with constant stirring. The pigment was mixed thoroughly with the varnish with a stirring rod, then the mixture was run once through the Morehouse mill. This dispersed the pigment adequately so that a smooth textured enamel was obtained.

This enamel was stored in a paint can for two or three weeks during the time of its use. It skinned rather badly, but the skin was tough and could be removed without introducing many particles of the skin into the enamel. The pigment stayed very well dispersed and the enamel could be made ready for use with a small amount of stirring.

Spar Enamel with Zinc Oxide Pigment. This enamel was the spar varnish pigmented with zinc oxide. The weight ratio of resin solids to pigment was nineteen to twelve. The following amounts of varnish and pigment were used to make the enamel.

Spar varnish	300	grams
Zinc oxide	104.3	grams

The pigment was dispersed in the varnish in the same manner as the spar enamel with titanium dioxide pigment, page 37.

A skin was formed on this enamel during storage, but the skin was tough and easily removed. The pigment stayed dispersed very well.

Spar Enamel with Lead Carbonate Pigment. This enamel was the spar varnish pigmented with lead carbonate. The weight ratio of resin solids to pigment was nineteen to twelve. The following amounts of varnish and pigment were used to make the enamel.

Spar varnish	300	grams
Lead carbonate	104.3	grams

The pigment was dispersed in the varnish in the same manner as the spar enamel with titanium dioxide pigment, page 37.

A skin was formed on this enamel during storage, but the skin was tough and easily removed. The pigment settled rather rapidly and had to be stirred vigorously before and during use.

Spar Enamel with Aluminum Pigment. This enamel was the spar varnish pigmented with aluminum paste. The weight ratio of resin solids to paste was thirty-three to ten. Due to the large bulk of this pigment, it was not possible to use the same resin solids to paste ratio as in the other enamels. The ratio used gave an adequately pigmented film. The following amounts of varnish and paste were used to make the enamel.

Spar varnish	300 grams
Aluminum paste	50 grams

The paste was dispersed in the varnish in the same manner as the spar enamel with titanium dioxide pigment, page 37.

A skin was formed on this enamel during storage, but the skin was tough and easily removed. The pigment remained dispersed very well.

Spar Enamel with Lampblack Pigment. This enamel was the spar varnish pigmented with lampblack. The weight ratio of resin solids to pigment was thirty-three to ten. Due to the large bulk of this pigment, it was not possible to use the same resin solids to pigment ratio as in the other enamels. The ratio used gave an adequately pigmented film. The following amounts of varnish and pigment were used to make the enamel.

Spar varnish	300	grams
Lampblack	50	grams

The pigment was dispersed in the varnish in the same manner as the spar enamel with titanium dioxide pigment, page 37.

A skin was formed on this enamel during storage, but the skin was tough and easily removed. The pigment remained dispersed very well.

Alkyd Enamel with Titanium Dioxide Pigment. This enamel was the alkyd varnish pigmented with titanium dioxide. The weight ratio of resin solids to pigment was nineteen to twelve. The following amounts of materials were used to make the enamel.

Alkyd resin solution	250	milliliters
Mineral spirits	125	milliliters
Cobalt drier	1.85	milliliters
Titanium dioxide	106	grams

A varnish was made by combining the alkyd resin solution, mineral spirits, and drier in a similar manner as described for the alkyd varnish on page 34. The titanium dioxide was added to the varnish slowly with constant stirring. The pigment was mixed thoroughly with the varnish with a stirring rod, then the mixture was run once through the Morehouse mill. This dispersed the pigment adequately so that a smooth-textured enamel was obtained.

This enamel was stored in a paint can for two or three weeks during the time of its use. It skinned rather badly and the skin was very weak and difficult to remove without introducing small particles of the skin into the enamel. It was necessary to strain the enamel several times during its use to remove these particles.

Alkyd Enamel with Zinc Oxide Pigment. This enamel was the alkyd varnish pigmented with zinc oxide. The weight ratio of resin solids to pigment was nineteen to twelve. The following amounts of materials were used to make the enamel.

Alkyd resin solution	250	milliliters
Mineral spirits	125	milliliters
Cobalt drier	1.85	milliliters
Zinc oxide	106	grams

The enamel was made in the same manner as the alkyd enamel pigmented with titanium dioxide, page 40.

This enamel skinned rather badly, and the skin was difficult to remove. It was necessary to strain the enamel several times during its use to remove small particles of the skin.

Alkyd Enamel with Lead Carbonate Pigment. This enamel was the alkyd varnish pigmented with lead carbonate. The weight ratio of resin solids to pigment was nineteen to twelve. The following amounts of materials were used to make the enamel.

Alkyd resin solution	250	milliliters
Mineral spirits	125	milliliters
Cobalt drier	1.85	milliliters
Lead carbonate	106	grams

The enamel was made in the same manner as the alkyd enamel pigmented with titanium dioxide, page 40.

This enamel skinned rather badly, and the skin was difficult to remove. It was necessary to strain the enamel several times during its use to remove small particles of the skin.

Preparation of Test Panels for Coating. It was necessary to do the following items of work on the test panels to prepare them for coating. The items are listed in the sequence in which they were performed.

(1) One hole, one-fourth inch in diameter, was drilled in each panel. The hole was centered between the sides and was one-half inch from an end of the panel. The

panels were hung on nails through these holes when they were not in use.

(2) The panels were examined individually for sharp projection of the edges. These were ground smooth with an emery grinder.

(3) A different number was stamped on each panel with metal dies. This number was located in the upper right corner of the panels (adjacent to the drilled hole in the panel). The numbers on the panels for this investigation ran consecutively from 25 to 300.

(4) Straight lines were scratched on both sides of the panels to serve as upper boundary lines for the coating. These lines were parallel to the ends and were six inches from the end opposite the hole that was drilled in the panel.

(5) The panels were wiped clean with a cloth dampened in acetone. After this, care was taken to handle the panels in the area around the holes and above the coating boundary line.

(6) The panels were hung individually on a line to let all traces of the acetone evaporate from them.

(7) The panels were weighed on the chainomatic balance to the nearest milligram and the weight recorded.

Coating the Test Panels. It was decided at the beginning of this investigation that the thickness of the coatings used for the study would be five mils. This is a normal film thickness specified for most protective coatings.

The coating was applied with a brush to both sides of the panels. The upper boundary line on the panels served as a guide so that equal areas on all the panels were coated.

The freshly coated panels were placed in a horizontal position to dry for approximately twelve hours. This allowed the coating to dry with a uniform thickness. The panels were then hung in a verticle position on a line with clothespins to dry for a varying length of time, depending on the coating. After a short period of drying in the oven, the panels were ready for another brush application of the coating.

The approximate average film thickness per brush application was one mil. It was found that thicker applications would cause the film to build up at the panel edges and either prolong the drying time of the film or cause it to wrinkle.

A summary of the painting schedule for the panels is shown in Table II, page 45.

Preparation of Test Panels for Shipment and Irradiation. The total thickness of the coatings on the test panels varied somewhat due to the method of application. This was taken into consideration when the panels were being chosen for irradiation and for control so that the tests to be performed later would be on panels with an approximate equal film thickness. The first step in choosing the panels was to calculate the film thickness on each panel. The panels

TABLE II

Summary of Painting Schedule

Film Former	Pigment	Minimum Drying Time Between Coats Horizontal Position	Minimum Drying Time Between Coats Vertical Position	Oven Drying Time Between Coats	Oven Temp	Number of Brush Applications for 5 mil Thickness
		hr	hr	hr	°F	
Vinyl Chloride Copolymer	none	12	0	1-2	125	2
	TiO_2	12	0	1-2	125	4
	ZnO	12	0	1-2	125	4
	$PbCO_3$	12	0	1-2	125	3
Spar Varnish	none	12	60	2-4	100	4
	TiO_2	12	60	2-4	100	5
	ZnO	12	60	2-4	100	5
	$PbCO_3$	12	60	2-4	100	6
	Al	12	60	2-4	100	4
	C	12	60	2-4	100	3
Alkyd Resin	Clear	12	60	4-6	100	5
	TiO_2	12	60	4-6	100	6
	ZnO	12	60	4-6	100	7
	$PbCO_3$	12	60	4-6	100	6

were divided into two groups. The panels in each group were given a number designation to indicate the group to which they belonged, and a letter designation to indicate a comparative film thickness. The letter was assigned on the basis of the weight of coating on the panel which was directly proportional to the film thickness. (The weight of the coating was a better basis, as the film thickness was calculated for only two significant figures.) Thus a panel designated A-1 belonged in group-one and would have an approximate equal film thickness to panel A-2 which belonged in group-two. Reference to Table III, page 47, will help to clarify this procedure. It may be seen from columns four and six that panels A-1 and A-2 have the heaviest coatings, B-1 and B-2, the next-heaviest coatings, and so forth. Also, it may be noted that the panel number (column one) on a group-one panel is always less than the number on the corresponding group-two panel. This made it convenient to differentiate between irradiated and non-irradiated panels during testing. The test that was performed on the panels is indicated in column seven.

The panels with each of the types of coatings in the different test series were grouped in this manner. One group of the panels were kept for control and the other group was sent for irradiation.

It was anticipated that during the irradiation of the test panels, the air atmosphere present could absorb radiation, undergo chemical changes (oxygen to ozone or nitrogen, oxygen, and water vapor to nitric acid) and cause effects that might later be thought due to the

TABLE III

Film Thickness, Panel Designation, and Test Performed on Test Panels Coated with Spar Enamel with Titanium Dioxide Pigment

Panel No.	Weight of Panel w/Enamel	Weight of Panel wo/Enamel	Weight of Enamel	Film Thickness	Panel Designation	Test Performed
	gm	gm	gm	mil		
1	2	3	4	5	6	7
139	29.967	24.950	5.02	4.9	J-1	none
140	30.086	24.961	5.12	4.9	H-1	none
141	29.550	24.555	4.99	4.8	J-2	none
142[a]	30.094	24.968	5.13	4.9	G-1	none
143	30.617	25.036	5.58	5.4	A-1	alkali
144[a]	30.470	24.927	5.54	5.3	B-1	acid
145	30.524	25.078	5.45	5.3	C-1	abrasion
146	30.352	24.683	5.67	5.5	A-2	alkali
147[a]	30.602	25.157	5.44	5.2	D-1	flexibility
148[a]	30.458	25.224	5.23	5.0	G-2	none
149	30.510	25.114	5.40	5.2	E-1	scratch
150	30.542	25.185	5.36	5.2	E-2	scratch
151	30.321	25.225	5.10	4.9	H-2	none
152[a]	30.548	25.138	5.41	5.2	D-2	flexibility
153	30.633	25.119	5.51	5.3	C-2	abrasion
154	30.413	25.052	5.36	5.2	F-1	impact
155	30.341	25.018	5.32	5.1	F-2	impact
156[a]	30.694	25.133	5.56	5.4	B-2	acid

[a] Panels on which light reflectivity tests were made.

radiation. To prevent any such products from reaching the coatings, the panels to be irradiated were wrapped individually in saran wrap.

The protection given the panels by the saran wrap was checked by including an extra group in the first test panel series for irradiation. This extra group was irradiated in the same manner as the other groups except that it was in an inert (argon) atmosphere during irradiation. It was then possible to compare the radiation effect on the panels irradiated in the air atmosphere with the ones irradiated in the argon atmosphere.

The saran wrap also kept the panels from sticking together in case the coatings softened during irradiation or from heating that might be caused by the irradiation. Toothpick spacers were included between the panels to allow any heat generated to be easily dissipated.

Twenty-seven to thirty-six panels were bound together in bundles for irradiation. The bundles were fastened securely with scotch tape. The thickness of the bundles was limited to a size that would fit into a number-two can (three inches in diameter).

The panels were packed carefully in a corrugated cardboard box to prevent them from being damaged during shipment to and from the gamma irradiation facility.

Irradiation of Test Panels. The coated test panels were sent for irradiation to the High Level Gamma Irradiation Facility, Argonne National Laboratory, Lemont, Illinois.

Table IV, page 49, shows the irradiation time and dosages received by the coated test panels sent to the facility for irradiation. The

TABLE IV

Irradiation Data on Panels Irradiated at Argonne National Laboratory

TABLE IV

Irradiation Data on Panels Irradiated at Argonne National Laboratory

Panel	Description		Number	Irradiation					
			of		Rate			Dosage	
Group	Film Former	Pigment	Panels	Date	Top	Bottom	Time	Top	Bottom
					rad/min		min	rad	
No. 1	Vinyl chloride copolymer	none	8	4-8-58	3.07×10^4	3.08×10^4	260	7.98×10^6	8.01×10^6
		TiO_2	8	"	"	"	"	"	"
		ZnO	8	"	"	"	"	"	"
		$PbCO_3$	8	"	"	"	"	"	"
No. 2[a]	Vinyl chloride copolymer	none	8	4-9-58	2.98×10^4	2.99×10^4	268	7.99×10^6	8.01×10^6
		TiO_2	8	"	"	"	"	"	"
		ZnO	8	"	"	"	"	"	"
		$PbCO_3$	8	"	"	"	"	"	"
No. 3	Spar varnish	none	9	5-20-58	2.23×10^4	2.27×10^4	356	7.94×10^6	8.08×10^6
		TiO_2	9	"	"	"	"	"	"
		ZnO	9	"	"	"	"	"	"
No. 4	Spar varnish	$PbCO_3$	9	5-21-58	2.23×10^4	2.27×10^4	356	7.94×10^6	8.08×10^6
		Al	9	"	"	"	"	"	"
		C	9	"	"	"	"	"	"
No. 5	Alkyd resin	none	9	6-4-58	0.79×10^4	0.77×10^4	1538	8.1×10^6	7.9×10^6
		TiO_2	9	"	"	"	"	"	"
No. 6	Alkyd resin	ZnO	9	6-4-58	0.79×10^4	0.77×10^4	1538	8.1×10^6	7.9×10^6
		$PbCO_3$	9	"	"	"	"	"	"

[a] Panel Group irradiated in inert (argon) atmosphere.

panels were too long to be enclosed in one number-two can, so they occupied a position normally used for the irradiation of two different samples. Because of this, each end of the panels received a slightly different dosage of gamma rays.

Unwrapping the Test Panels. When the panels were returned from being irradiated, they were unwrapped and inspected closely. Each group was compared individually with its corresponding non-irradiated group to detect any visible damage or color change in the film.

Testing the Panels. Seven tests were performed on the irradiated and non-irradiated test panels. The tests were: (1) Light reflectivity before and after irradiation, (2) alkali resistance, (3) acid resistance, (4) flexibility, (5) abrasion resistance, (6) scratch hardness, and (7) impact resistance. The procedure for each of the tests is discussed separately. A short description of the test and apparatus is included with the procedure. Each test was performed on one irradiated and one non-irradiated test panel from each type of coating in the three film former series, unless otherwise stated. Reference to Table III, page 47, will show the order in which the panels were chosen for the tests in the titanium dioxide group of the spar varnish series. The same order was followed throughout the entire investigation.

Light Reflectivity.(33) The recording spectrometer was used for making spectral-reflectance measurements of irradiated and non-irradiated protective coatings on the test panels. The device consists essentially of a double-prism monochromator equipped with an automatic slit adjustment for a ten-millimicron wavelength band; a polarization-type photoelectric photometer; and a recording mechanism.

The wavelength range of the light for the test was 400 to 700 millimicrons. The results of the test were obtained in the form of a graphic record.

The general operation procedure was to place two carbonate-of-magnesia standards in the sample and standard positions on the spectrometer. A sheet of graph paper manufactured for use with the machine was placed on the recording cylinder. The power switch for the machine and the light source was turned on, then after about a minute had elapsed, the balance motor was turned on. The graph paper, recorder, and monochromator were all set so that the recording pen rested on the graph paper at the point, 400 millimicrons wavelength and 100 per cent reflectance.

The carbonate-of magnesia standard at the sample position was replaced with a coated test panel. The recording mechanism was then started. This set the machine into operation so that the entire wavelength range was covered and the per cent reflectance recorded in two and one-half minutes.

The reader is referred to the instruction manual used as a reference at the beginning of this procedure if more detailed information about the operation of the spectrometer is desired.

Six panels (see Table III, page 47) were chosen from each type of coating in the three test series for the light-reflectance tests. Three of these panels were for control, and three were for irradiation. Tests were made on the six panels before and after three of the group were irradiated. These tests showed the amount that the light reflectivity of the control panels had changed during the time that elapsed between the tests as well as the light reflectance change in the panels that were irradiated. This made it possible to determine if the change in the reflectance of the panels was due to aging or irradiation.

Reproductions of the reflectance tests representing each type of coating in the test series were made and are presented on pages 60 to 91. A better idea of this test may be had by referring to these figures.

Alkali Resistance.(32) The alkali-resistance test is frequently required for varnishes and may be used for pigmented coatings. The test is made with solutions of sodium hydroxide ranging from 0.5 to 10 per cent concentration. The test has many acceptable variations.

The alkali test for this investigation was made with approximately five per cent sodium hydroxide solution. The solution was made by weighing out 50.00 grams of sodium hydroxide, placing it in a one-liter volumetric flask, and filling the flask to the one-liter mark with distilled water.

The test was made by placing a drop of the alkali on the coated panels at specified time intervals. One dram shell vials were used to cover the drops of alkali to prevent them from evaporating and from spreading over the panel. At the end of a specified period, all the drops of alkali were washed off. The panels were allowed to dry for two hours. The visible effect of the alkali on the coating for the different time intervals was given a numerical rating. Two panels, one irradiated and one non-irradiated of each coating type, were tested and rated at the same time so that they could be compared.

The time interval and test periods for the various types of coatings may be seen by referring to Table V, page 54.

Acid Resistance.(10) Concentrated mineral-acid tests are frequently specified for coatings when it is desired to know the resistance of a coating to various reagents. Sulfuric, nitric, or hydrochloric acids may be used.

The acid test for this investigation was made with approximately twelve-normal hydrochloric acid solution. This concentration of acid was obtained directly from the bottle in which it was purchased.

TABLE V

Time Interval and Test Period for Alkali and Acid Test

		Alkali Test		Acid Test	
Film Former	Pigment	Interval	Period	Interval	Period
		min	hr	min	hr
Vinyl Chloride Copolymer	none	60	8.00	60	8.00
	TiO_2	"	"	"	"
	ZnO	"	"	"	"
	$PbCO_3$	"	"	"	"
Spar Varnish	none	15	2.25	15	3.25
	TiO_2	"	"	"	"
	ZnO	"	"	"	"
	$PbCO_3$	"	2.00	"	3.00
	Al	"	"	"	"
	C	"	"	"	"
Alkyd Resin	none	15	2.50	15	2.50
	TiO_2	"	"	"	"
	ZnO	"	"	"	"
	$PbCO_3$	"	"	"	"

The test was made by placing drops of the acid on the panels in a manner similar to the alkali resistance test described on page 53. Reference may be made to Table V, page 54, for the time interval and test period for the acid resistance test.

Two panels, one irradiated and one non-irradiated of each coating type, were tested and rated at the same time so that they could be compared.

Flexibility. (29) The flexibility of a coating may be measured by bending a coated metal panel over a mandrel, and noting the size of the mandrel at which the coating begins to crack.

Flexibility tests for this investigation were made with a mandrel set consisting of nine mandrels, ranging from one-eighth to one inch in diameter. Most of the films tested had good flexibility, so the test panels were first tested with the smallest (one-eighth inch) mandrel. The coating was immediately inspected for any cracking. The first test was performed toward one end of the coating so that other areas of the coating could be used for further tests. If the coating had cracked at any place along the bend, tests were continued with successively larger mandrels until the coating did not crack.

The data for this test were recorded as the smallest size mandrel over which the coating could be bent without any cracking appearing in the film. If more than one panel was needed for the test, others were used that have "none" listed as the test performed on them as in column seven, Table III, page 47.

Abrasion Resistance.(31) Abrasion resistance is a measure of the toughness of a coating or its resistance to wear. Extremely hard coatings usually have less resistance to wear than softer, rubbery coatings.

The abrasion tester used in this investigation consisted of an apparatus that caused a brush to be rubbed back and forth across the coated surfaces of the test panels. The brush weighed 62 grams. It was rubbed back and forth across the panel at 37.5 cycles per minute in a semicircular path approximately two feet in diameter. Panels were placed perpendicular to the path so that each would have the same area exposed to the path.

Two panels with one type of coating, one irradiated and one non-irradiated, were tested concurrently. The panels were weighed to the nearest one-tenth of a milligram before and after the test. The weight loss of the panel and the number of cycles of abrasion to which it had been subjected was recorded.

The majority of the panels were subjected to about 14,500 abrasion cycles. Some coatings with high abrasion resistance were subjected to many more than this in order that a significant weight difference might be obtained.

Scratch Hardness.(9) Scratch hardness is a measure of the physical hardness or mar resistance of a coating.

The Hoffman scratch hardness tester was used to determine the hardness of the films. The tester is a small portable device. It consists of a carriage, riding on four wheels, that has an extension on one end on which a scratching tool is arranged. The tool is fixed to one end of a beam that is fulcrumed to the extension. The scratching tool rests on the test surface when the four wheels of the carriage are pressed against the surface. The load on the tool is adjusted by means of a rider on a beam.

The tests were made on the panels by using a constant load on the beam and noting the difference in the type of scratch that was made on the coatings. The scratch was examined with a reading glass, and a description of the scratch and the beam load were recorded. The beam load was determined by trial and error so that different types of scratches were made on a series of panels.

Impact Resistance.(30) The impact resistance test measures a combination of toughness and adhesion of coatings. The impact tester used in this investigation was the falling-weight type. It consisted essentially of a weight that could be dropped on the panels from different heights, the height of the drop corresponding to the amount of impact. The falling weight causes the side of the panel to bulge out at the point of impact. The height is adjusted until the coating fails, the failure indicated by cracks radiating from the point of impact.

The tests were made to determine the impact for which the coating on the panels failed. The least impact that would cause failure was recorded.

An indication of the relative adhesion of the coating to the panels was obtained by noting the ease with which the coating could be removed from the bulged-out area on the panel with a knife.

Data and Results

The data and results obtained by this investigation for the different types of coatings have been grouped according to the test performed on the coating, and are presented under that heading.

Light Reflectivity. The panels to be irradiated were tested before and after irradiation. The test panels used for control were tested at the same time.

The results of the light reflectivity tests are presented in Figures 4 to 35, pages 60 to 91. These are copies of the graphs that were obtained from the recording spectrophotometer. The curves show the per cent reflectance of the coatings.

The figures are presented in groups so that they may be observed more easily. The tests for one certain type of coating are presented consecutively. For example, Figures 4, 5, and 6, pages 60, 61, and 62, are the light reflectance tests on the clear vinyl chloride varnish. The first figure in this group (Figure 4) is the light reflectance test on the control panel. The solid curve represents the reflectance of the coating when the panel was first tested. The dotte curve represents the reflectance of the same coating after it had age until the other panels in the group had been irradiated. The second figure in the group (Figure 5) is the light reflectance test on the coating that was irradiated in an air atmosphere. The solid curve represents the reflectance of the coating before irradiation, and the dotted curve represents the reflectance of the same coating after it was irradiated. The last figure in the group (Figure 6) is the light reflectance test on the coating that was irradiated in an argon atmosphere. The solid curve represents the reflectance of the coating before irradiation, and the dotted curve represents the reflectance of the same coating after it was irradiated.

The tests for the other types of coatings follow in the same order. The vinyl chloride copolymer series was the only one in which an inert atmosphere was used.

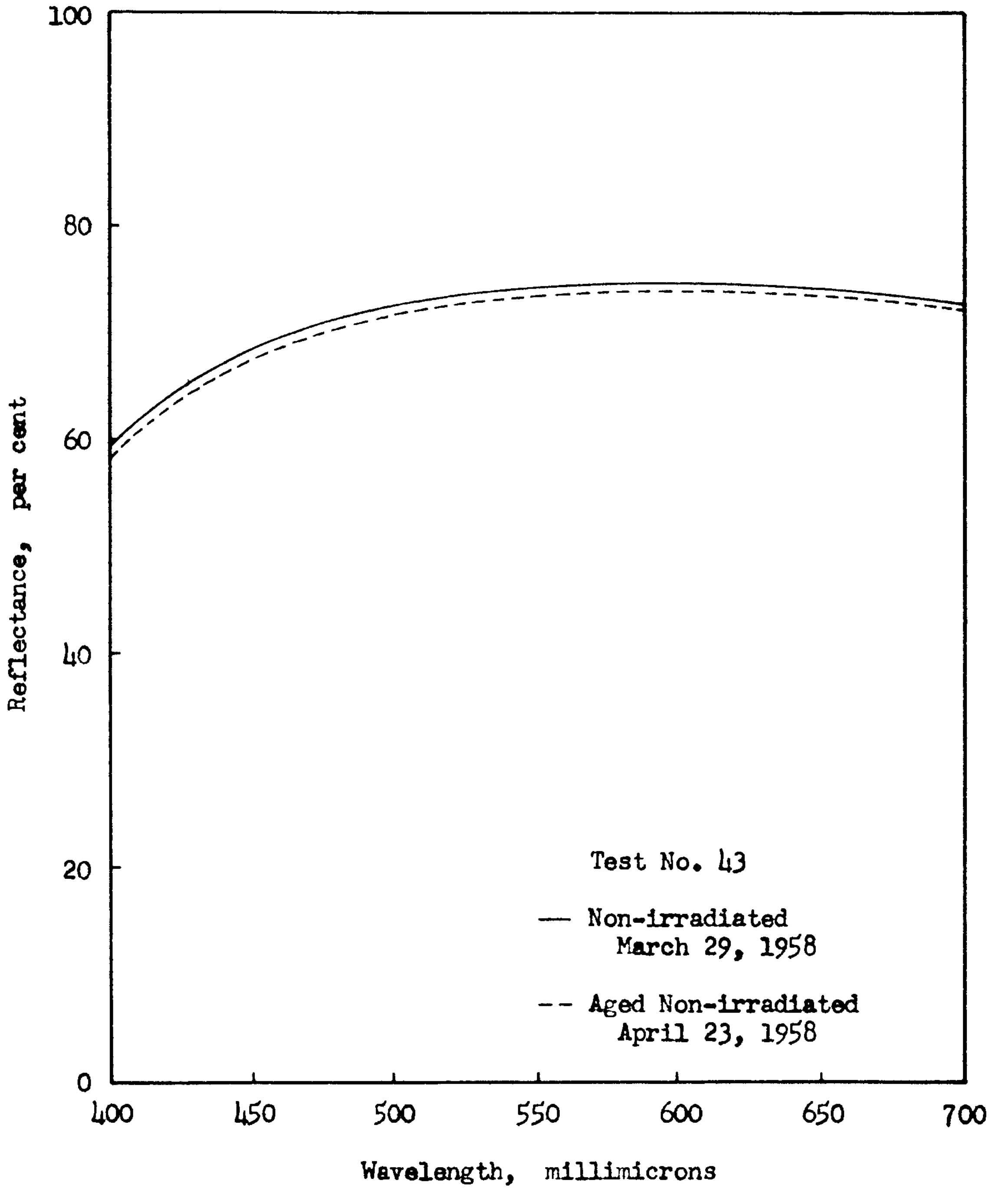

Figure 4. Light Reflectance of Clear Vinyl Chloride Copolymer Varnish.

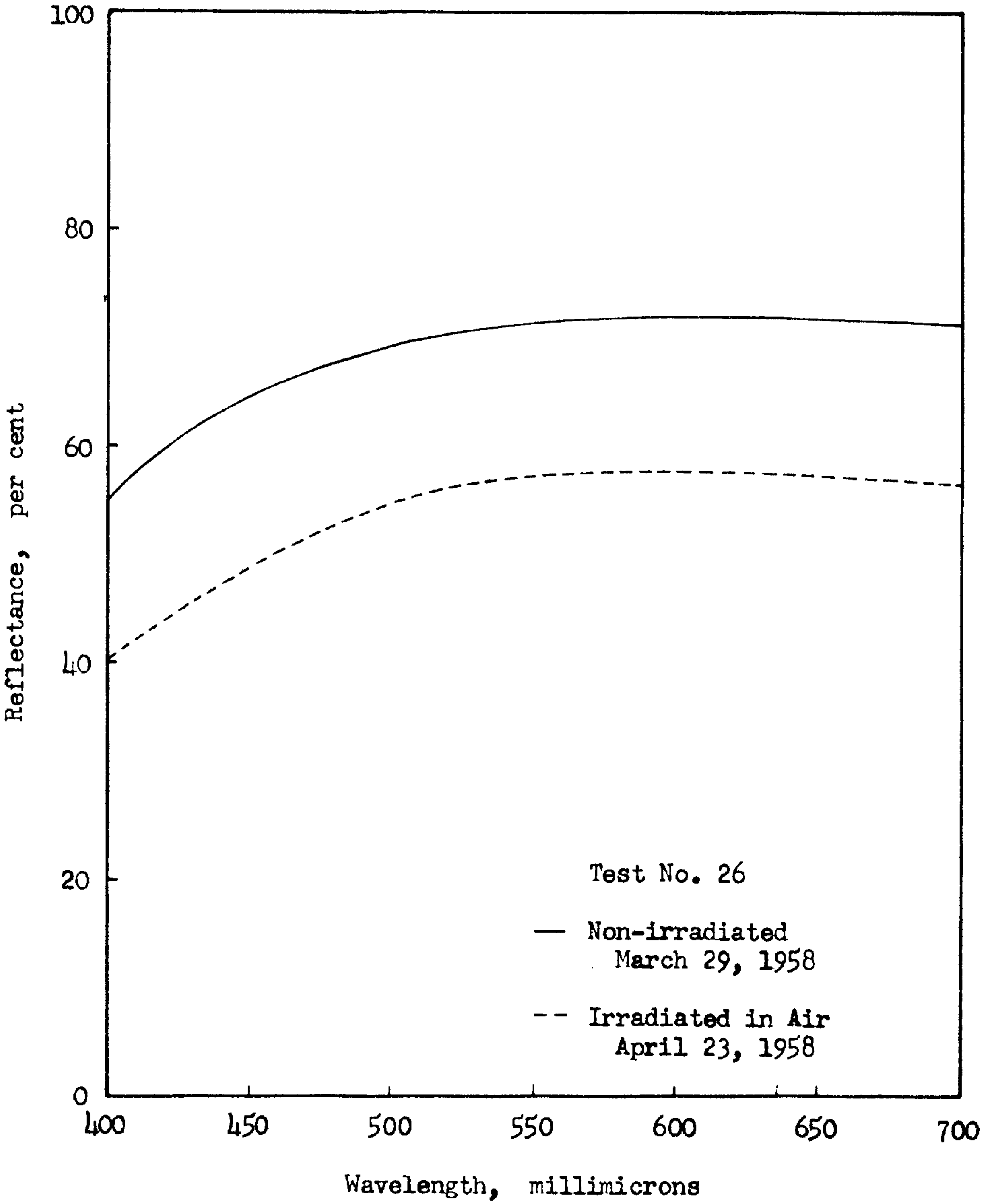

Figure 5. Light Reflectance of Clear Vinyl Chloride Copolymer Varnish.

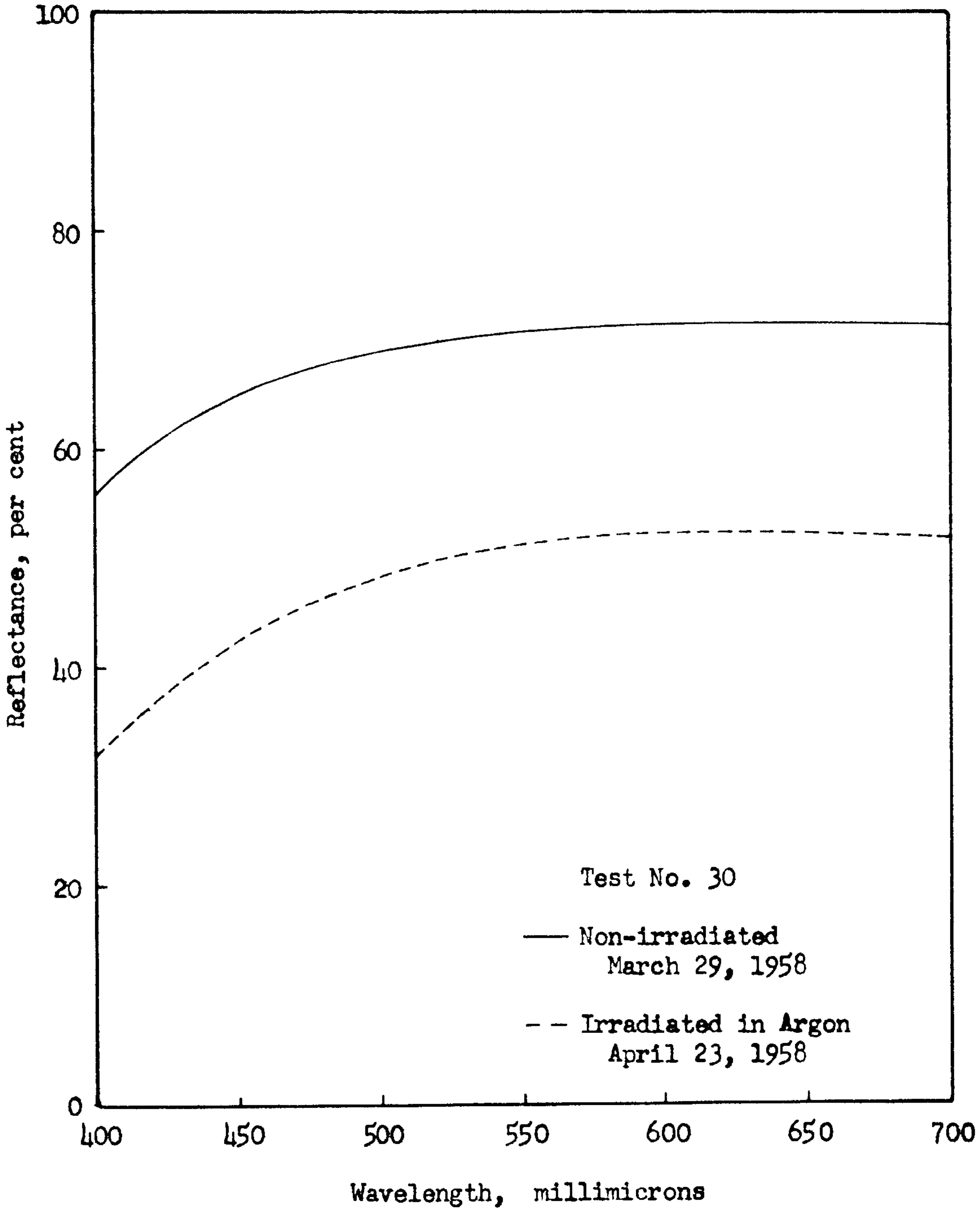

Figure 6. Light Reflectance of Clear Vinyl Chloride Copolymer Varnish.

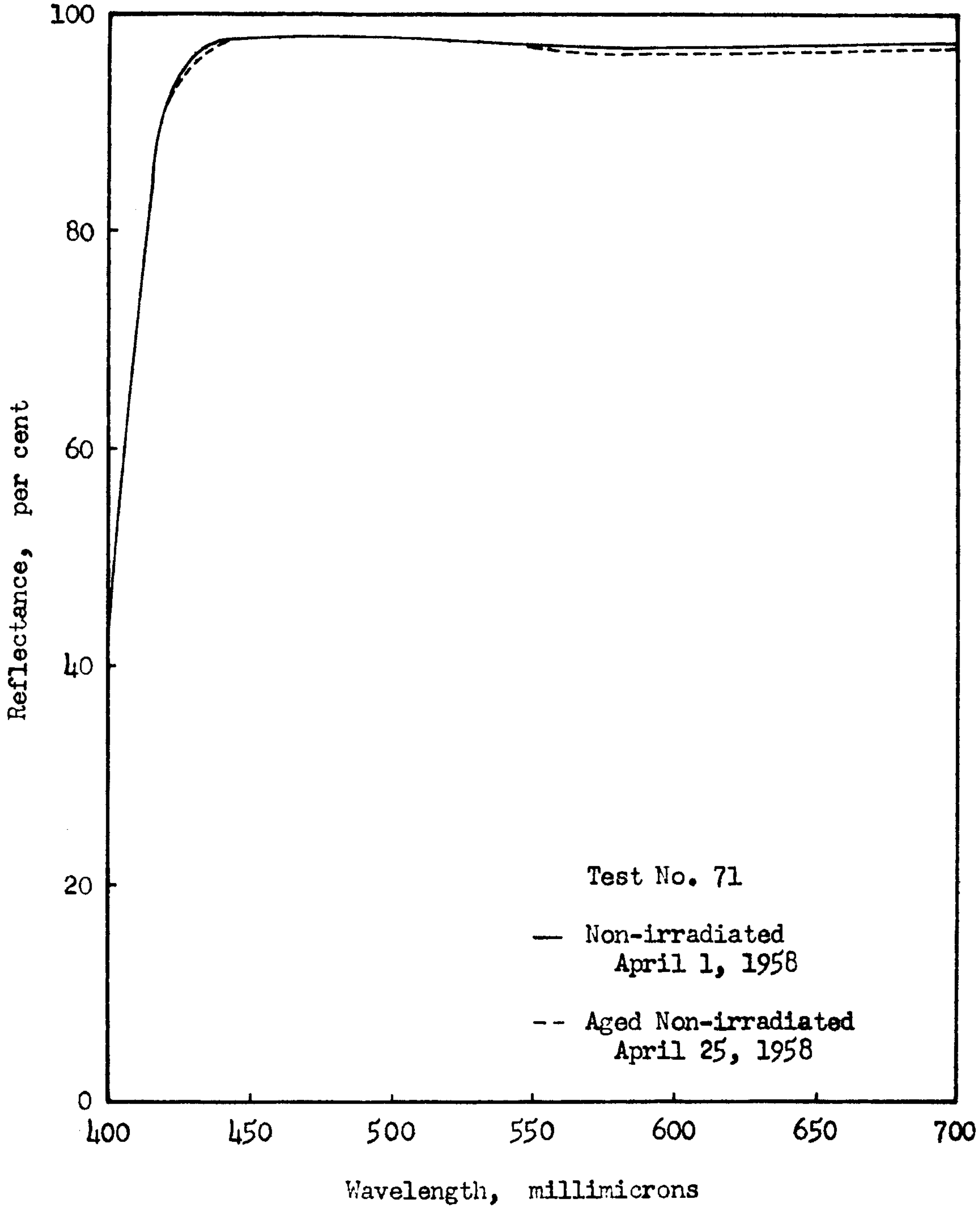

Figure 7. Light Reflectance of Vinyl Chloride Copolymer Enamel Pigmented with Titanium Dioxide.

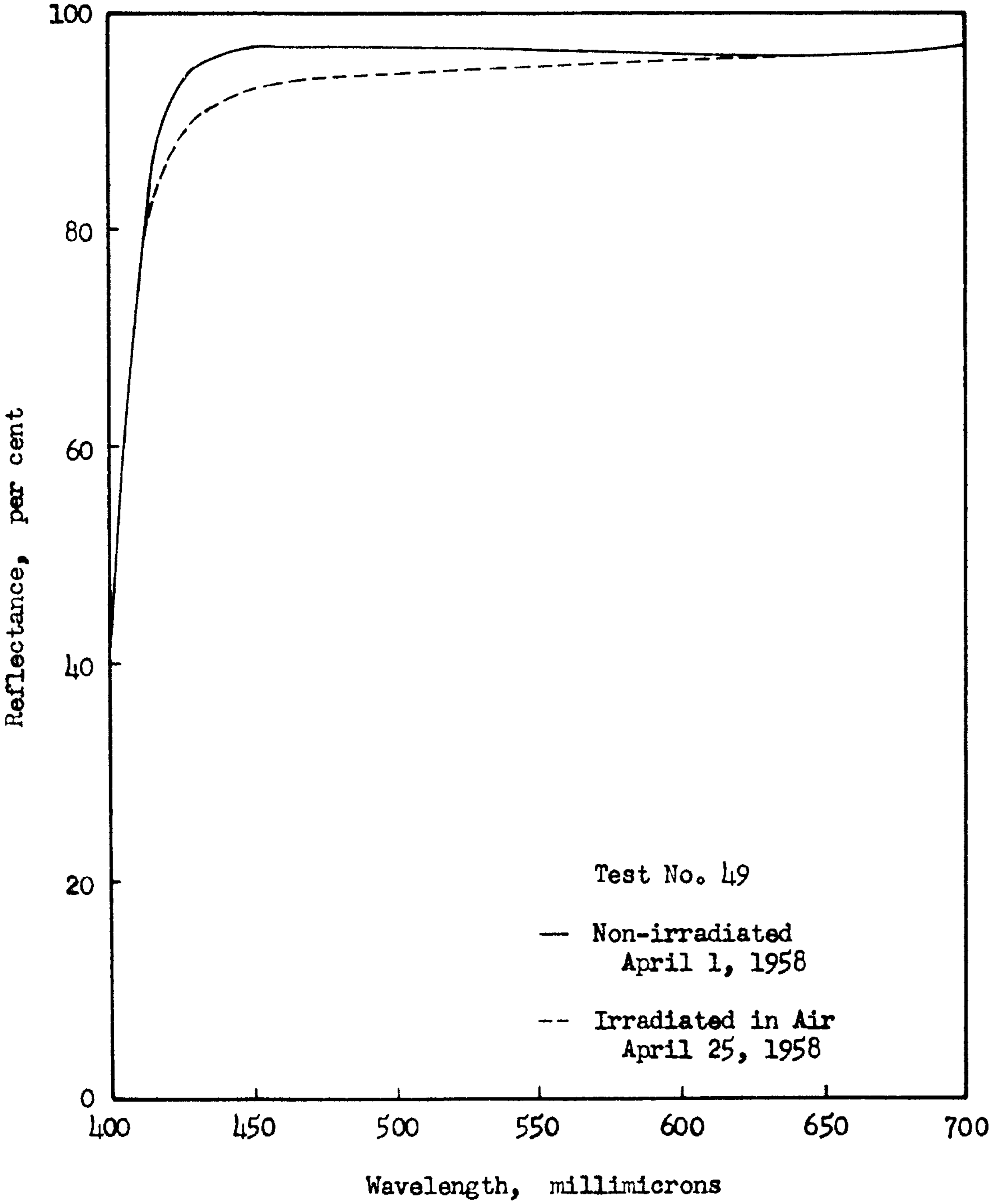

Figure 8. Light Reflectance of Vinyl Chloride Copolymer Enamel Pigmented with Titanium Dioxide.

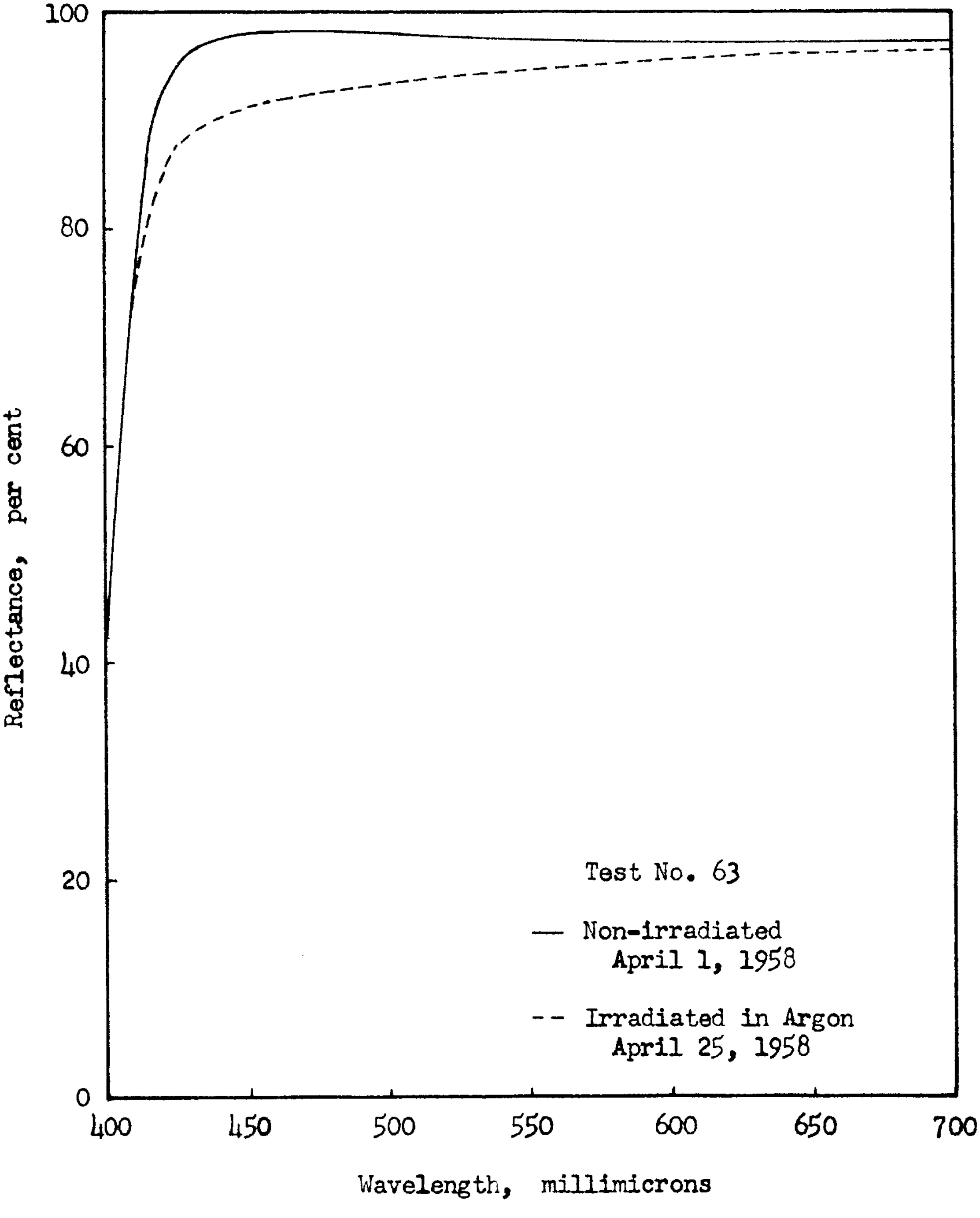

Figure 9. Light Reflectance of Vinyl Chloride Copolymer Enamel Pigmented with Titanium Dioxide.

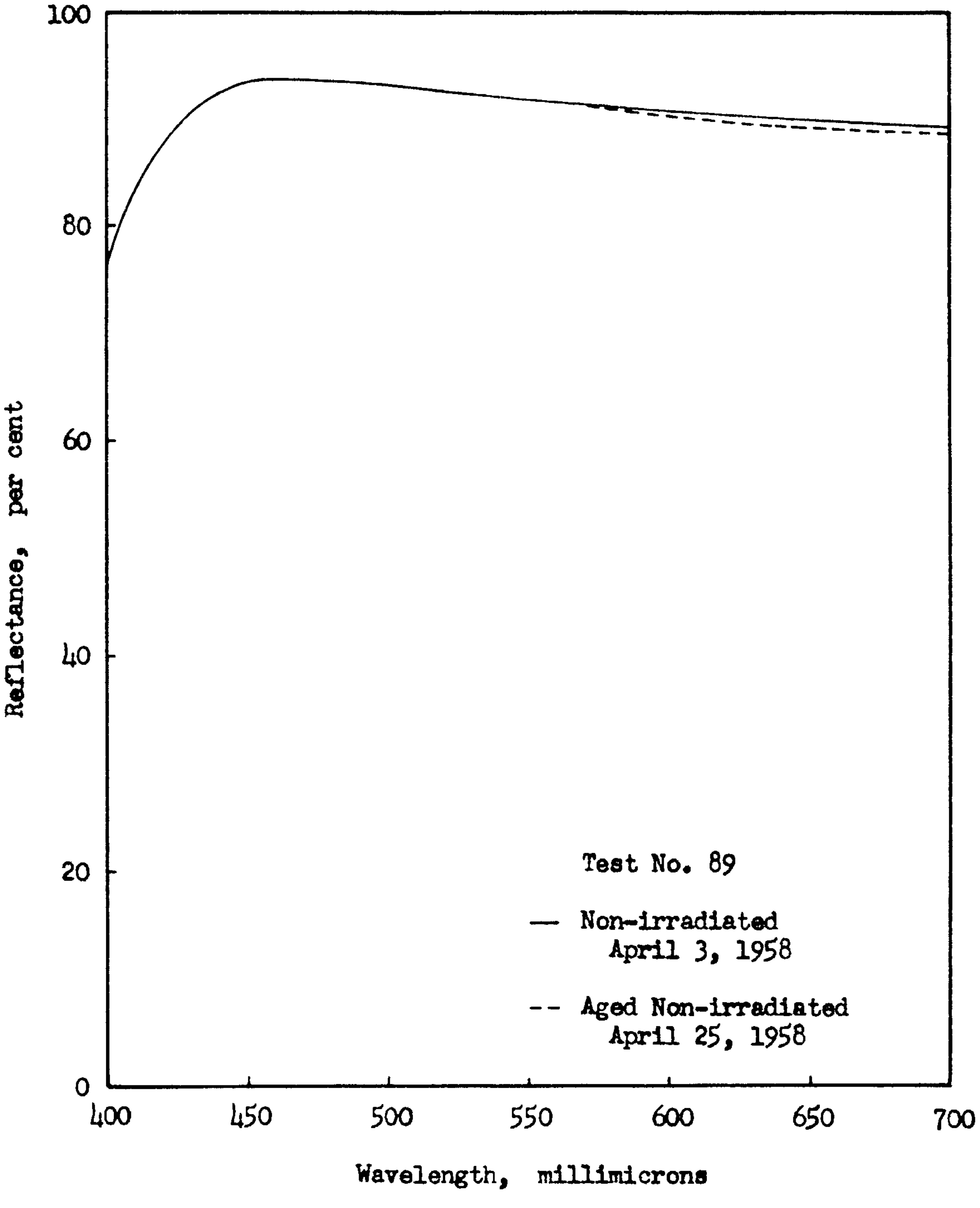

Figure 10. Light Reflectance of Vinyl Chloride Copolymer Enamel Pigmented with Zinc Oxide.

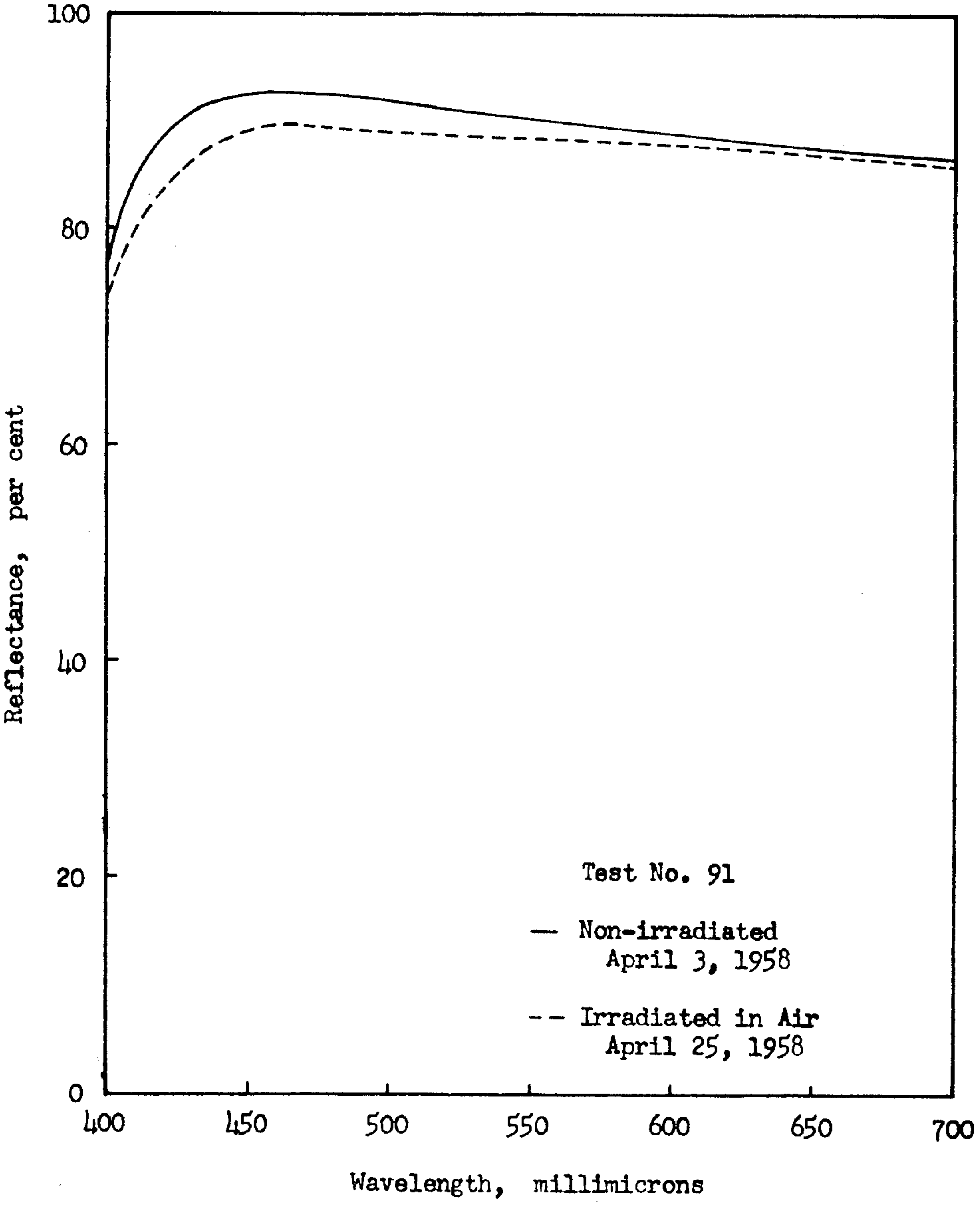

Figure 11. Light Reflectance of Vinyl Chloride Copolymer Enamel Pigmented with Zinc Oxide.

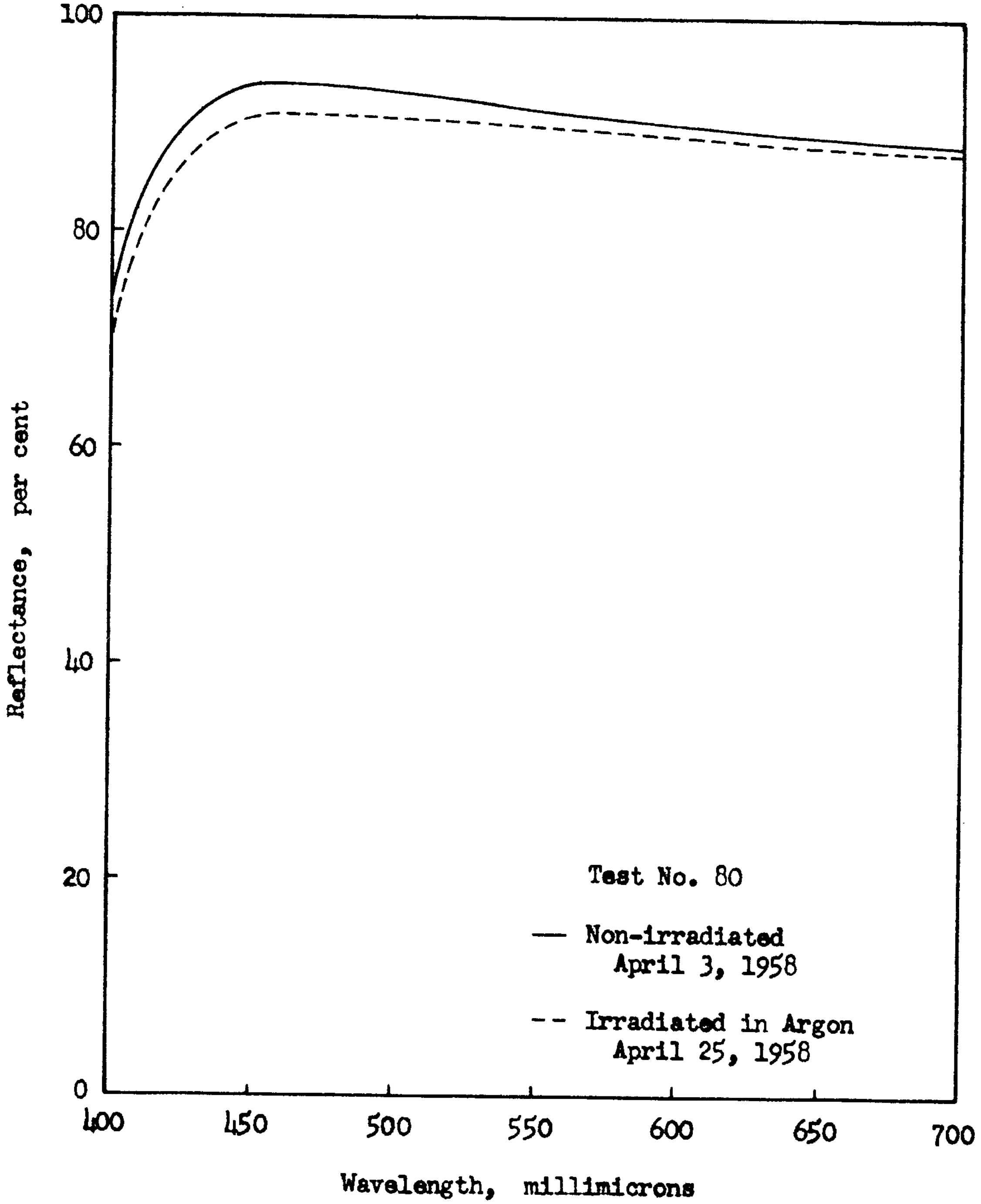

Figure 12. Light Reflectance of Vinyl Chloride Copolymer Enamel Pigmented with Zinc Oxide.

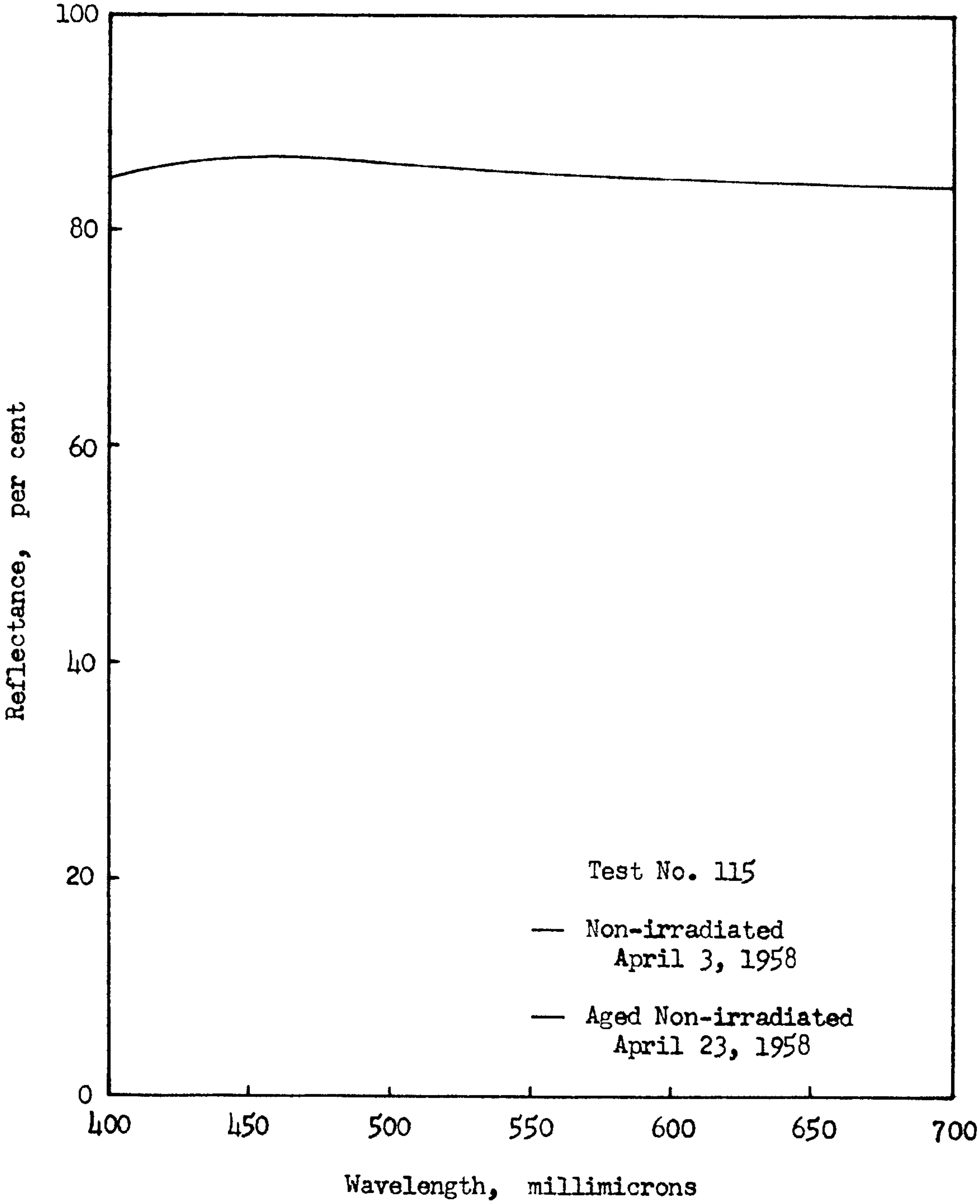

Figure 13. Light Reflectance of Vinyl Chloride Copolymer Enamel Pigmented with Lead Carbonate.

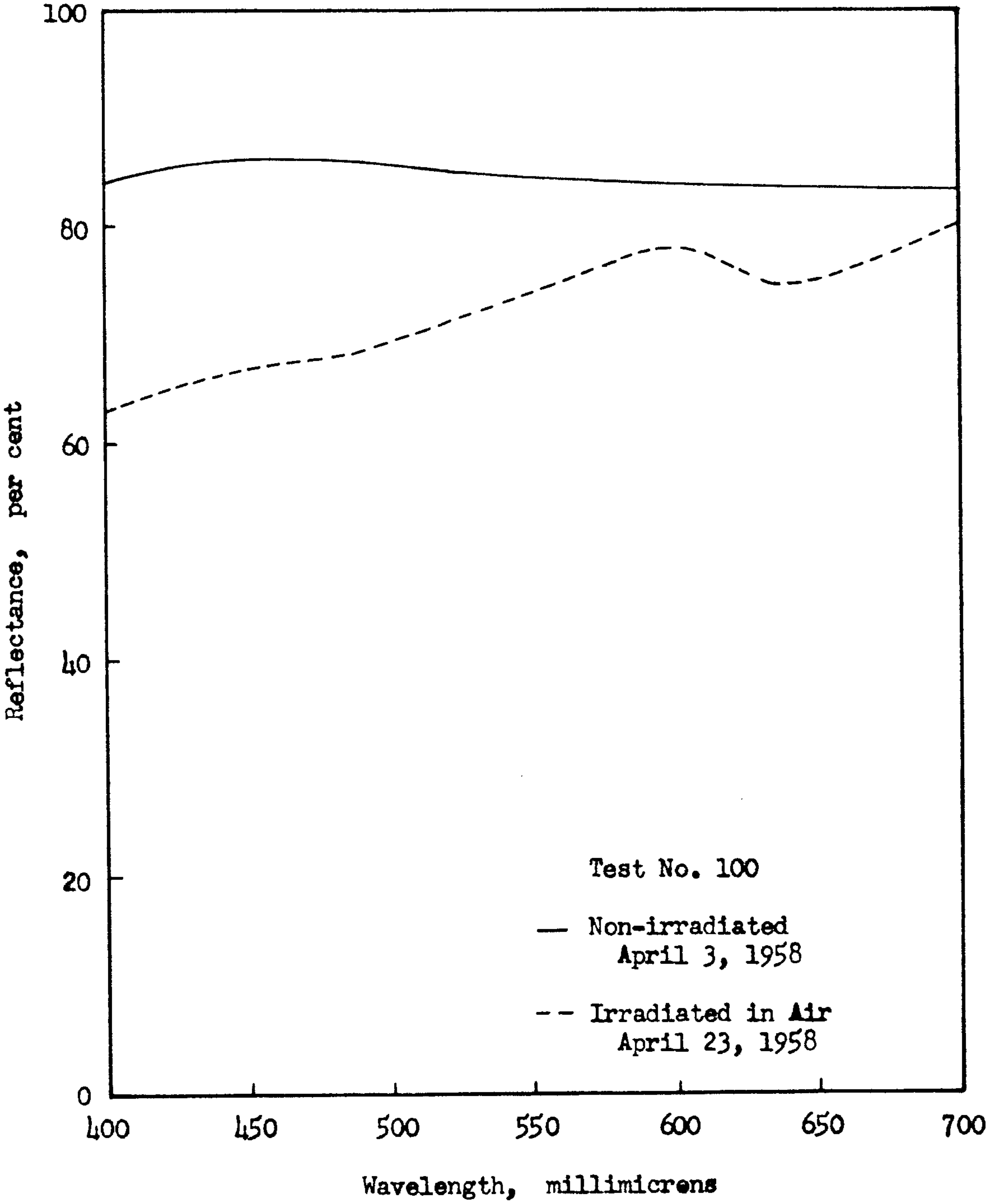

Figure 14. Light Reflectance of Vinyl Chloride Copolymer Enamel Pigmented with Lead Carbonate.

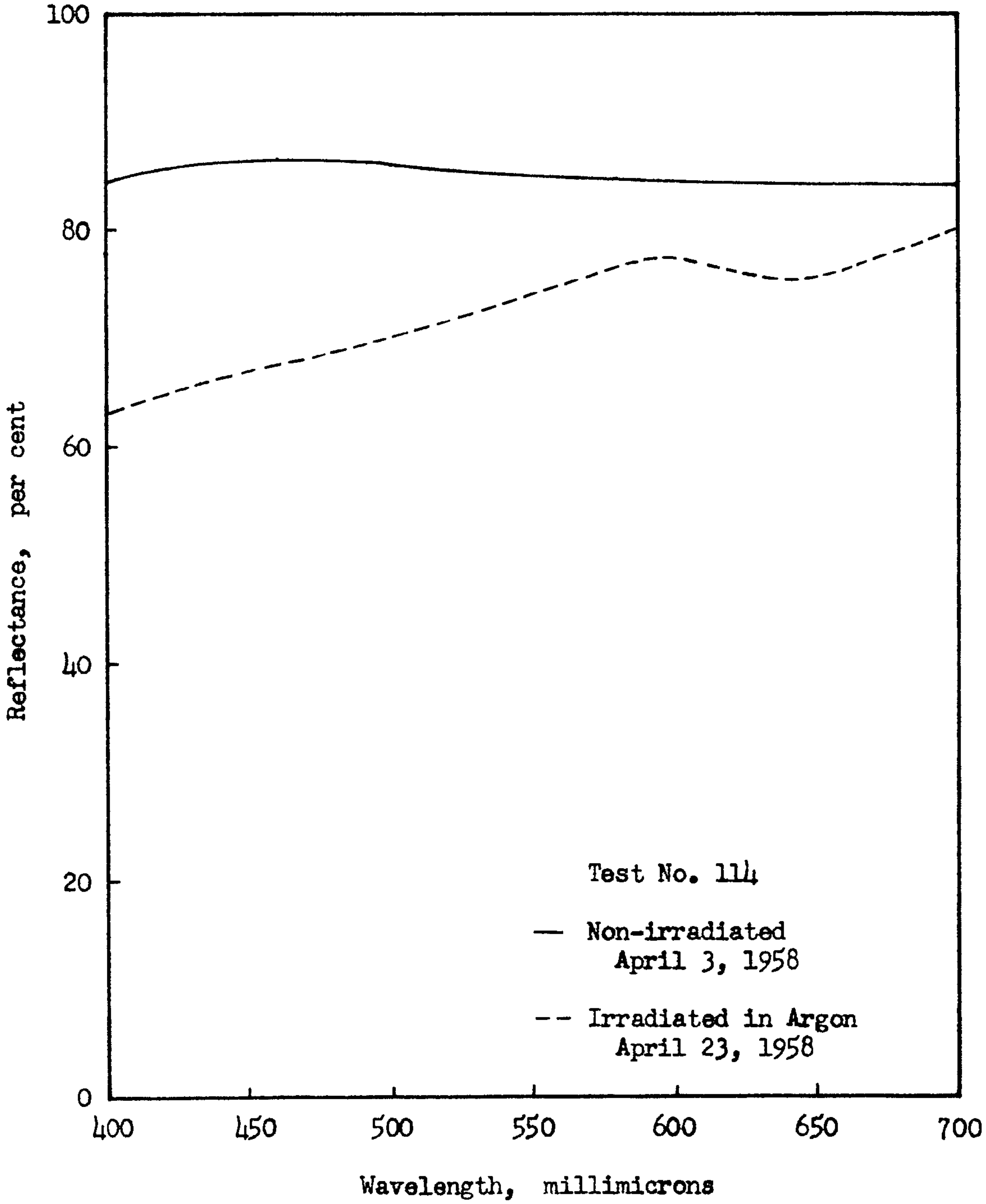

Figure 15. Light Reflectance of Vinyl Chloride Copolymer Enamel Pigmented with Lead Carbonate.

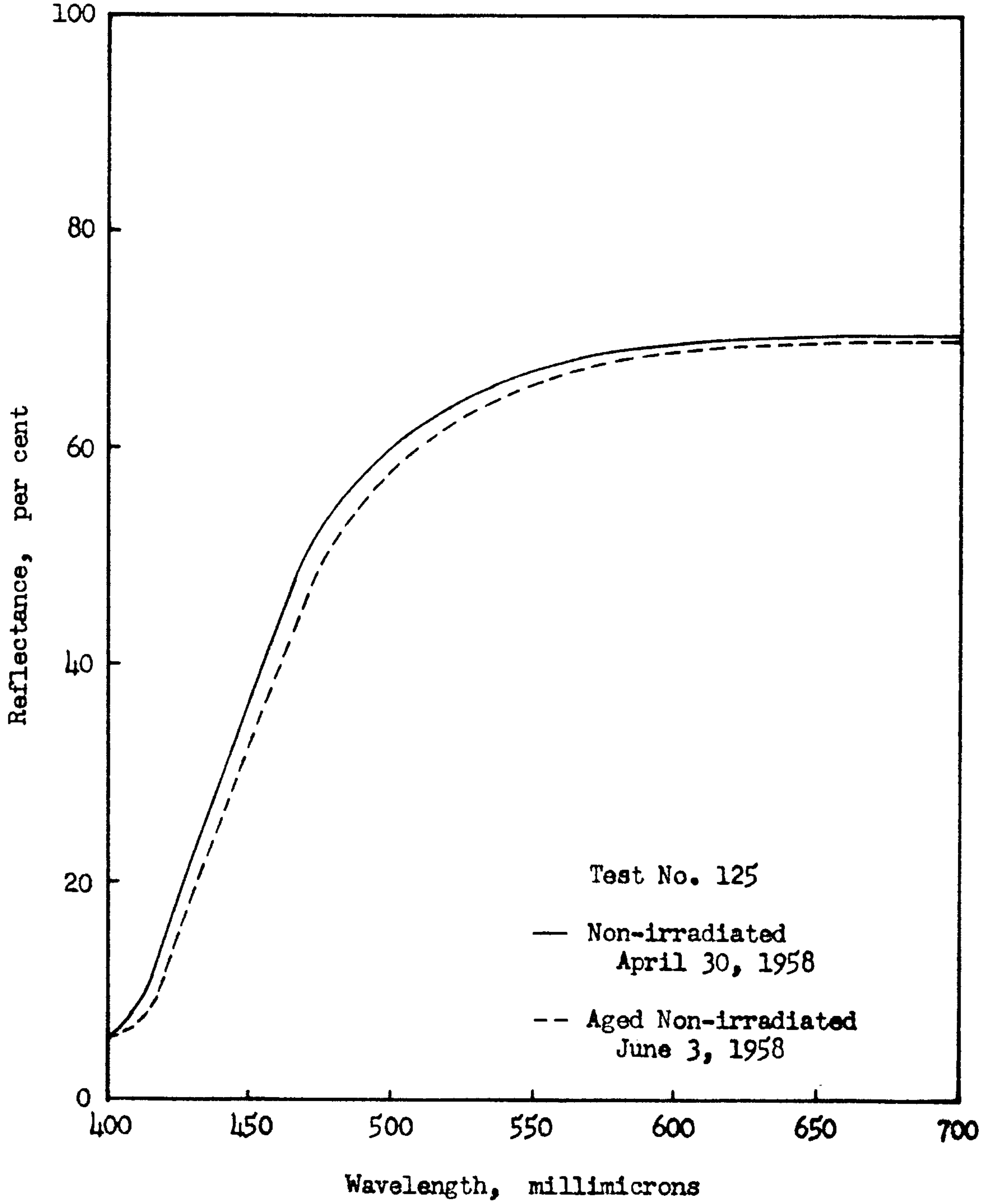

Figure 16. Light Reflectance of Clear Spar Varnish.

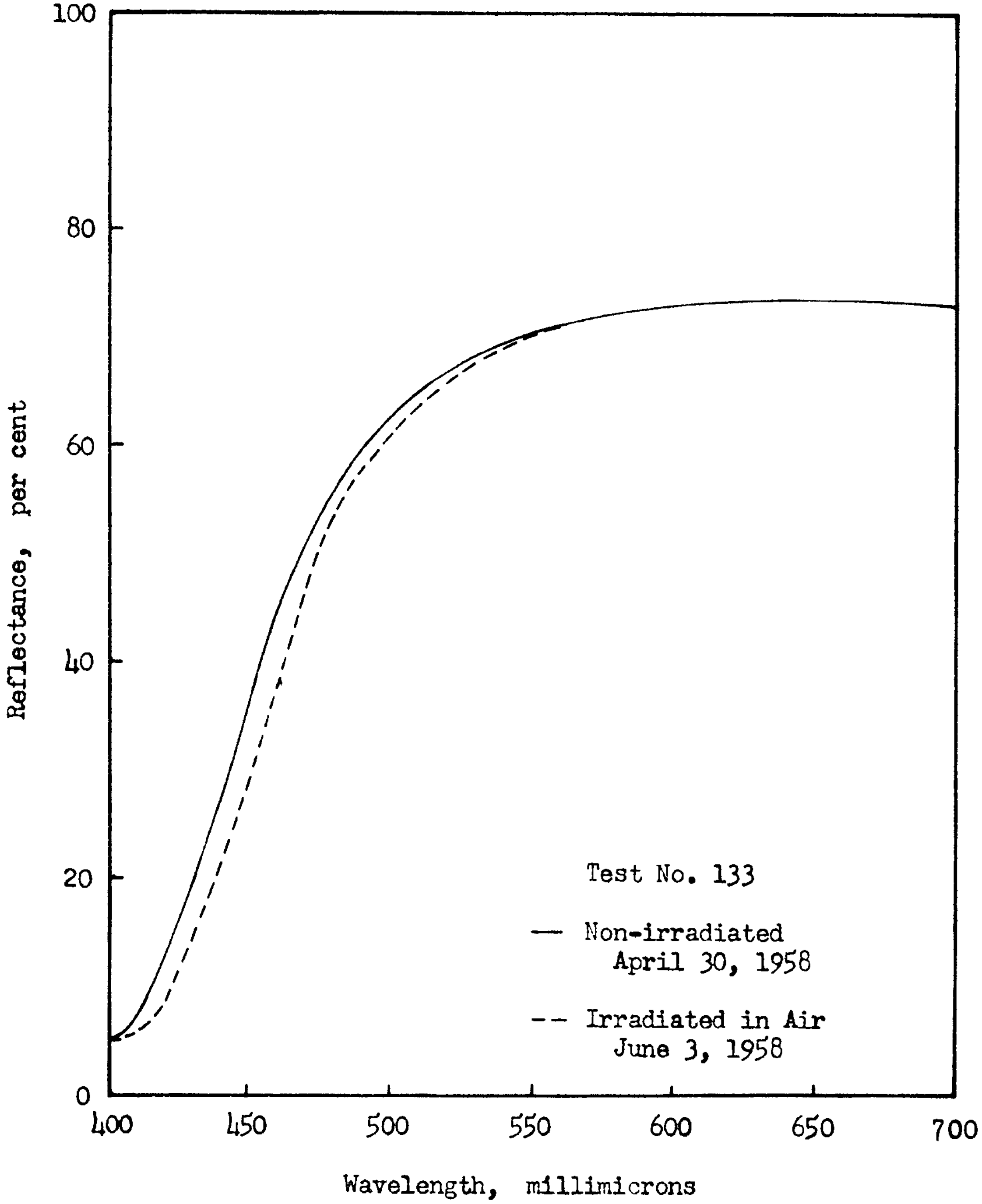

Figure 17. Light Reflectance of Clear Spar Varnish.

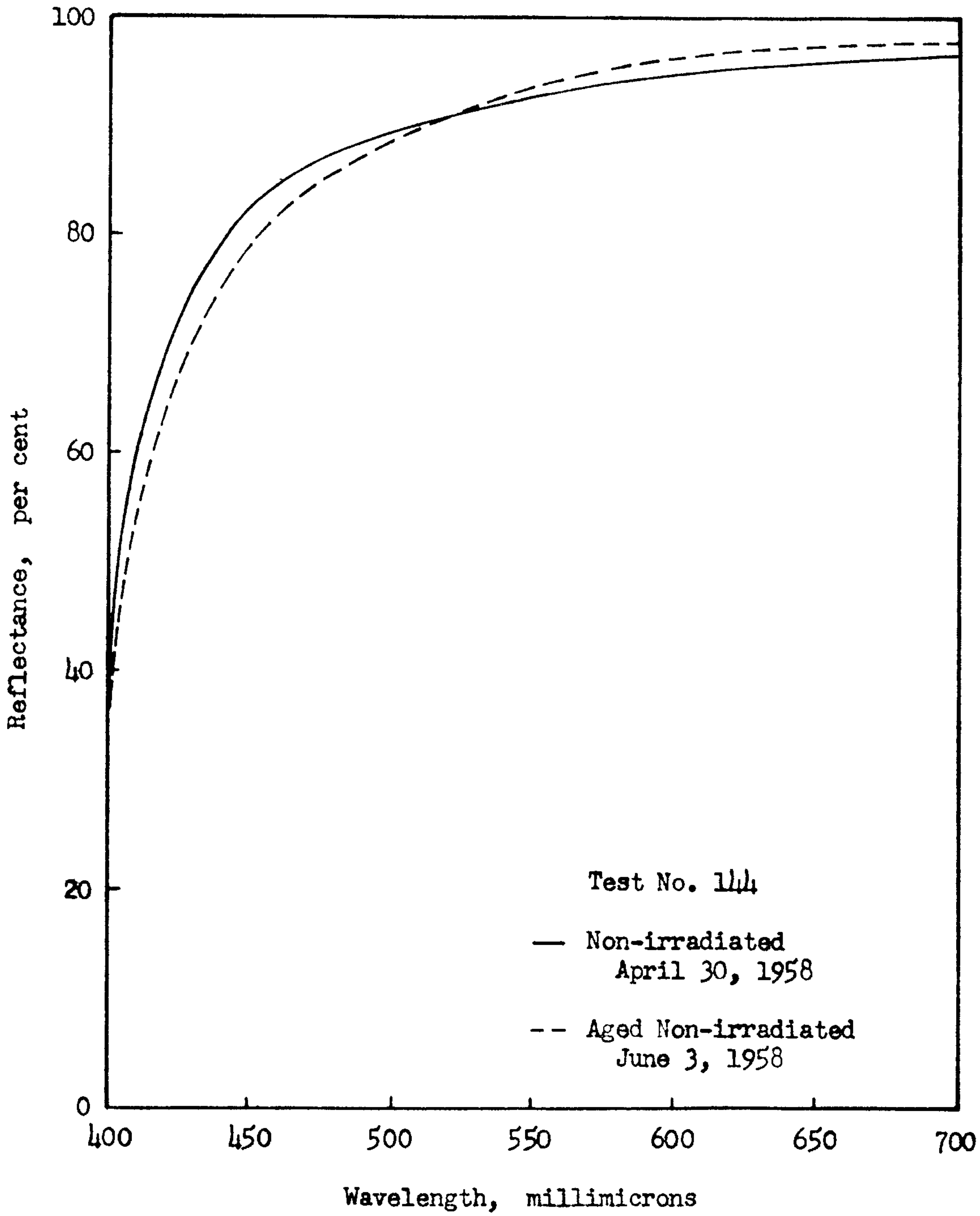

Figure 18. Light Reflectance of Spar Enamel Pigmented with Titanium Dioxide.

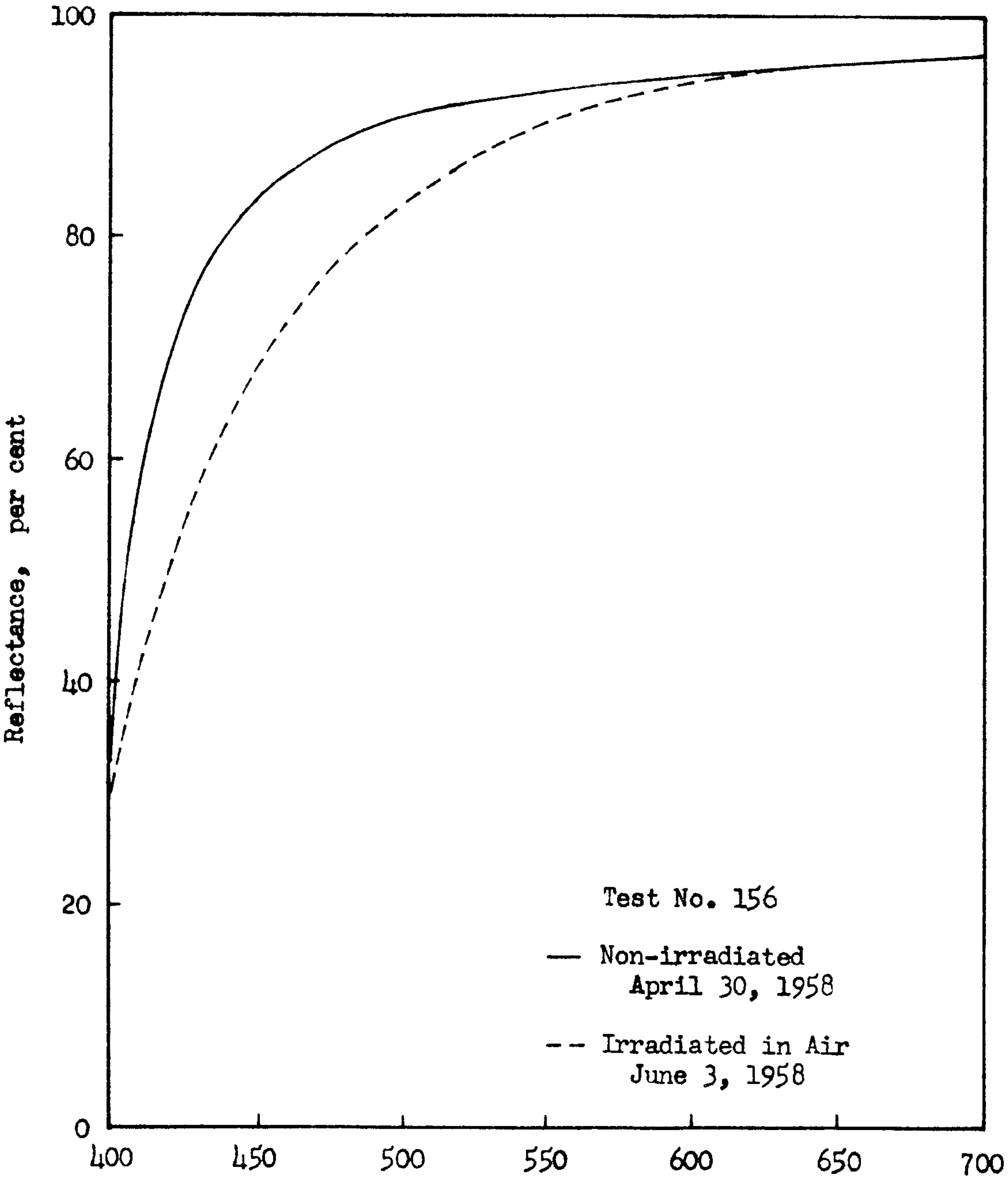

Figure 19. Light Reflectance of Spar Enamel Pigmented with Titanium Dioxide.

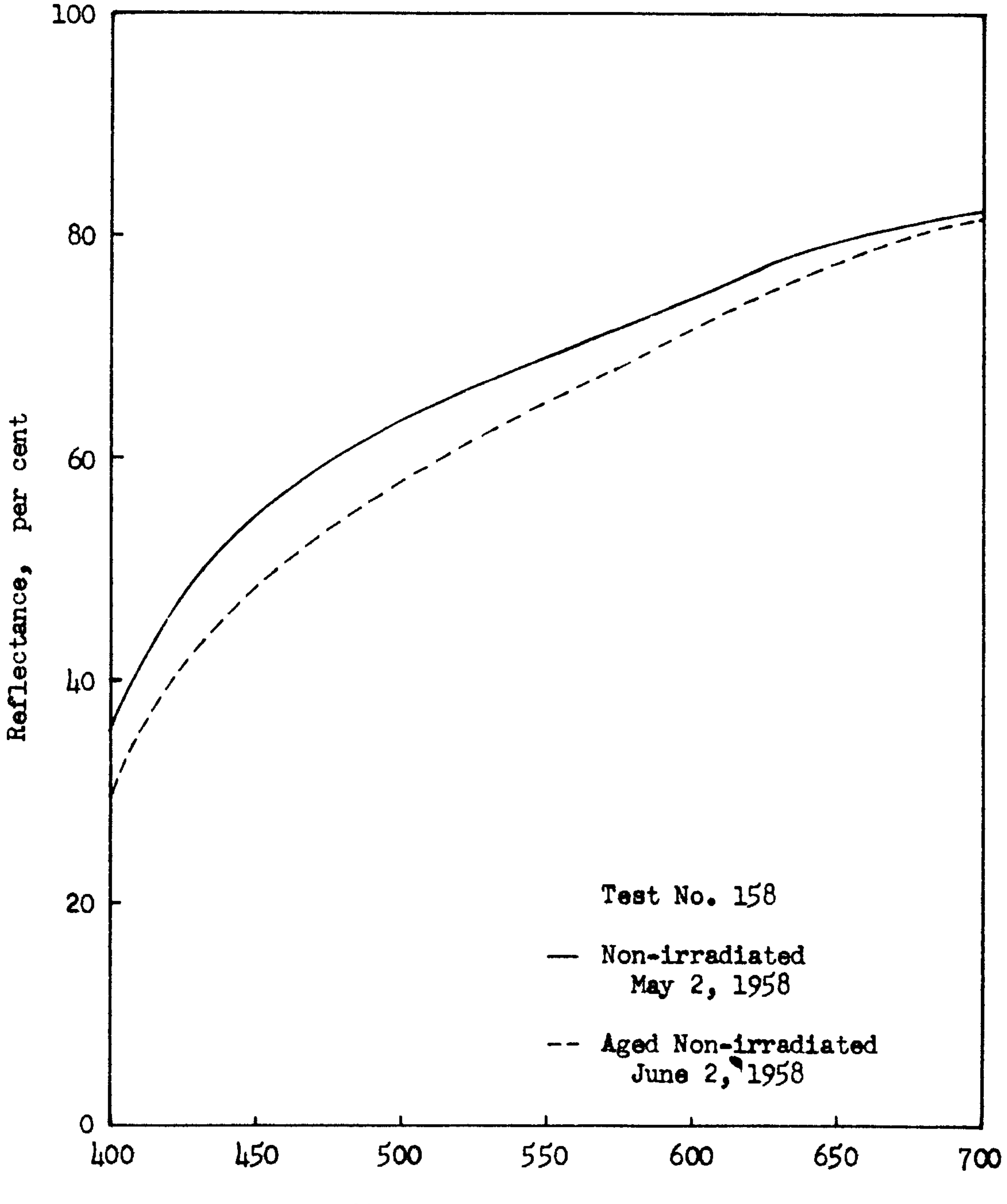

Figure 20. Light Reflectance of Spar Enamel Pigmented with Zinc Oxide.

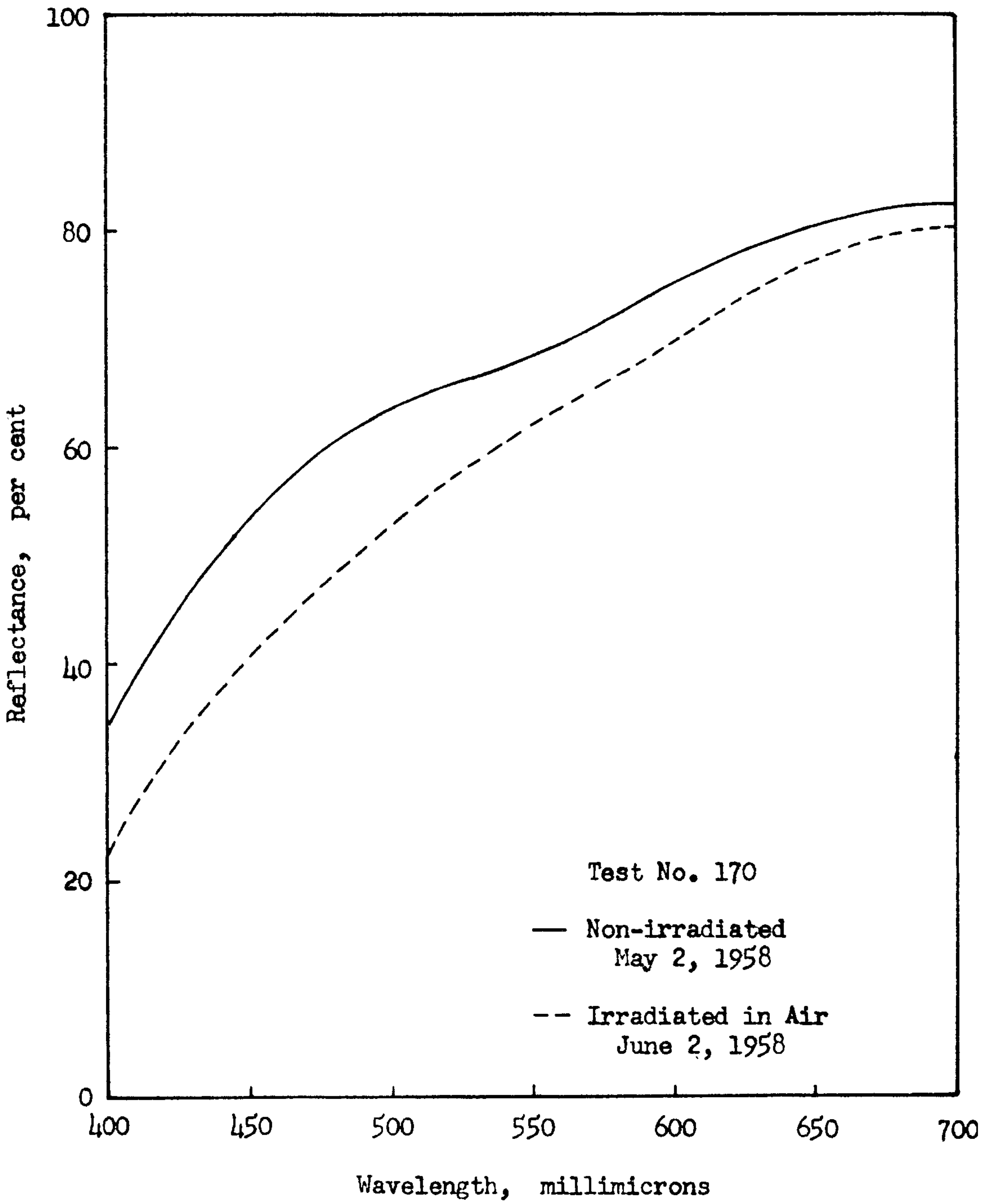

Figure 21. Light Reflectance of Spar Enamel Pigmented with Zinc Oxide.

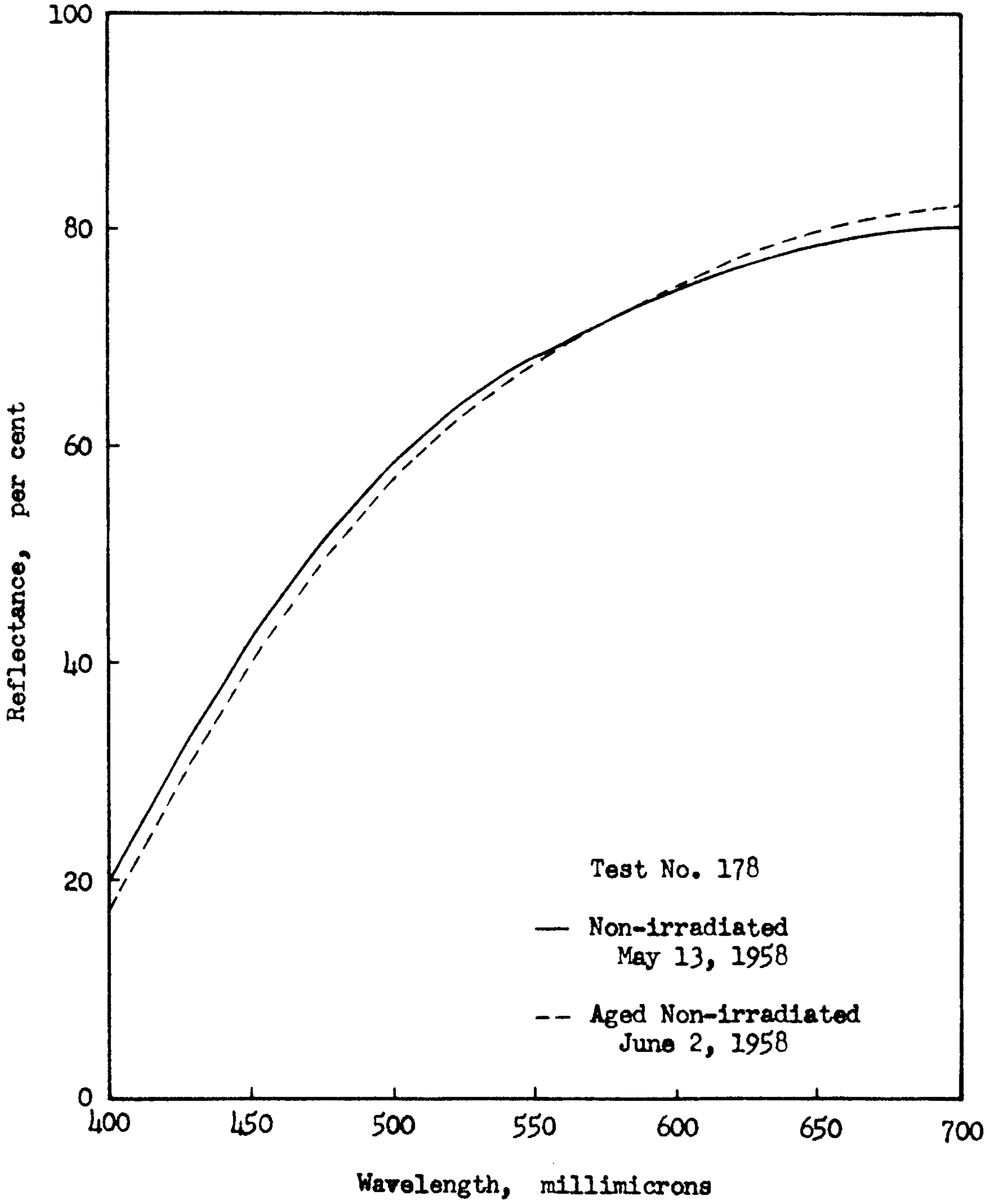

Figure 22. Light Reflectance of Spar Enamel Pigmented with Lead Carbonate.

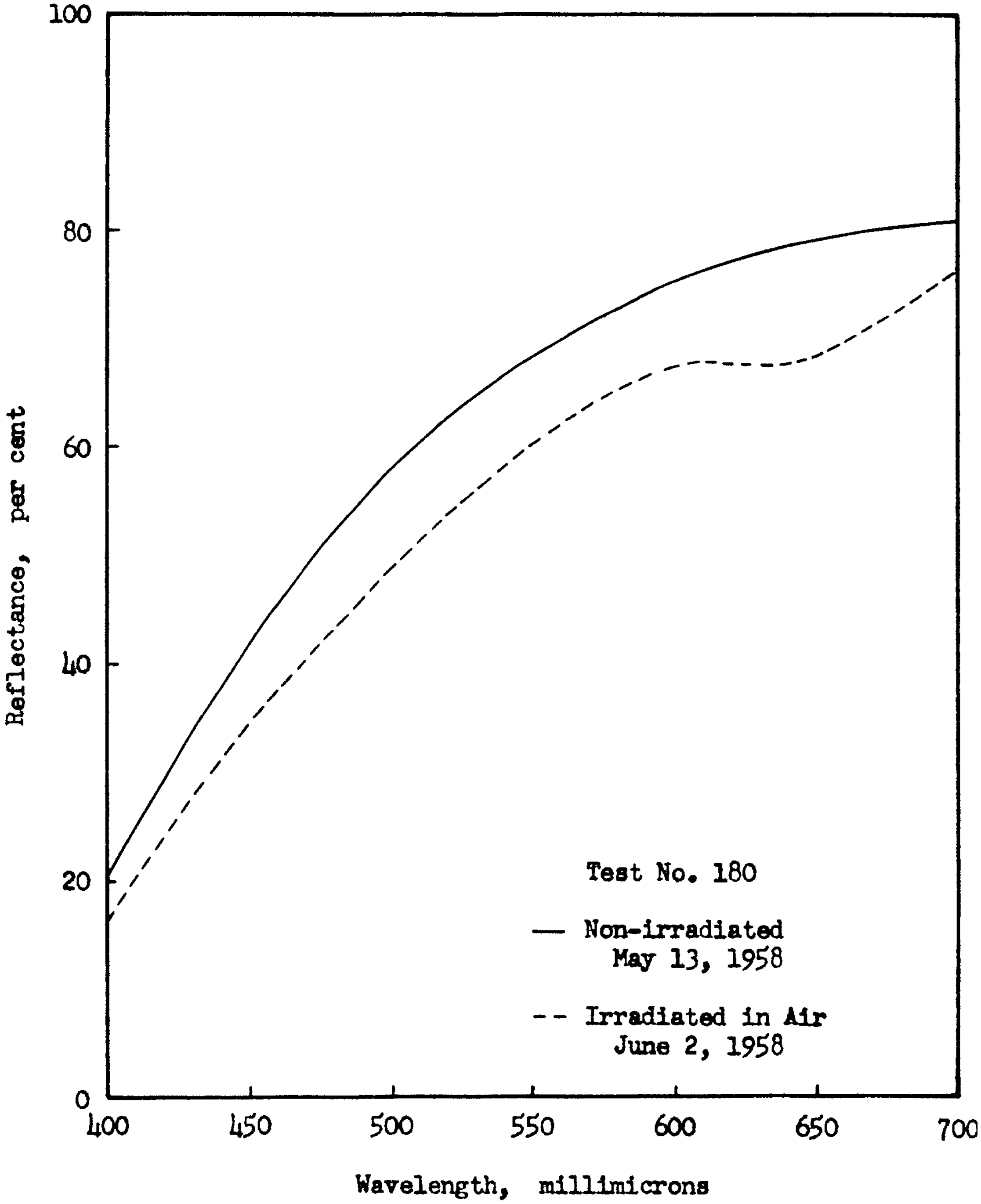

Figure 23. Light Reflectance of Spar Enamel Pigmented with Lead Carbonate.

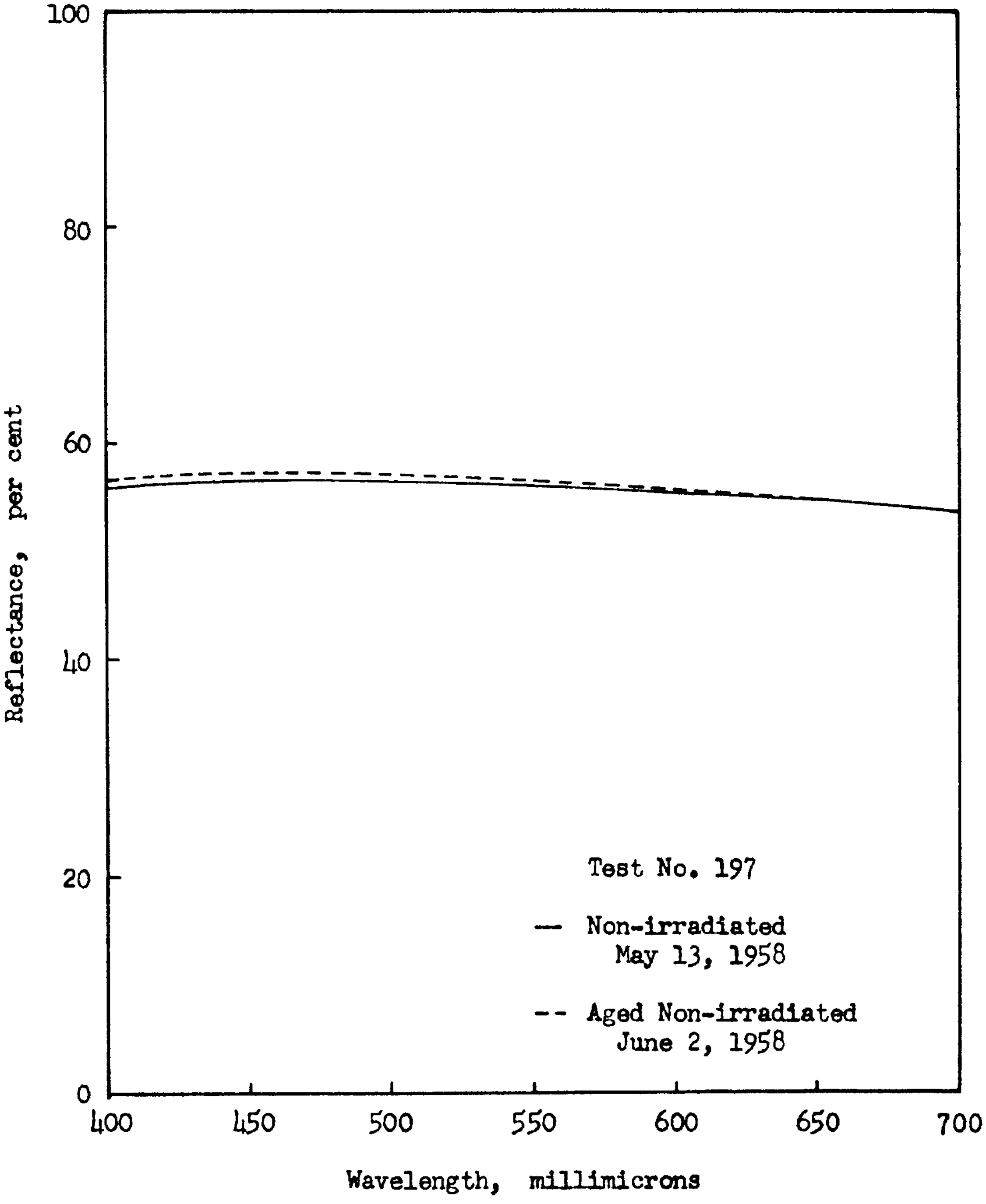

Figure 24. Light Reflectance of Spar Enamel Pigmented with Aluminum Paste.

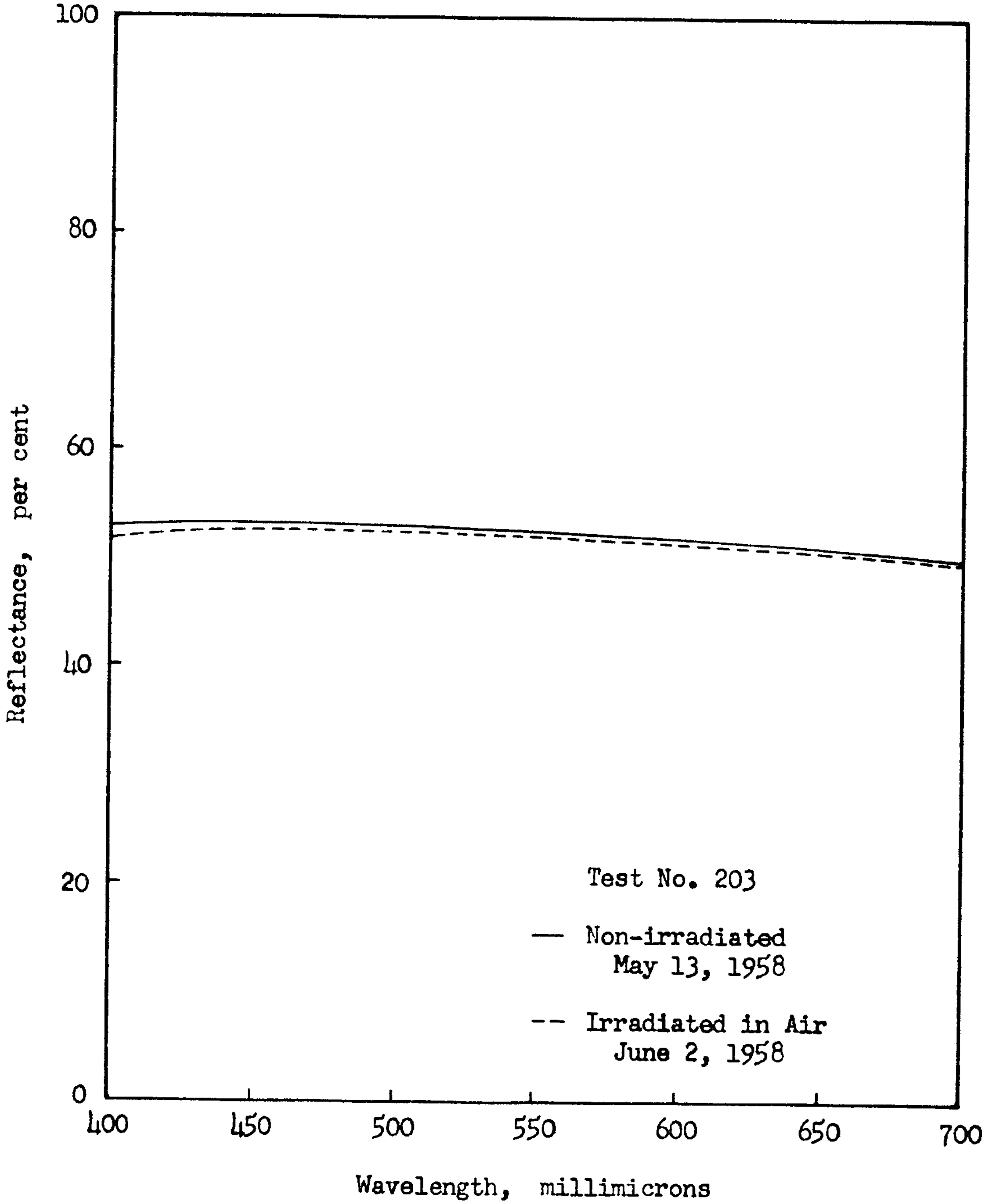

Figure 25. Light Reflectance of Spar Enamel Pigmented with Aluminum Paste.

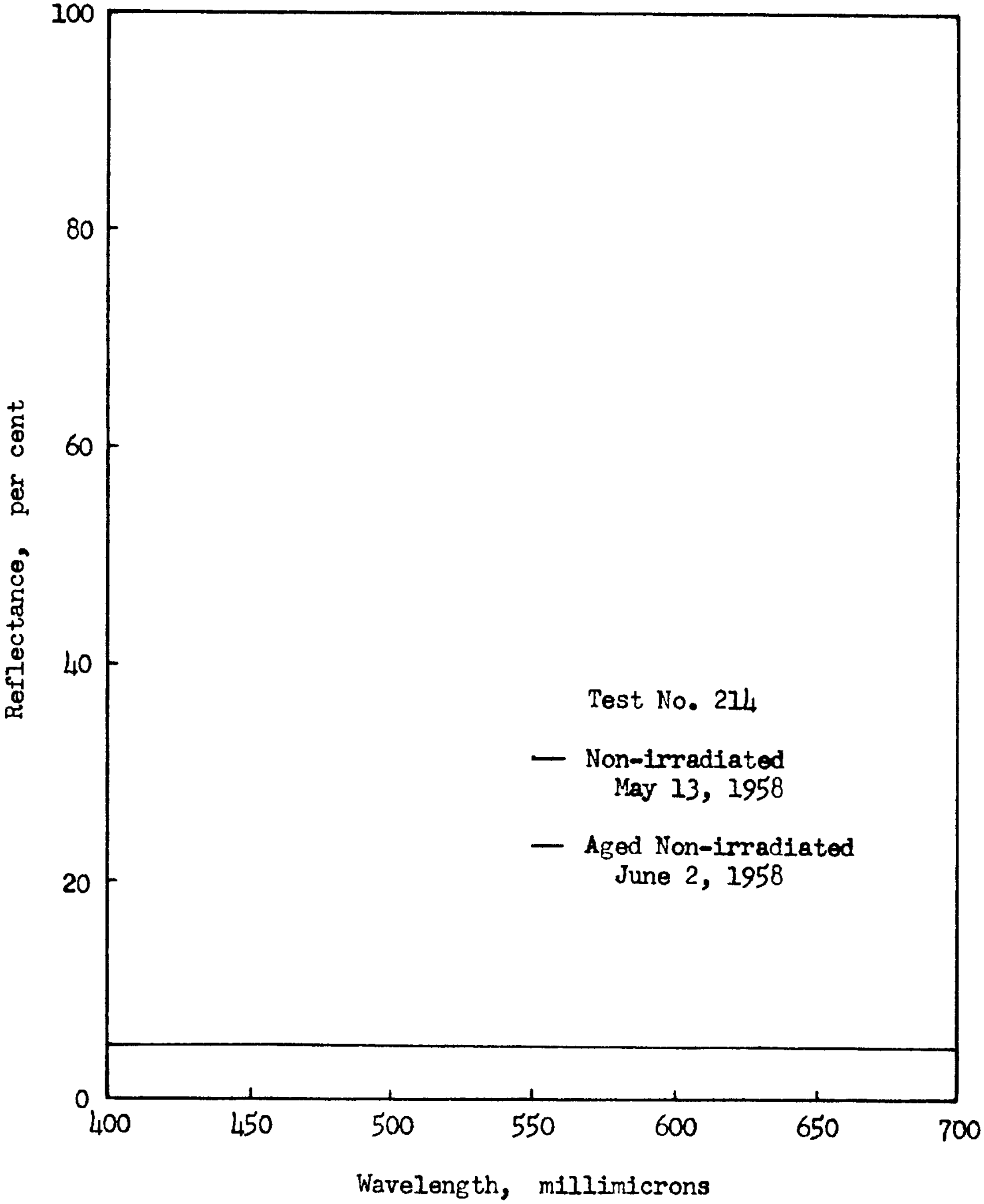

Figure 26. Light Reflectance of Spar Enamel Pigmented with Lampblack.

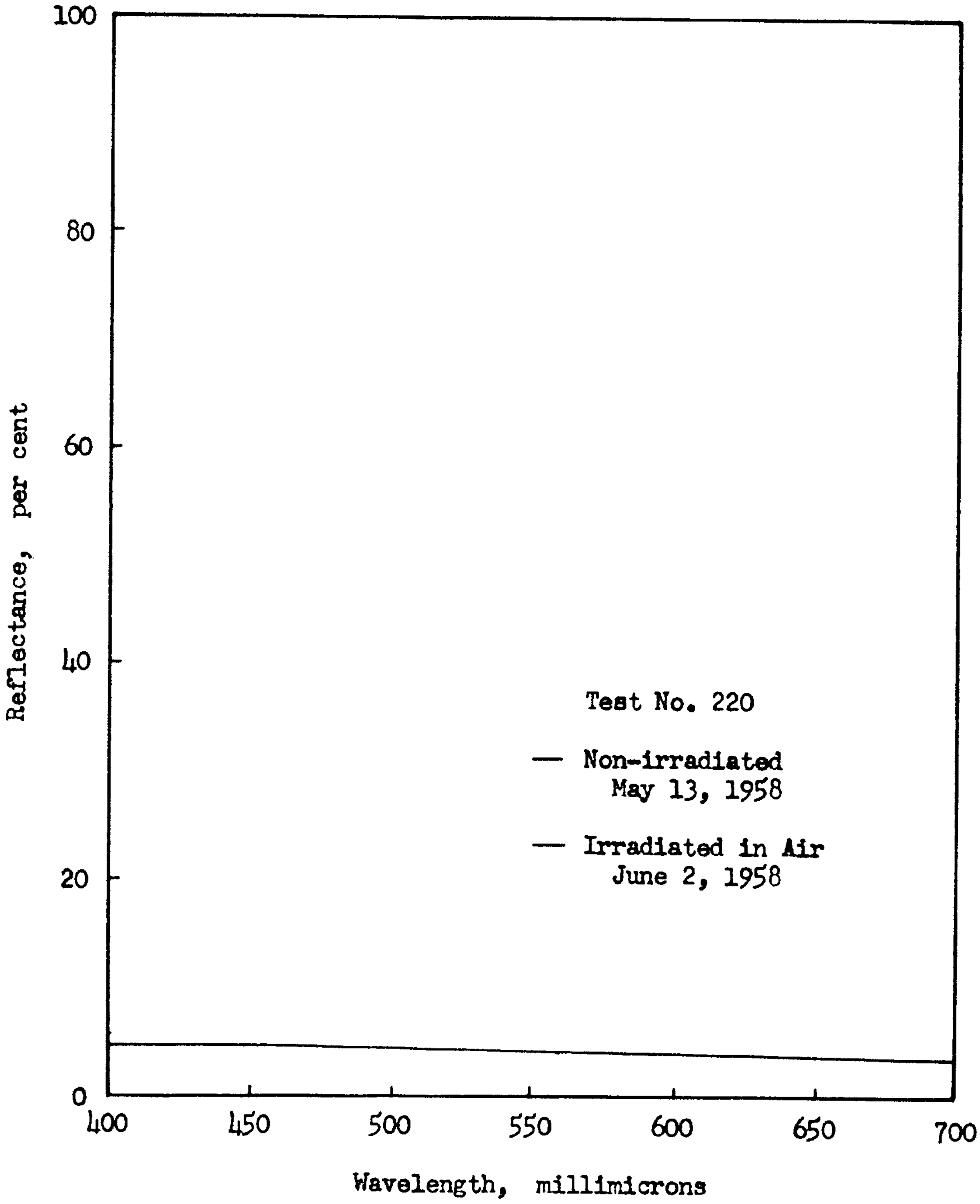

Figure 27. Light Reflectance of Spar Enamel Pigmented with Lampblack.

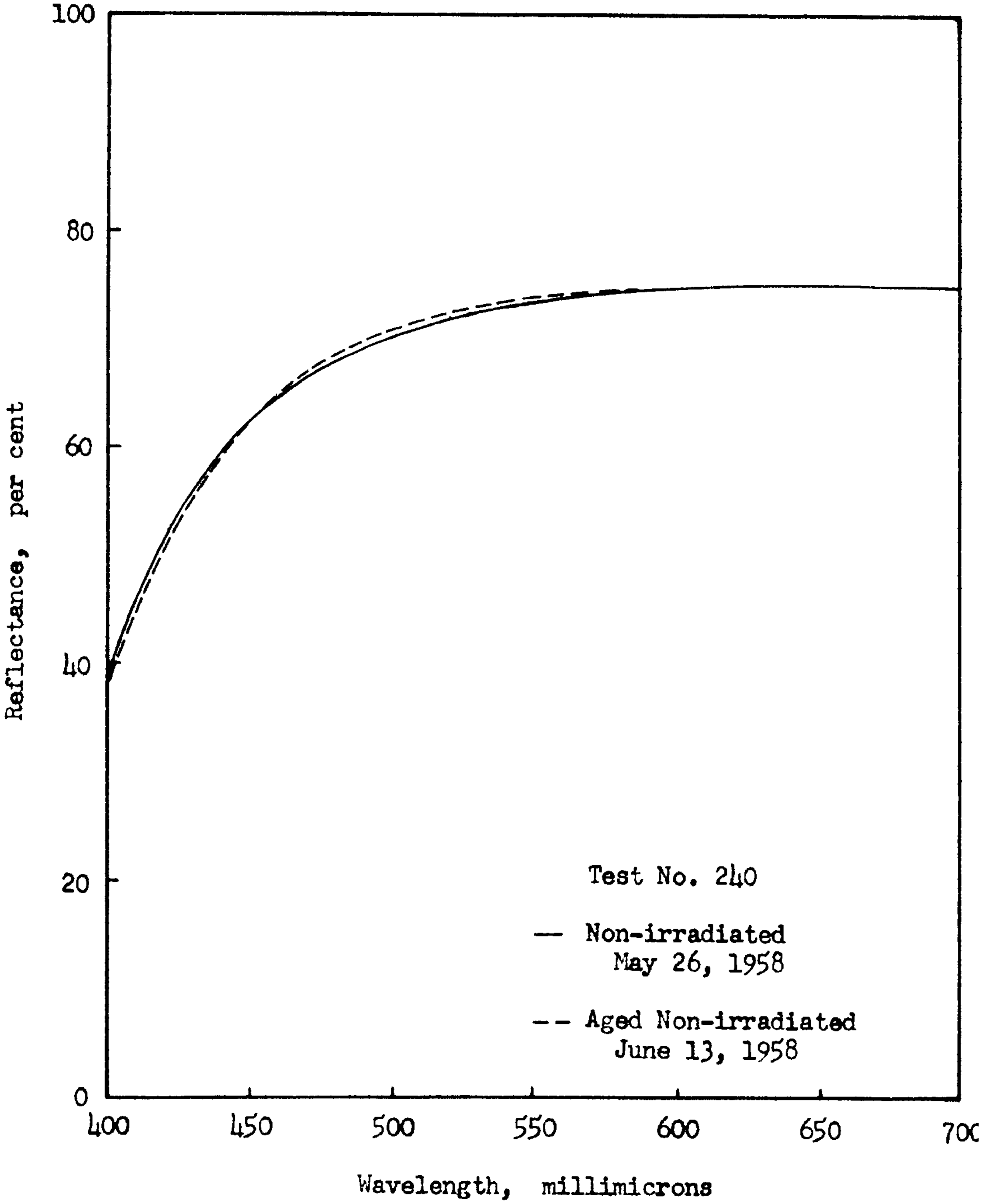

Figure 28. Light Reflectance of Clear Alkyd Varnish.

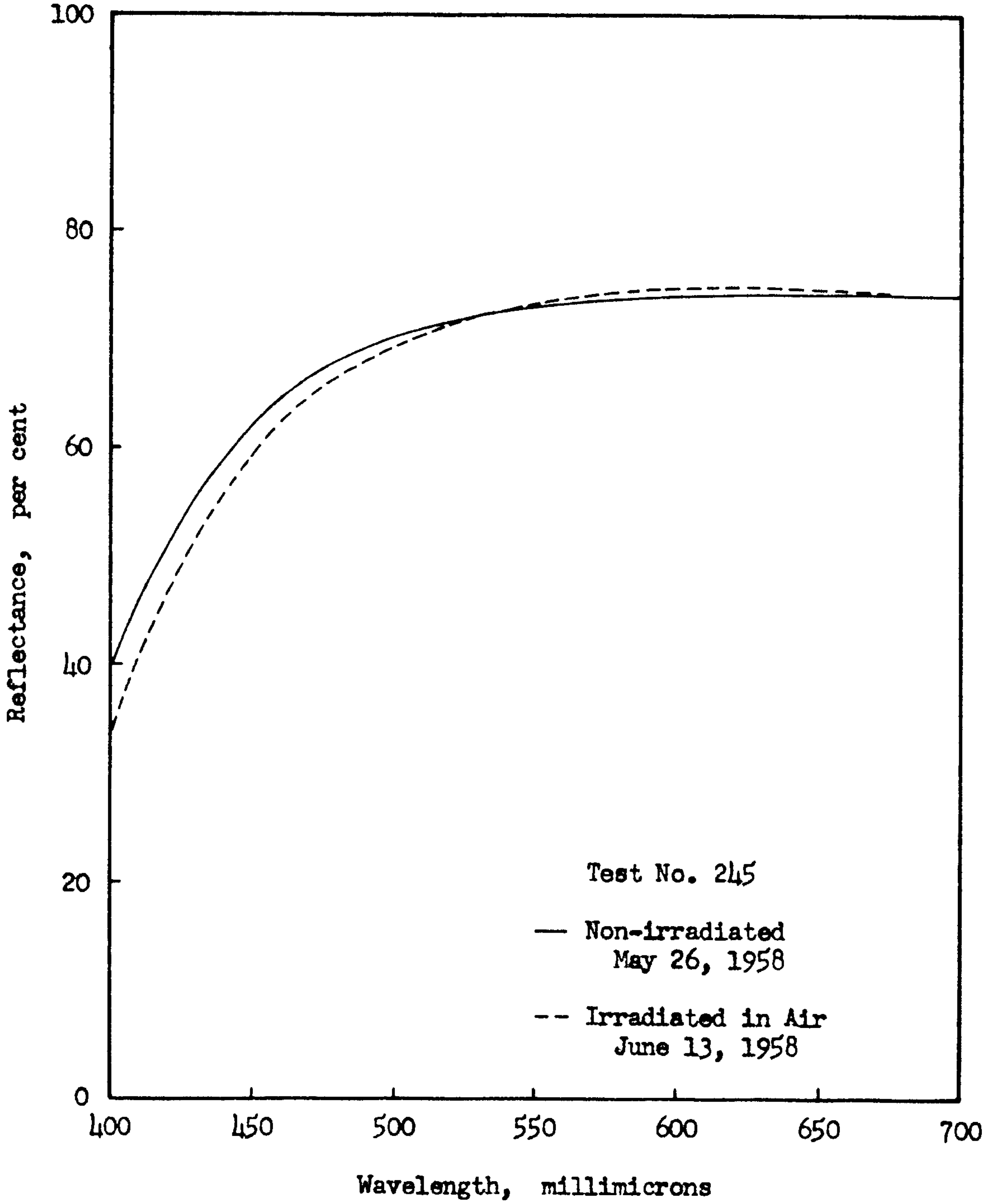

Figure 29. Light Reflectance of Clear Alkyd Varnish.

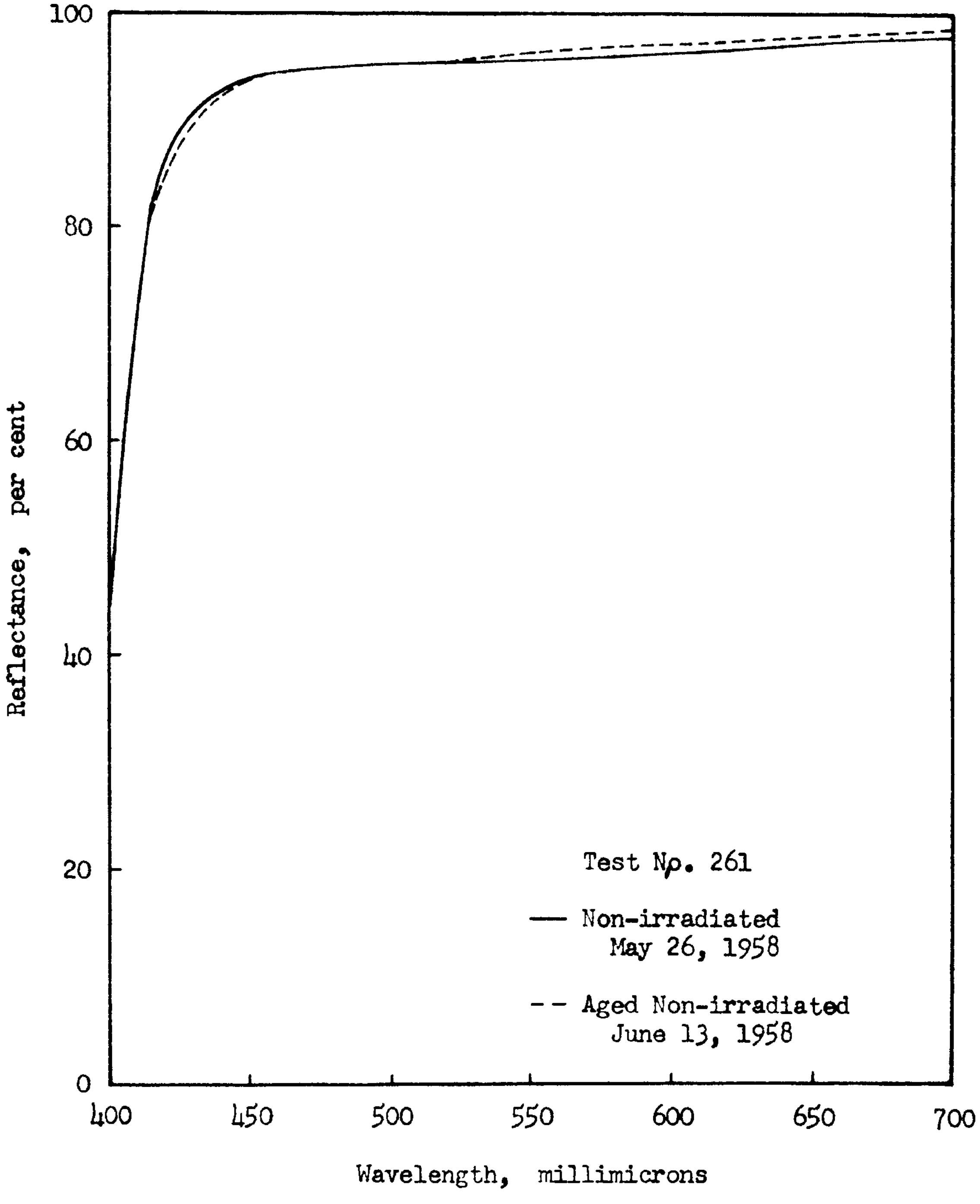

Figure 30. Light Reflectance of Alkyd Enamel Pigmented with Titanium Dioxide.

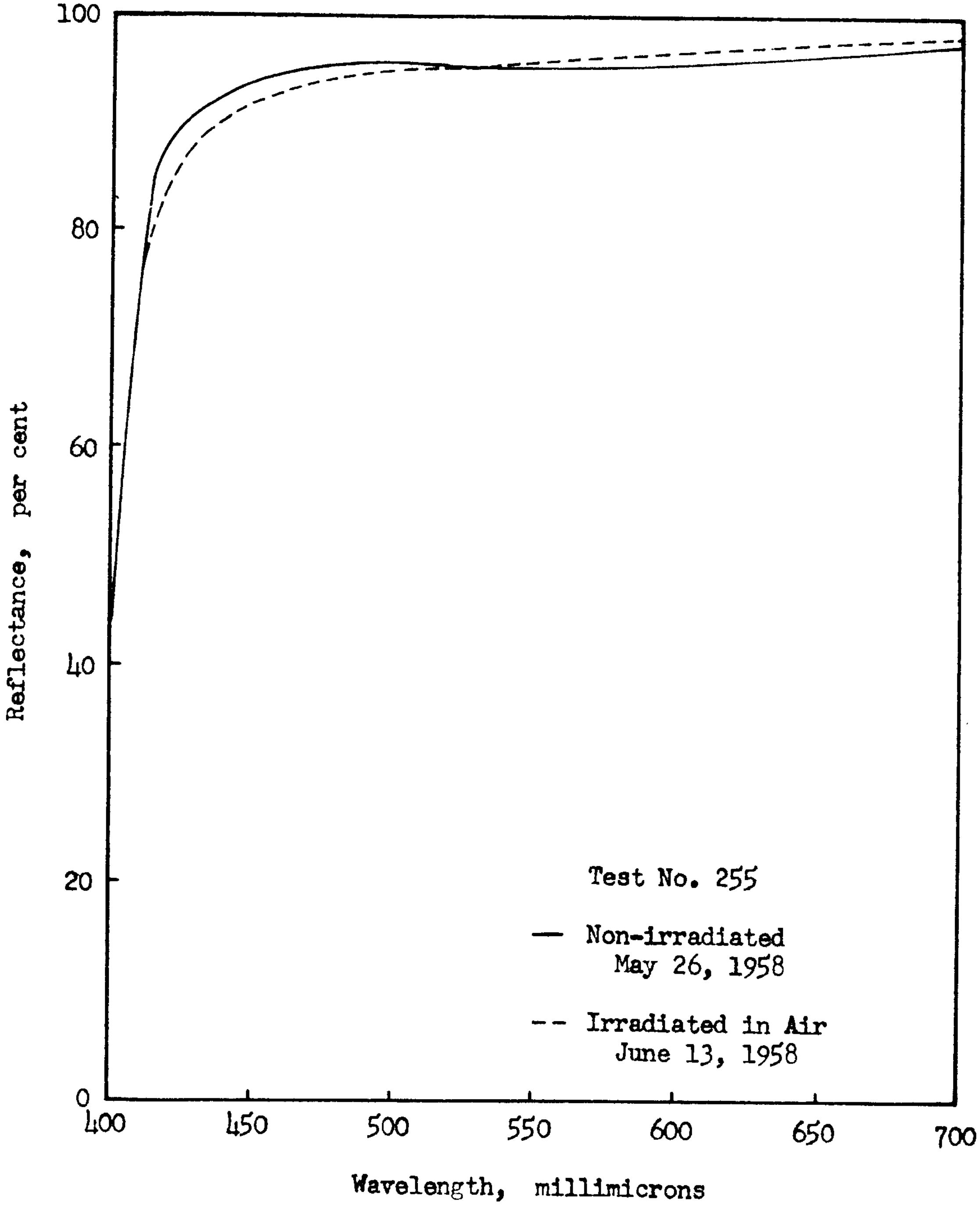

Figure 31. Light Reflectance of Alkyd Enamel Pigmented with Titanium Dioxide.

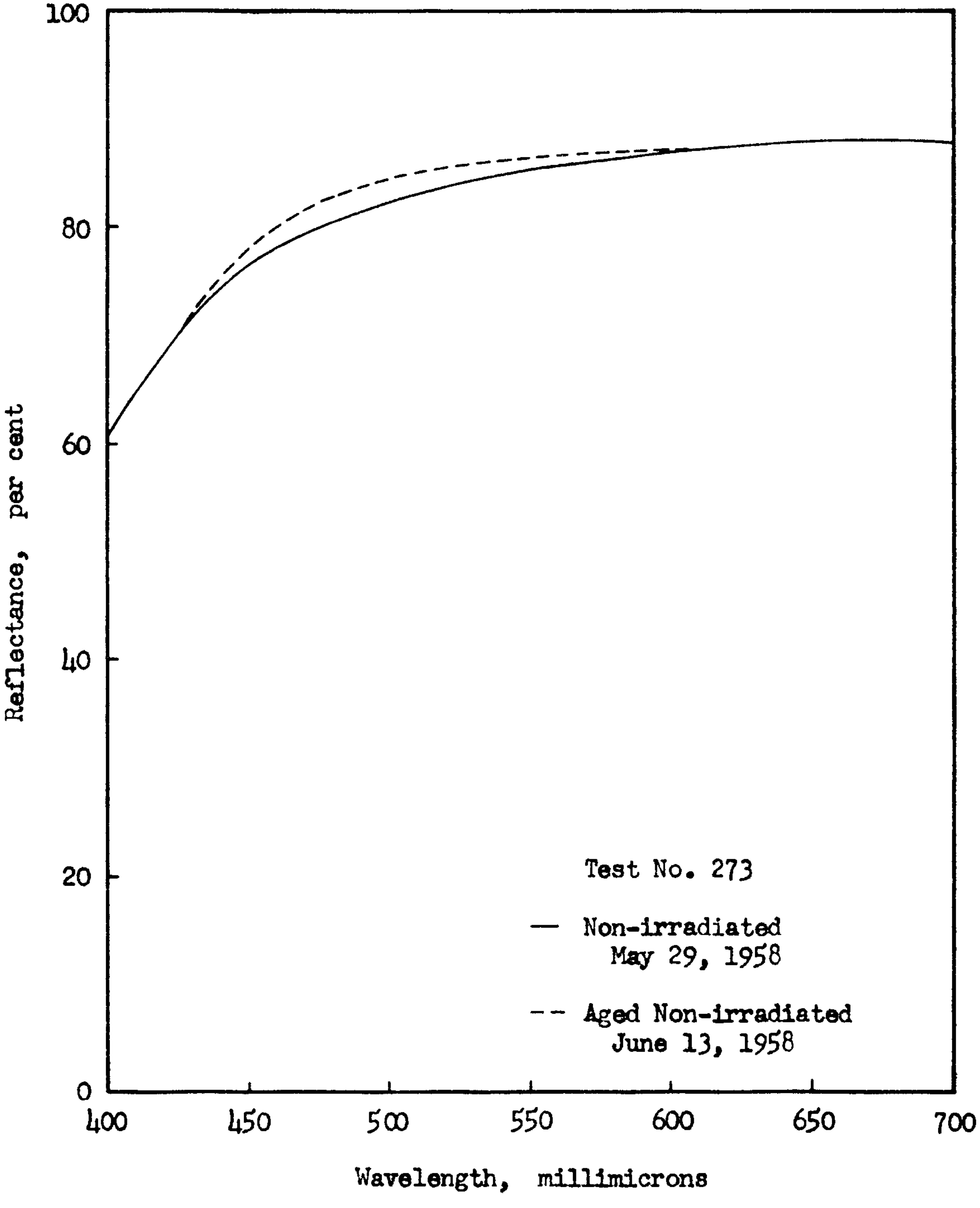

Figure 32. Light Reflectance of Alkyd Enamel Pigmented with Zinc Oxide.

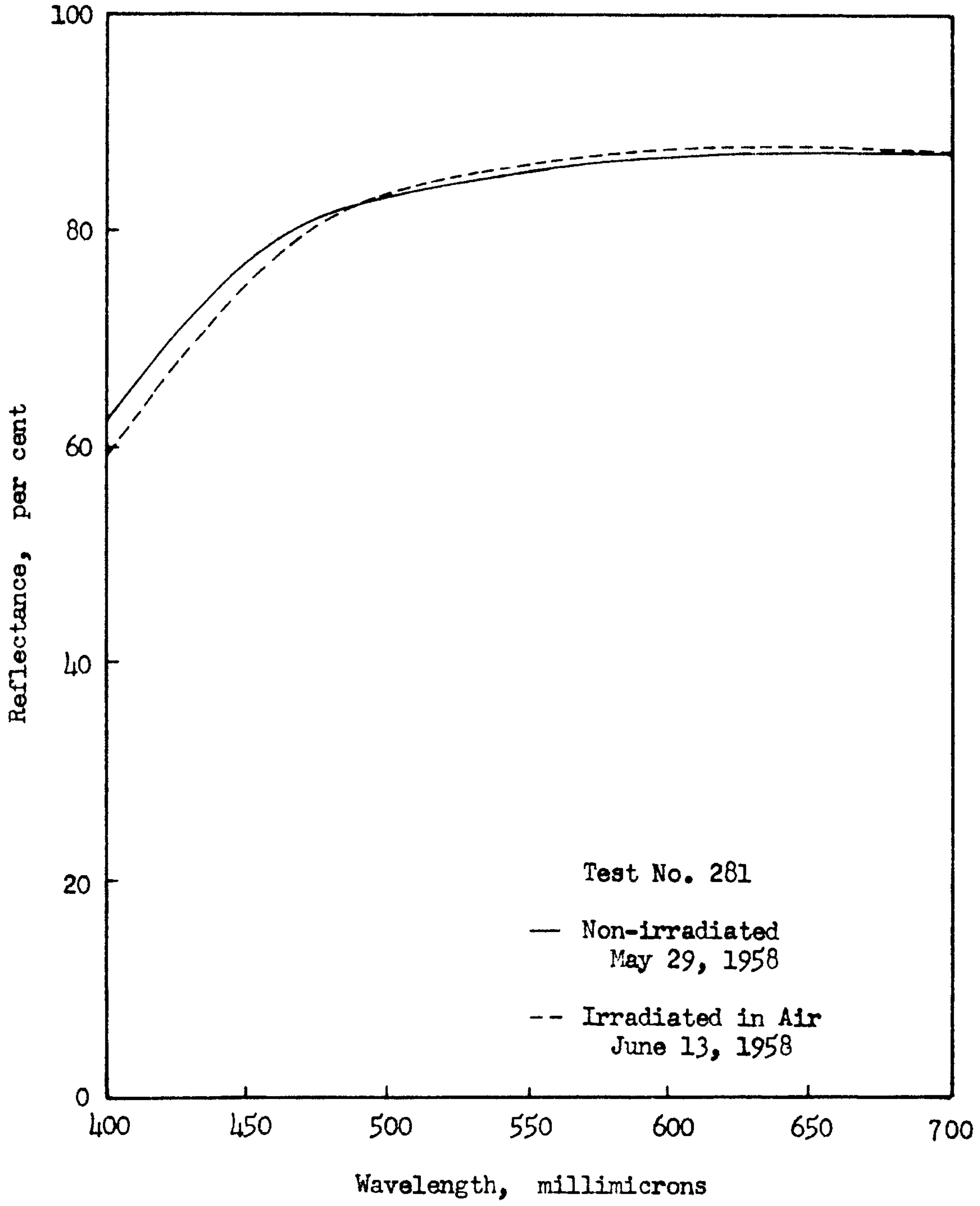

Figure 33. Light Reflectance of Alkyd Enamel Pigmented with Zinc Oxide.

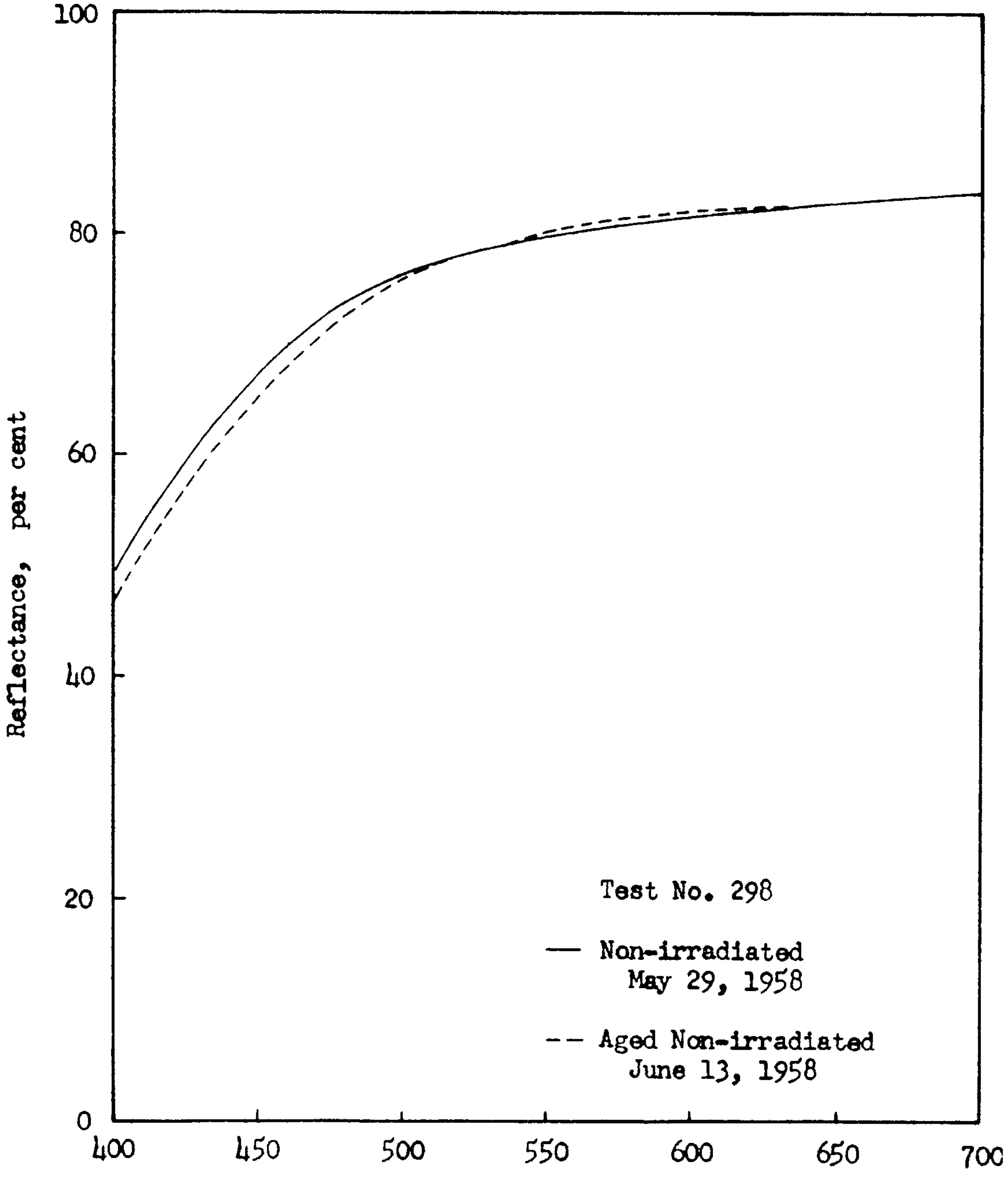

Figure 34. Light Reflectance of Alkyd Enamel Pigmented with Lead Carbonate.

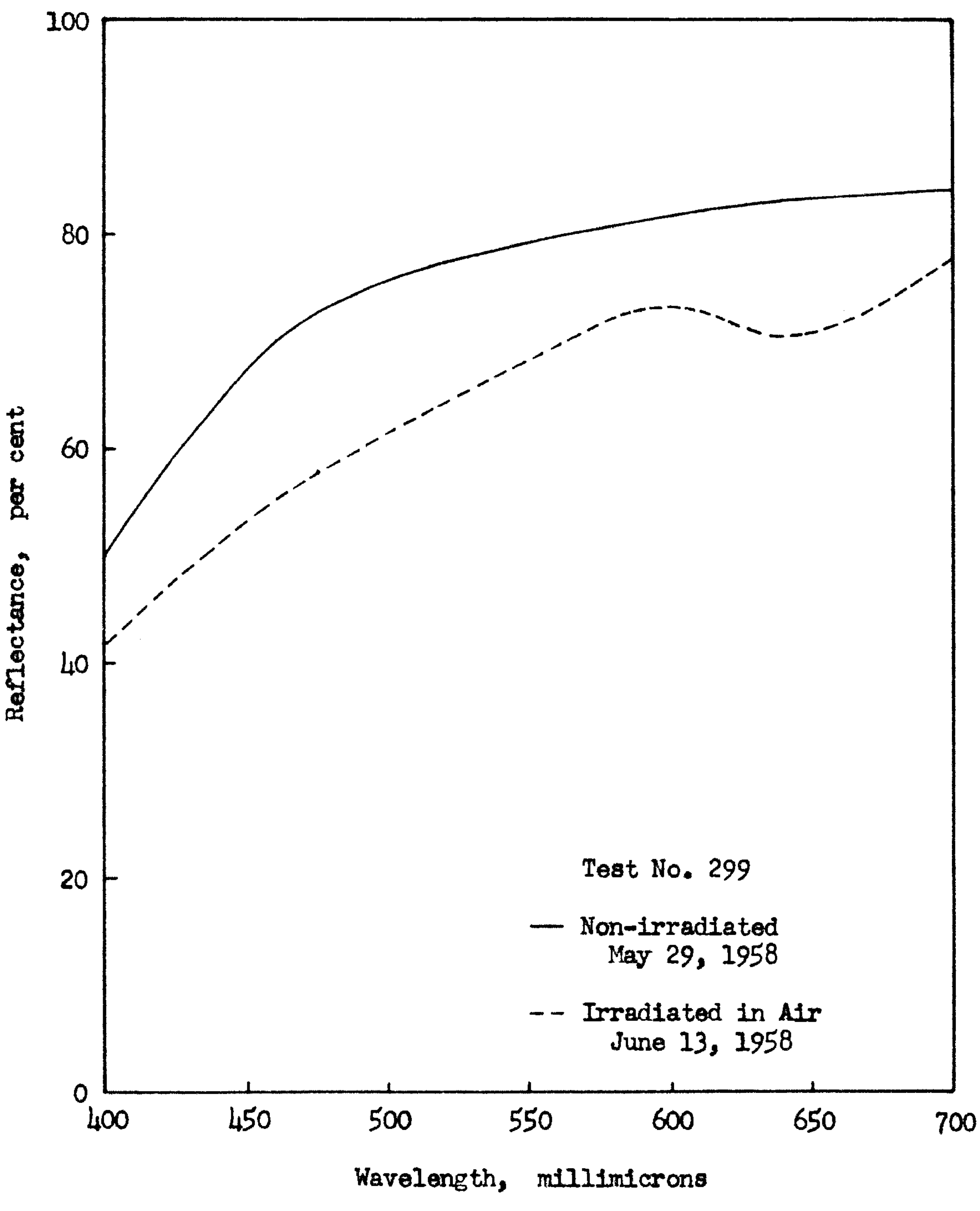

Figure 35. Light Reflectance of Alkyd Enamel Pigmented with Lead Carbonate.

Alkali Resistance. The results of the alkali tests on the coatings are presented in Table VI, page 93, and Figures 36 to 47, pages 95 to 106.

The overall effect of the alkali attack on the panels may be seen by referring to Table VI. The test was limited to any visible changes that could be observed. The changes fall into two types: (1) Discoloration to the film, and (2) damage that caused a physical change in the film other than color, such as wrinkling, blistering, or softening.

In the cases where positive results were obtained from the tests, figures have been drawn that show the time variation of the relative effect of the alkali on the irradiated and non-irradiated panels. Reference is made to these figures in Table VI opposite the coating they represent.

The relative effect is meant only for comparing the irradiated and non-irradiated panels with the same type of coating. Different colors, color intensities, and types of physical changes made it impossible to rate all the coatings on the same scale.

The irradiated and non-irradiated panels of one type of coating were rated at the same time. The test spot on either panel that had the most severe discoloration or other physical change was given a number rating of ten. Other test spots on both the panels were then given comparative number ratings from zero to ten. A zero rating indicated there was no visible effect. Other proportional ratings between zero and ten were given as the magnitude of the discoloration or other

TABLE VI

Results of Alkali Test on Irradiated and Non-Irradiated Coatings

Film Former	Pigment	Visible Effect on Panels	Relative Effect on Irradiated and Non-irradiated Panels
Vinyl Chloride Copolymer	none	FC[a]	See Figure 36, page 95.
	TiO_2	none	
	ZnO	none	
	$PbCO_3$	FC	See Figure 37, page 96.
Spar Varnish	none	FC, FF[b]	See Figure 38, page 97.
	TiO_2	FC, FF	See Figure 39, page 98.
	ZnO	FC, FF	See Figure 40, page 99.
	$PbCO_3$	FC, FF	See Figure 41, page 100.
	Al	FC, FF	See Figure 42, page 101.
	C	FF	See Figure 43, page 102.
Alkyd Resin	none	FC, FF	See Figure 44, page 103.
	TiO_2	FC, FF	See Figure 45, page 104.
	ZnO	FC, FF	See Figure 46, page 105.
	$PbCO_3$	FC, FF	See Figure 47, page 106.

---

[a] FC--Formation of colored reaction products at the spots where the drops of reagent were placed on the panel.

[b] FF--Failure of the film by wrinkling, blistering, or softening at the spots where the drops of reagent were placed on the panel.

physical change varied between none and the most severe. The film was said to have failed if it became wrinkled, blistered, of softened through to the metal substrate of the panel.

Acid Resistance. The results of the acid tests on the coatings are presented in Table VII, page 107, and Figures 48 to 54, pages 108 to 114.

The overall effect of the acid attack on the panels may be seen by referring to Table VII. The test was limited to any visible changes that could be observed. The only change observed was a discoloration of the film at spots where the drops of acid were placed.

In cases where positive results were obtained from the tests, figures have been drawn that show the time variation of the relative effect of the acid on the irradiated and non-irradiated panels. Reference is made to these figures in Table VII opposite the coating they represent.

The results of this test were obtained by giving numerical ratings to the test spots on the panels in the same method used for the alkali test (see page 92).

Flexibility. The results of the flexibility tests are shown in Table VIII, page 115. The minimum diameter of the bend in the test panel that did not cause failure of the coating is tabulated opposite the coating it represents. The diameter of the bend corresponds to the size of mandrel used for the test. The smallest size mandrel available was one-eighth inch in diameter.

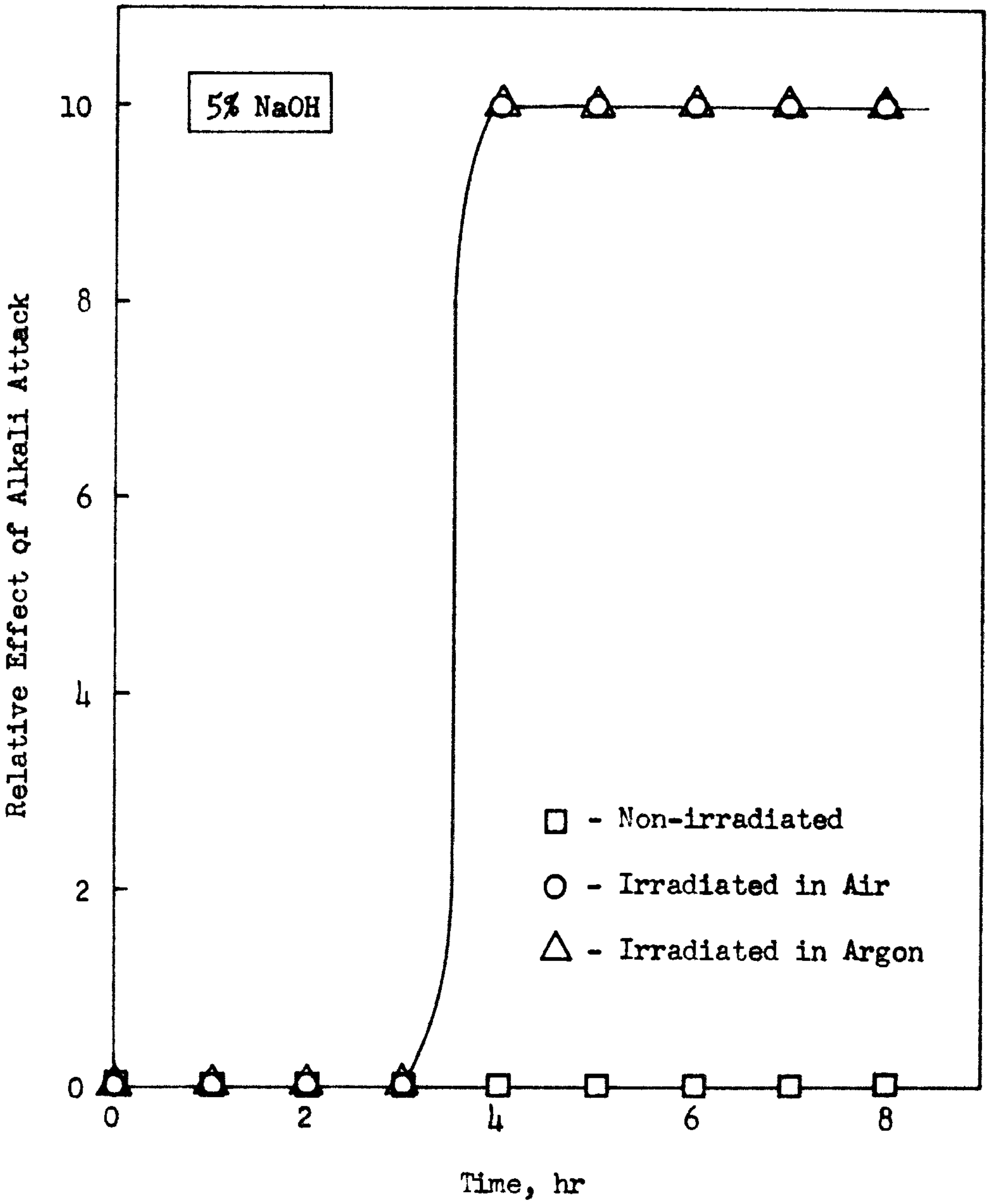

Figure 36. Relative Effect of Alkali Attack on Vinyl Chloride Copolymer Varnish.

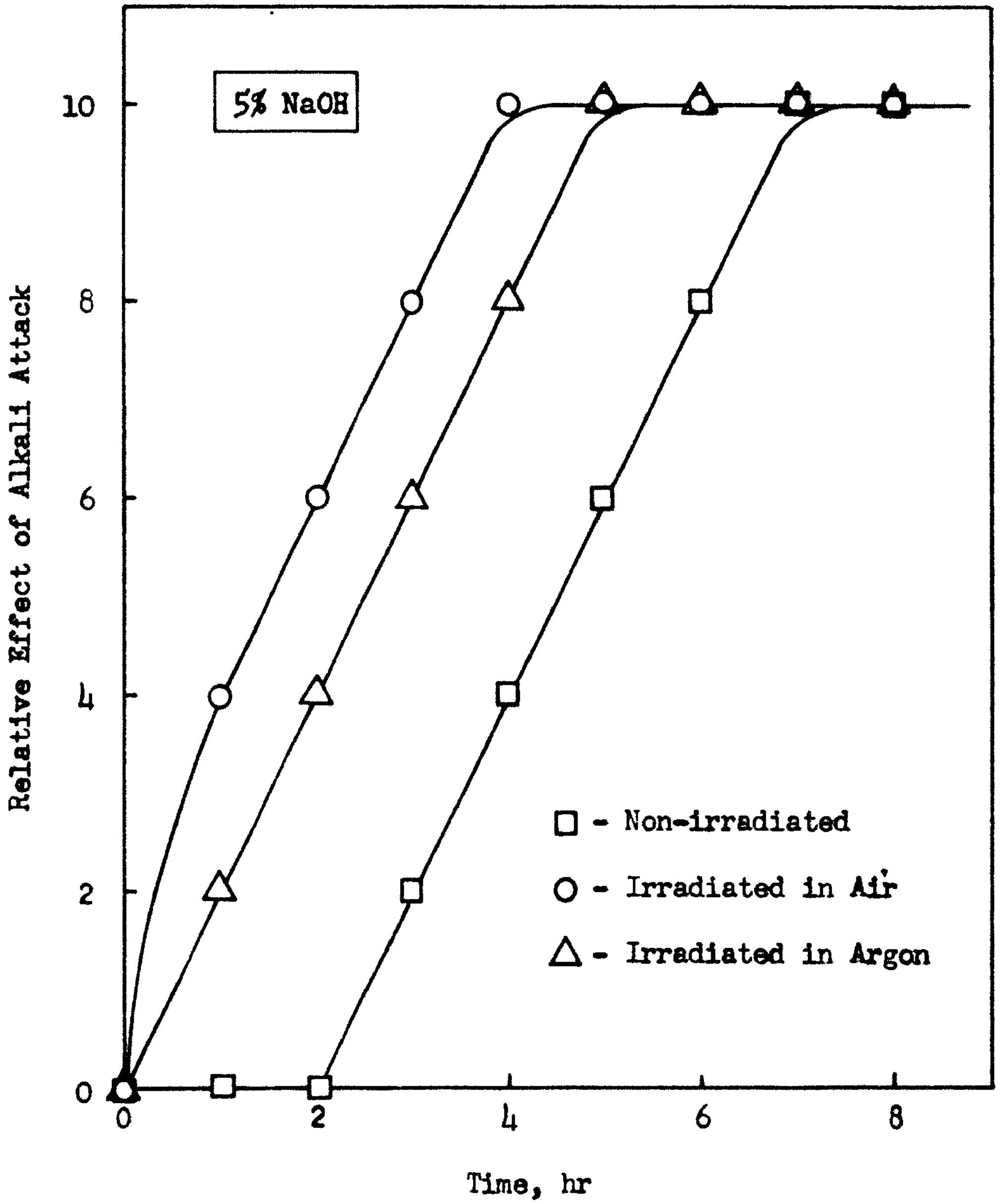

Figure 37. Relative Effect of Alkali Attack on Vinyl Chloride Copolymer Enamel Pigmented with Lead Carbonate.

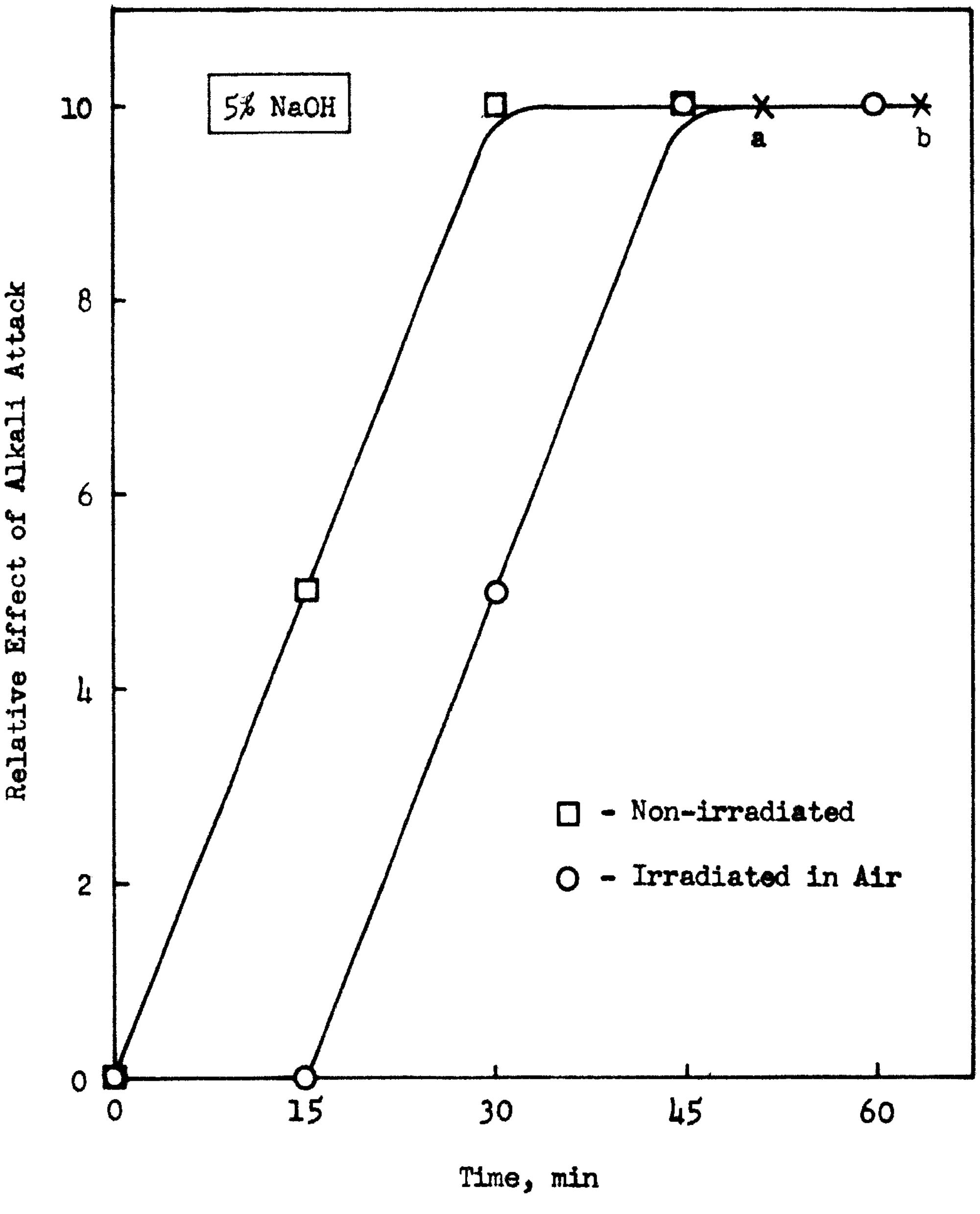

Figure 38. Relative Effect of Alkali Attack on Clear Spar Varnish.

[a] Non-irradiated film failed.
[b] Irradiated film failed.

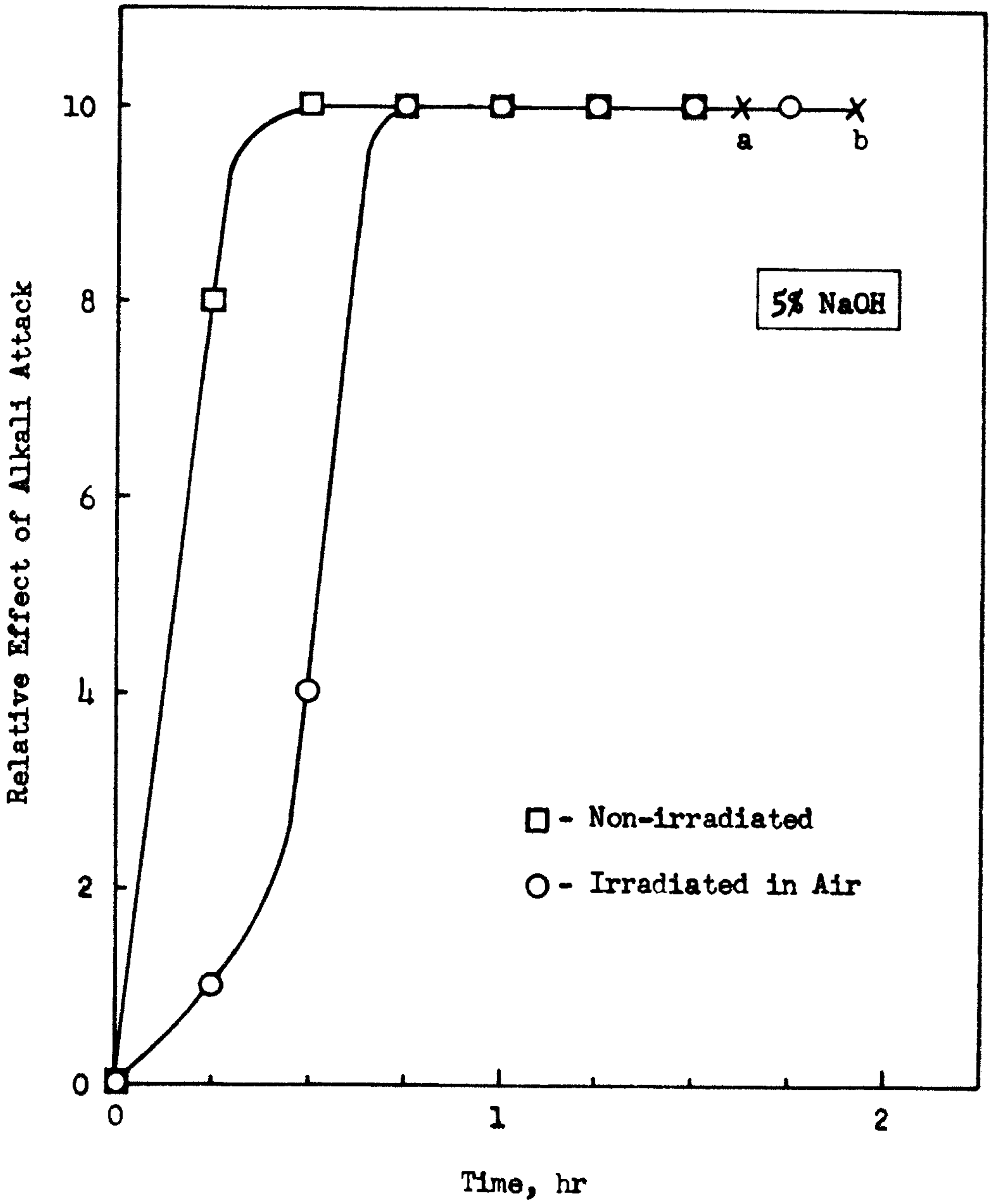

Figure 39. Relative Effect of Alkali Attack on Spar Enamel Pigmented with Titanium Dioxide.

[a] Non-irradiated film failed.
[b] Irradiated film failed.

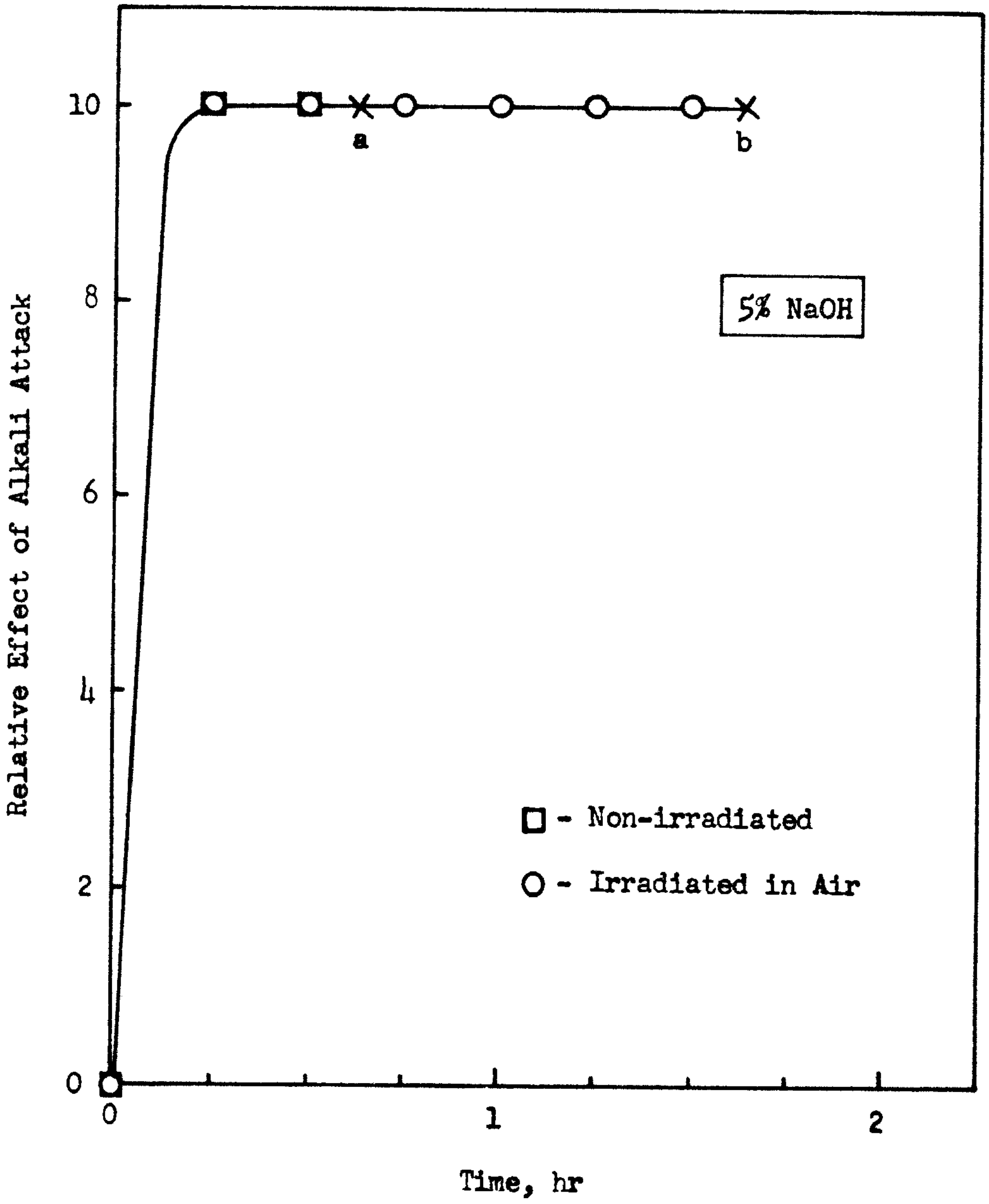

Figure 40. Relative Effect of Alkali Attack on Spar Enamel Pigmented with Zinc Oxide.

[a] Non-irradiated film failed.
[b] Irradiated film failed.

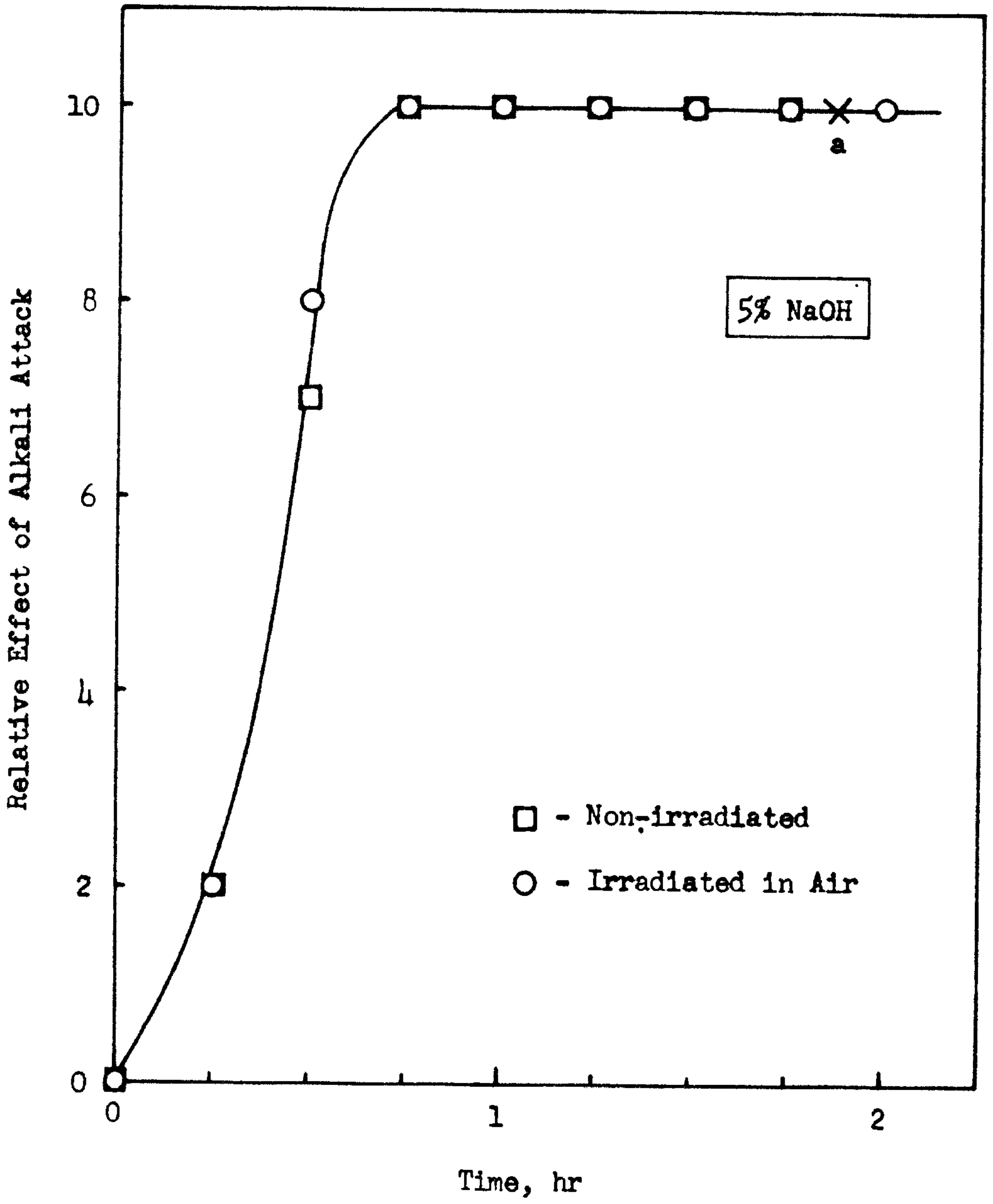

Figure 41. Relative Effect of Alkali Attack on Spar Enamel Pigmented with Lead Carbonate.

[a]Non-irradiated film failed.

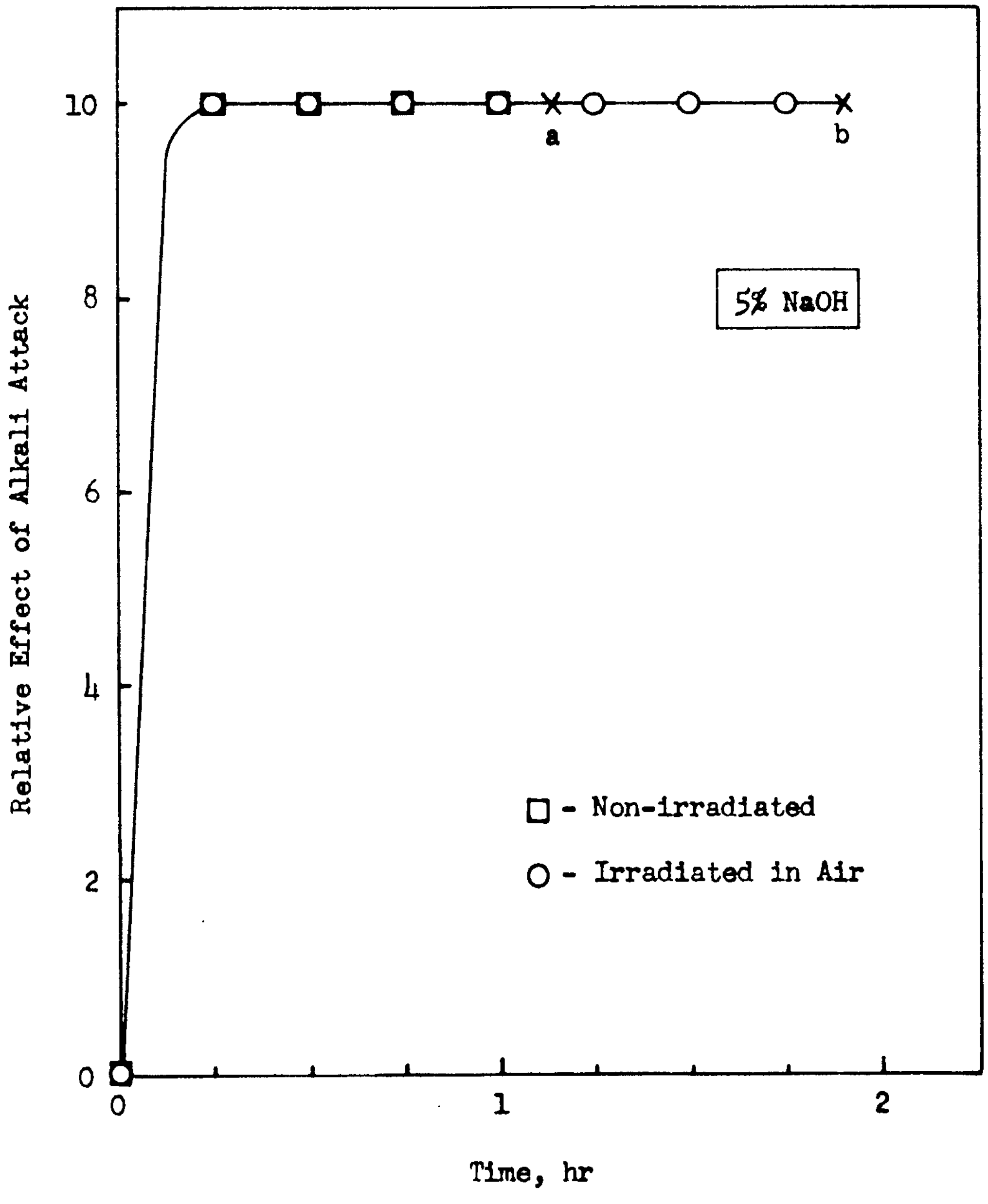

Figure 42. Relative Effect of Alkali Attack on Spar Enamel Pigmented with Aluminum Paste.

[a] Non-irradiated film failed.
[b] Irradiated film failed.

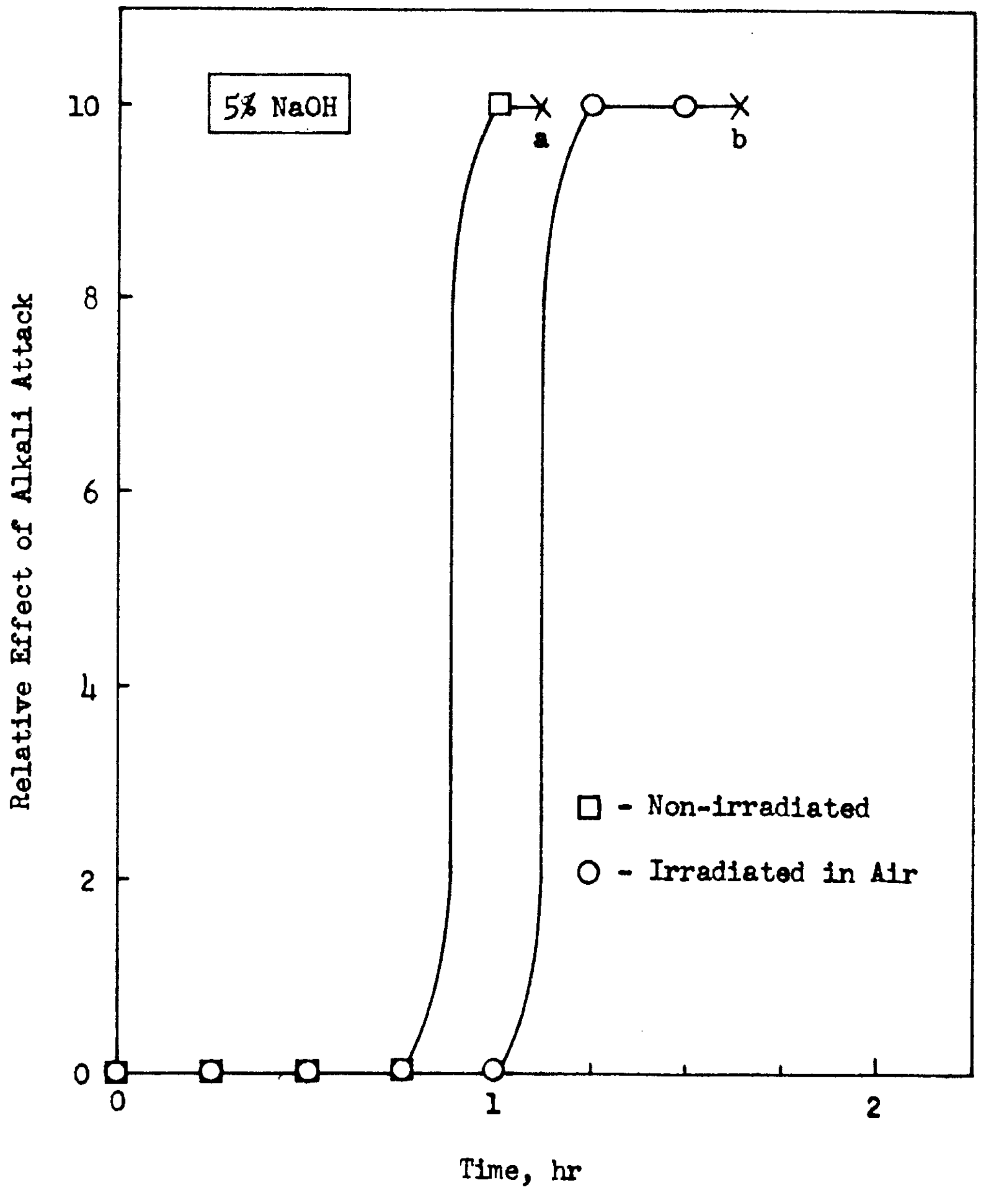

Figure 43. Relative Effect of Alkali Attack on Spar Enamel Pigmente with Lampblack.

[a] Non-irradiated film failed.
[b] Irradiated film failed.

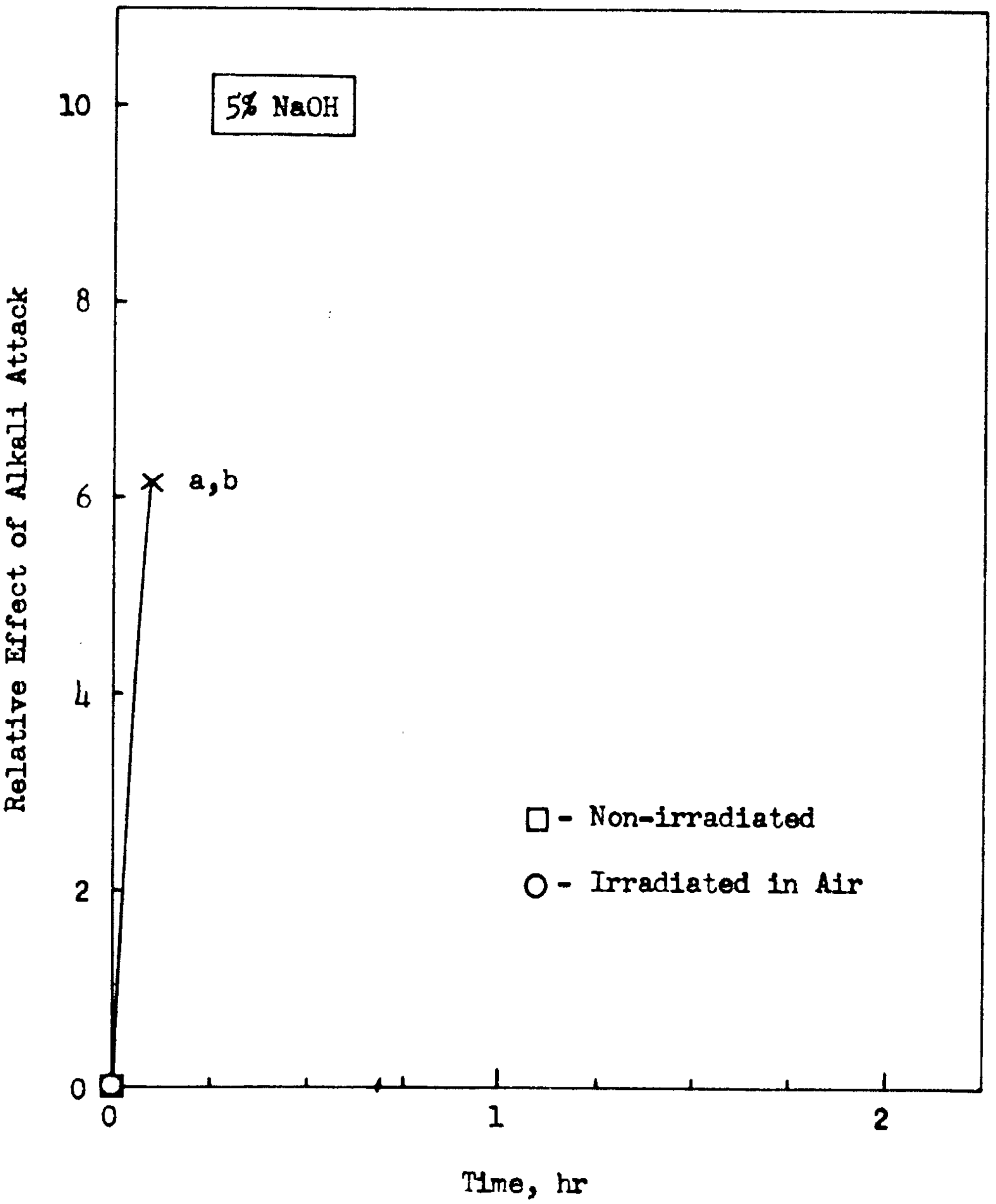

Figure 44. Relative Effect of Alkali Attack on Clear Alkyd Varnish.

[a]Non-irradiated film failed.
[b]Irradiated film failed.

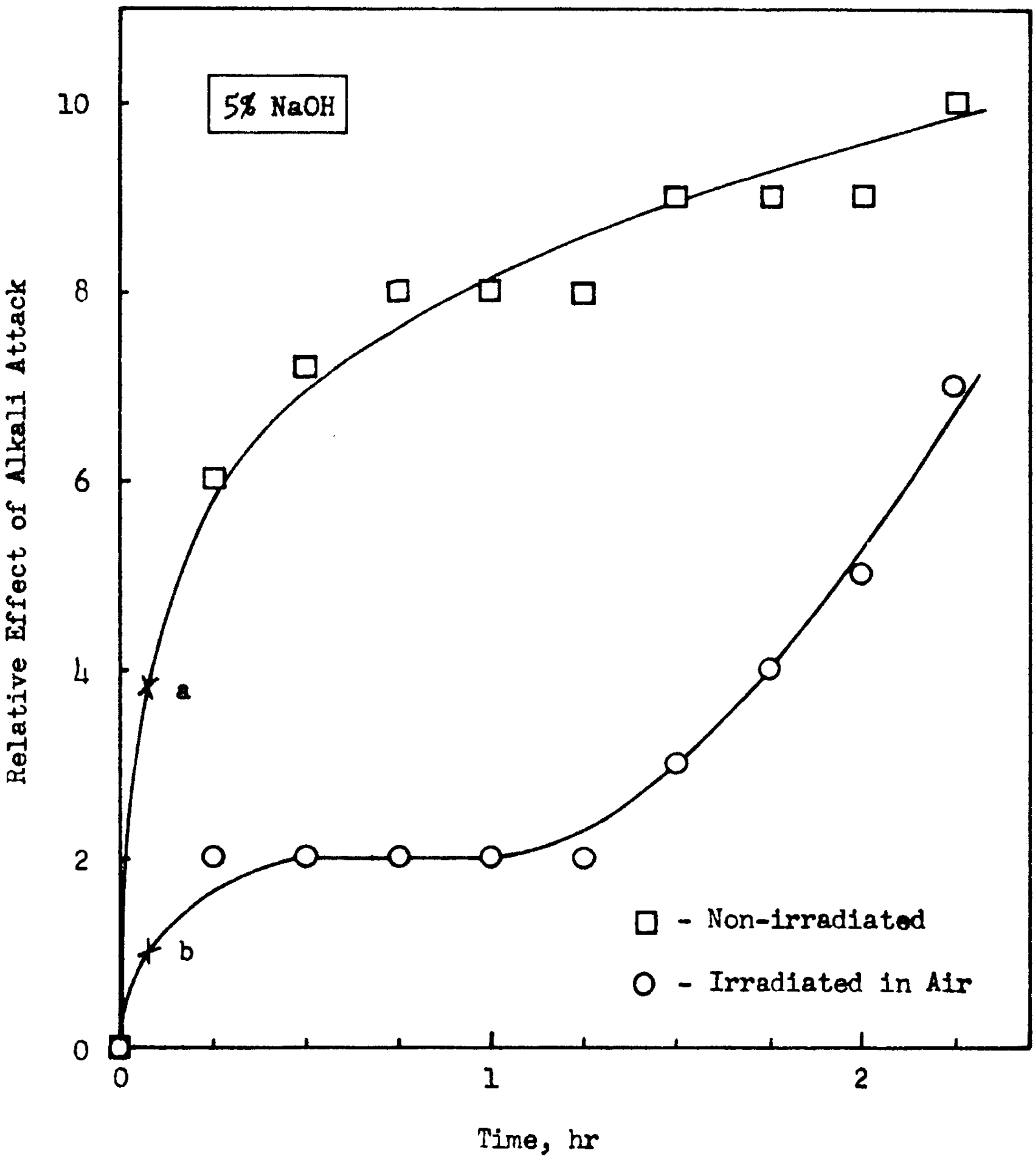

Figure 45. Relative Effect of Alkali Attack on Alkyd Enamel Pigmente with Titanium Dioxide.

[a] Non-irradiated film failed.
[b] Irradiated film failed.

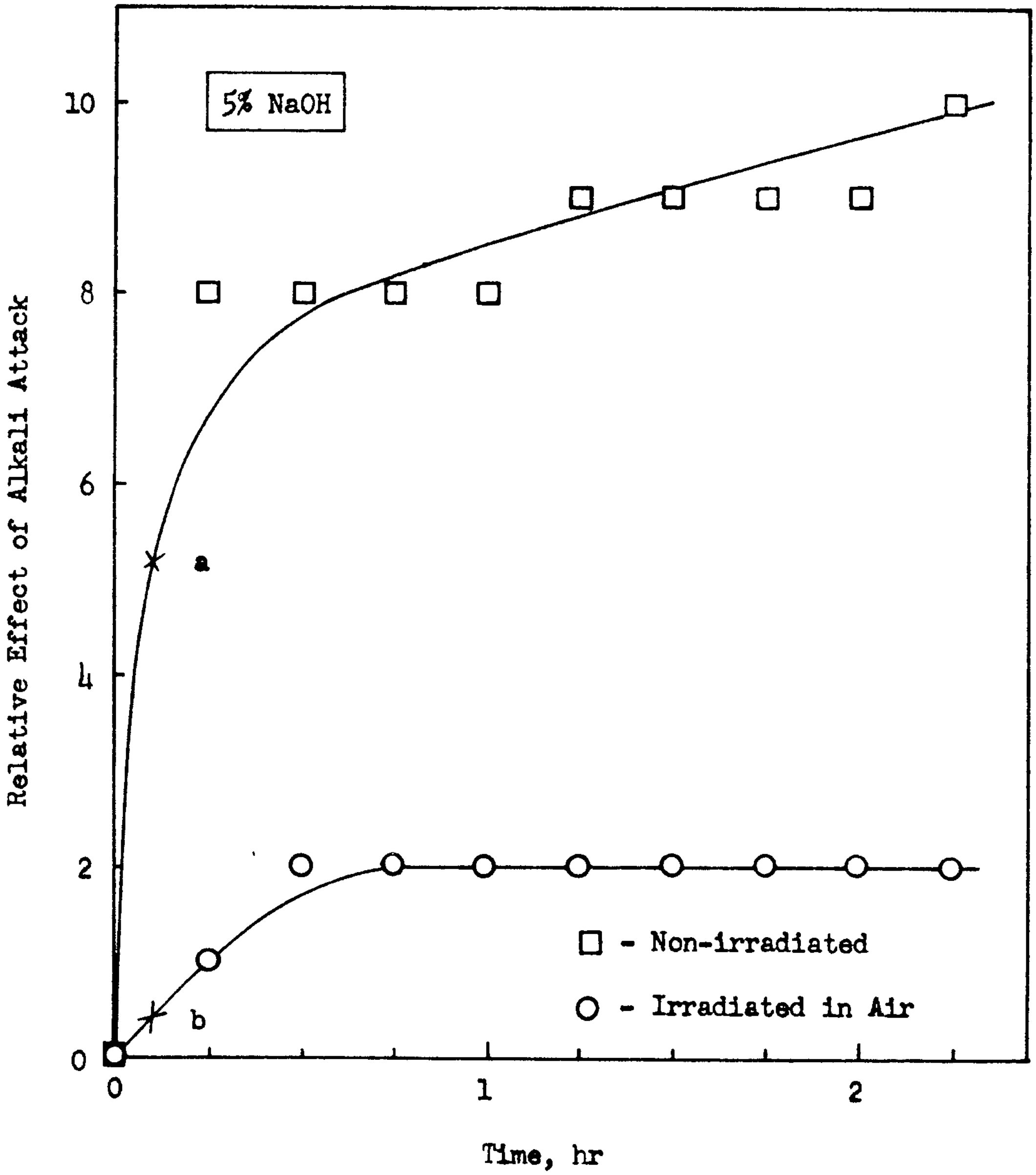

Figure 46. Relative Effect of Alkali Attack on Alkyd Enamel Pigmente with Zinc Oxide.

[a] Non-irradiated film failed.
[b] Irradiated film failed.

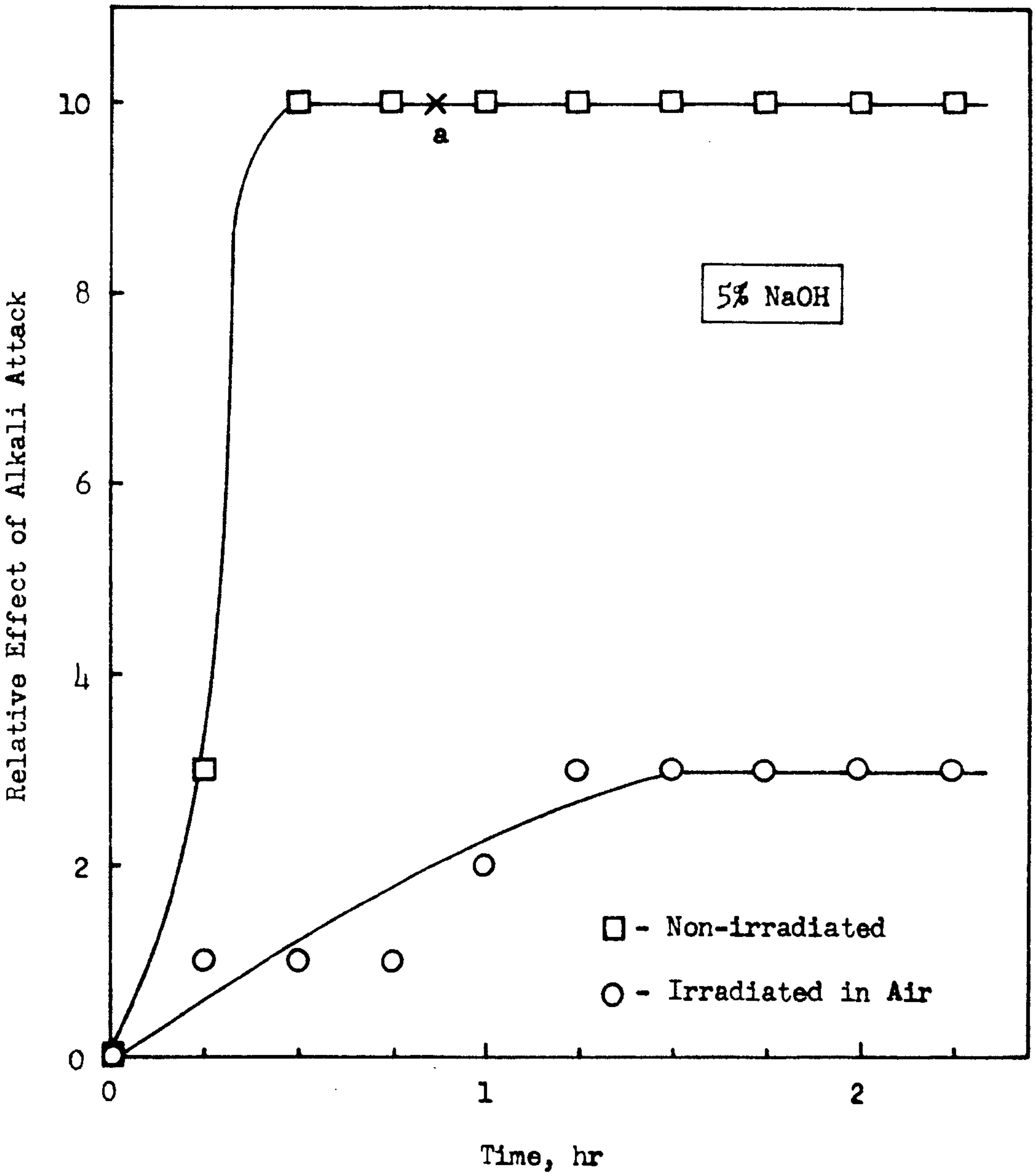

Figure 47. Relative Effect of Alkali Attack on Alkyd Enamel Pigment with Lead Carbonate.

[a]Non-irradiated film failed.

TABLE VII

Results of Acid Test on Irradiated and Non-Irradiated Coatings

Film Former	Pigment	Visible Effect on Panels	Relative Effect on Irradiated and Non-irradiated Panels
Vinyl Chloride Copolymer	none	none	
	TiO_2	none	
	ZnO	none	
	$PbCO_3$	none	
Spar Varnish	none	FC[a]	See Figure 48, page 108
	TiO_2	FC	See Figure 49, page 109
	ZnO	FC	See Figure 50, page 110
	$PbCO_3$	none	
	Al	FC	See Figure 51, page 111
	C	none	
Alkyd Resin	none	FC	See Figure 52, page 112
	TiO_2	FC	See Figure 53, page 113
	ZnO	FC	See Figure 54, page 114
	$PbCO_3$	none	

[a] FC--Formation of colored reaction products at the spots where the drops of reagent were placed on the panel.

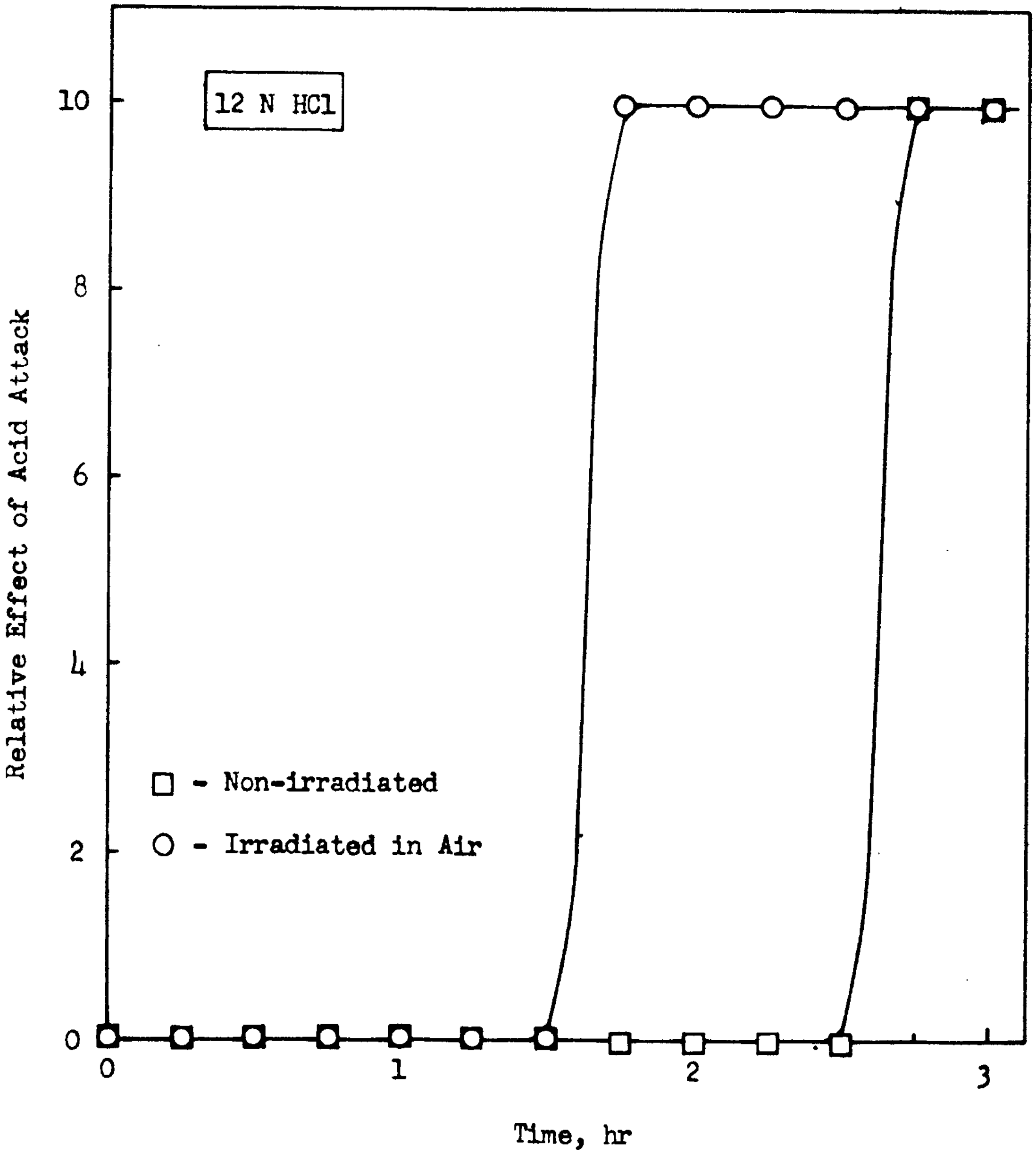

Figure 48. Relative Effect of Acid Attack on Clear Spar Varnish.

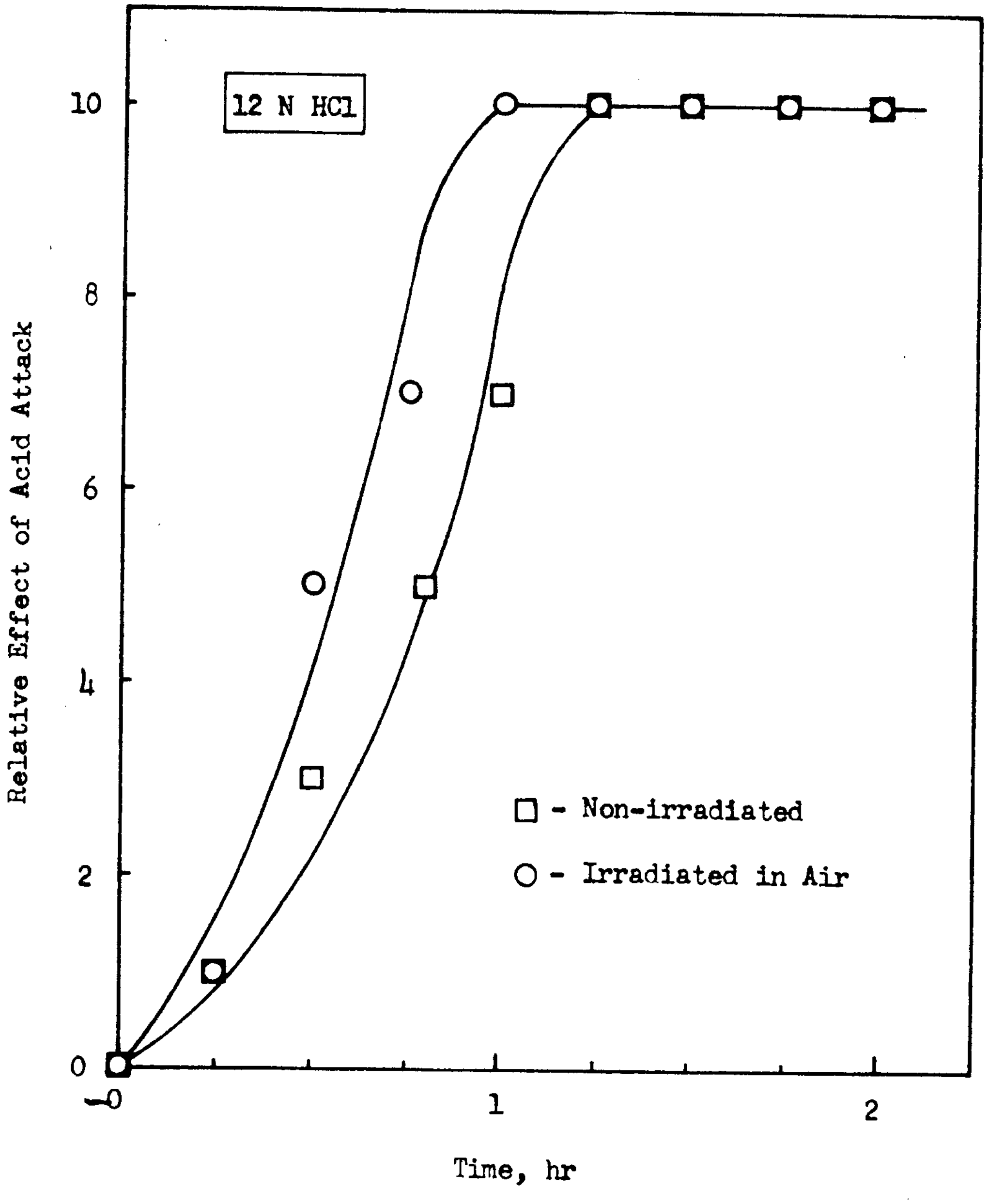

Figure 49. Relative Effect of Acid Attack on Spar Enamel Pigmented with Titanium Dioxide.

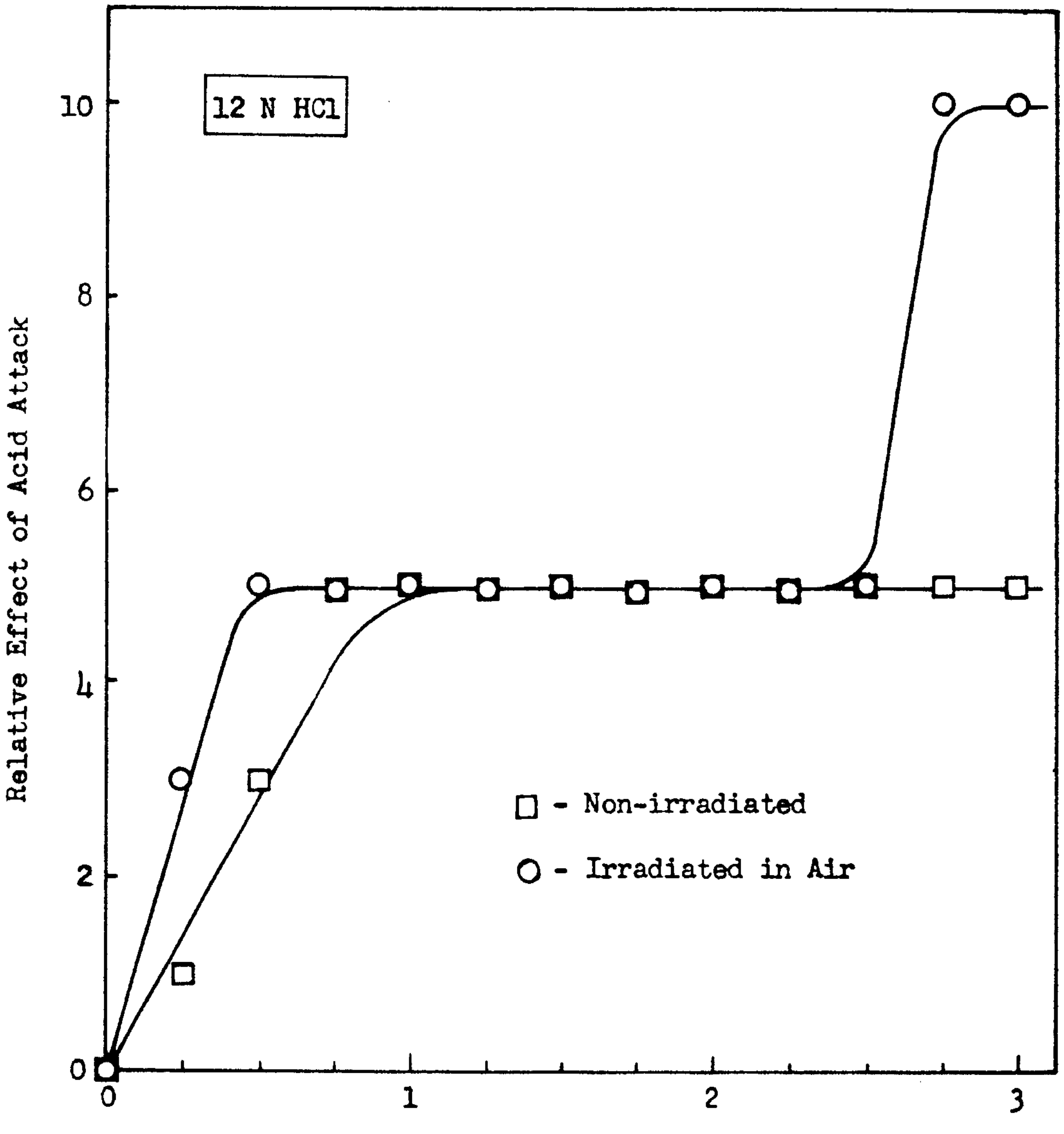

Figure 50. Relative Effect of Acid Attack on Spar Enamel Pigmented with Zinc Oxide.

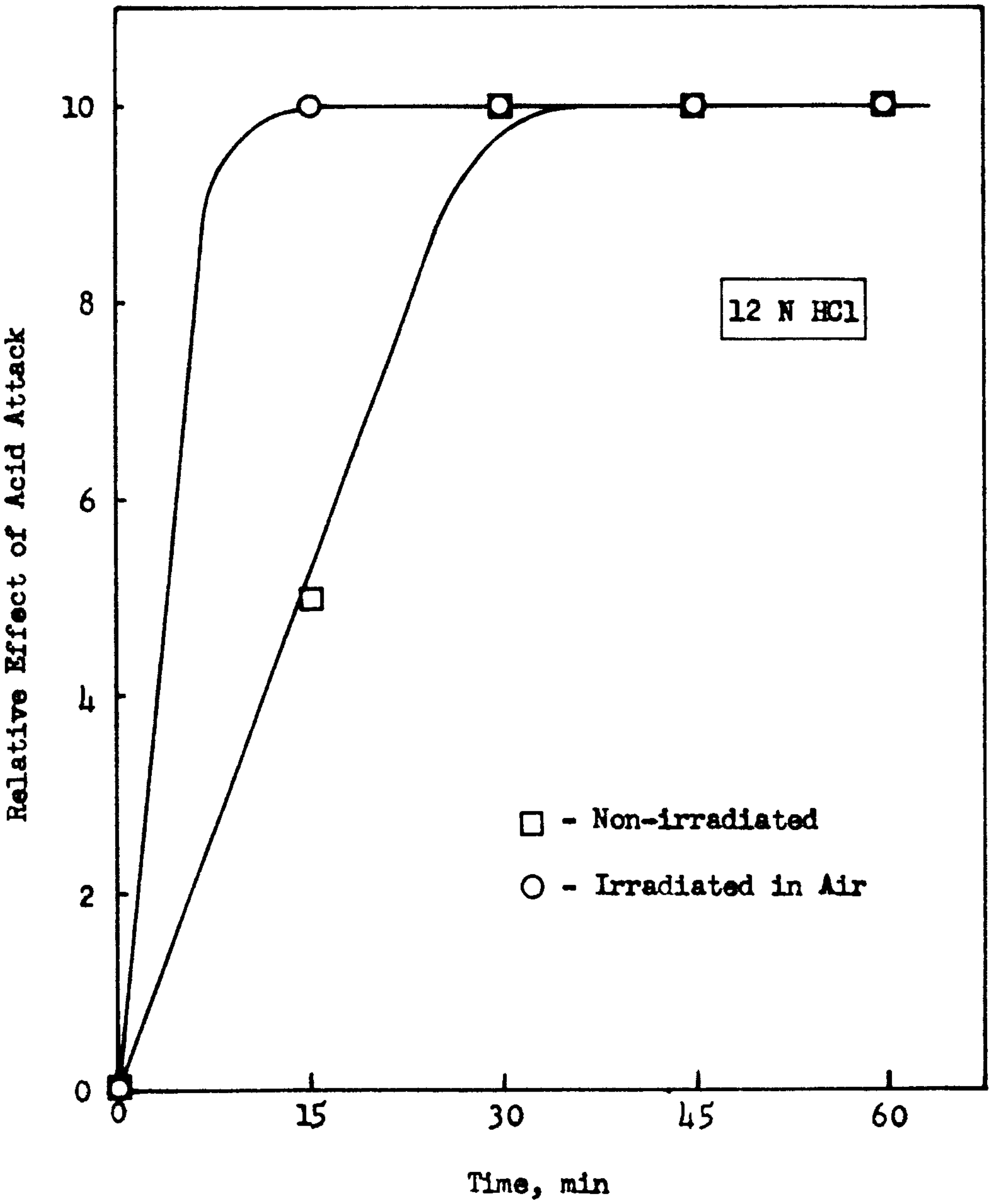

Figure 51. Relative Effect of Acid Attack on Spar Enamel Pigmented with Aluminum Paste.

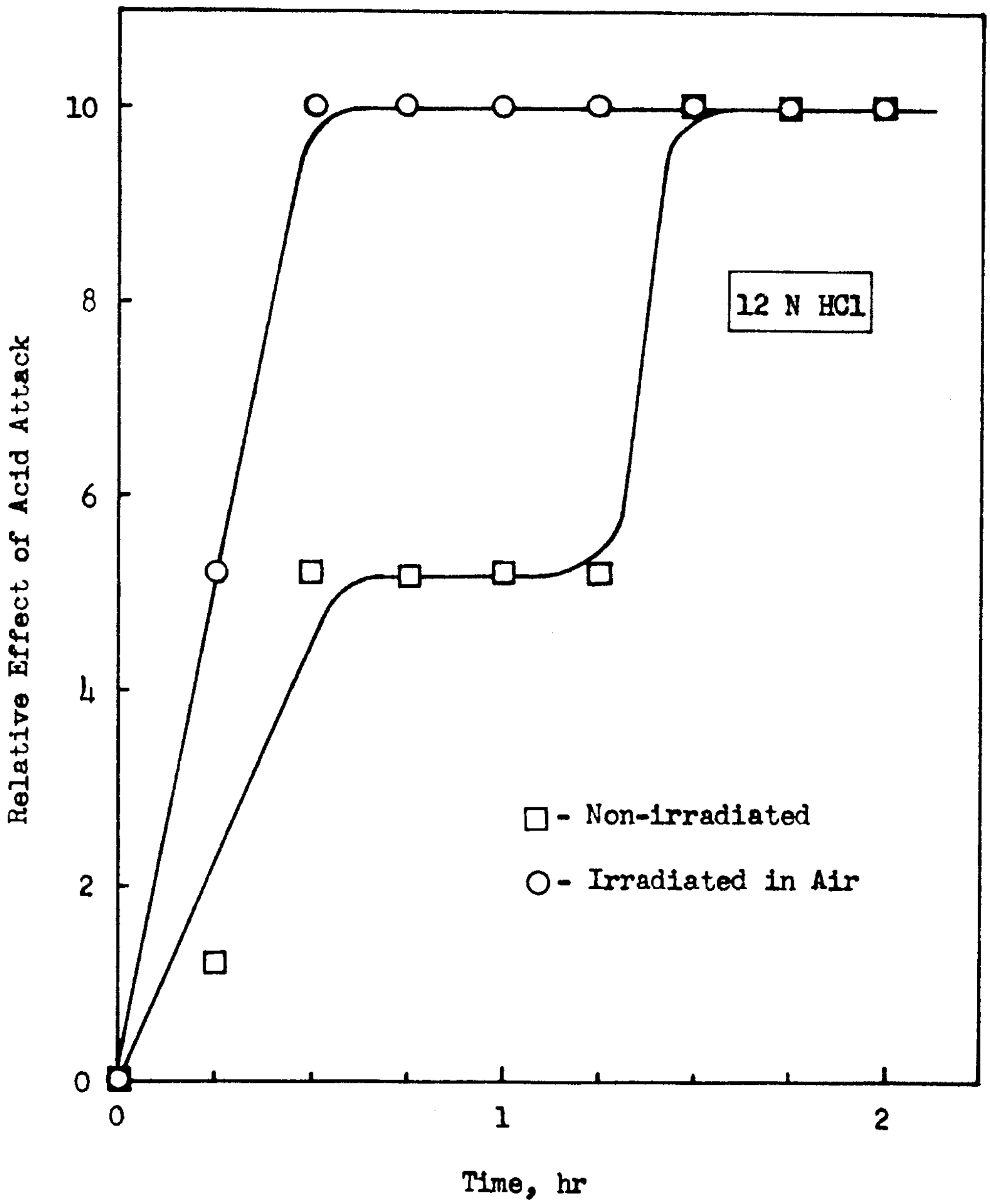

Figure 52. Relative Effect of Acid Attack on Clear Alkyd Varnish.

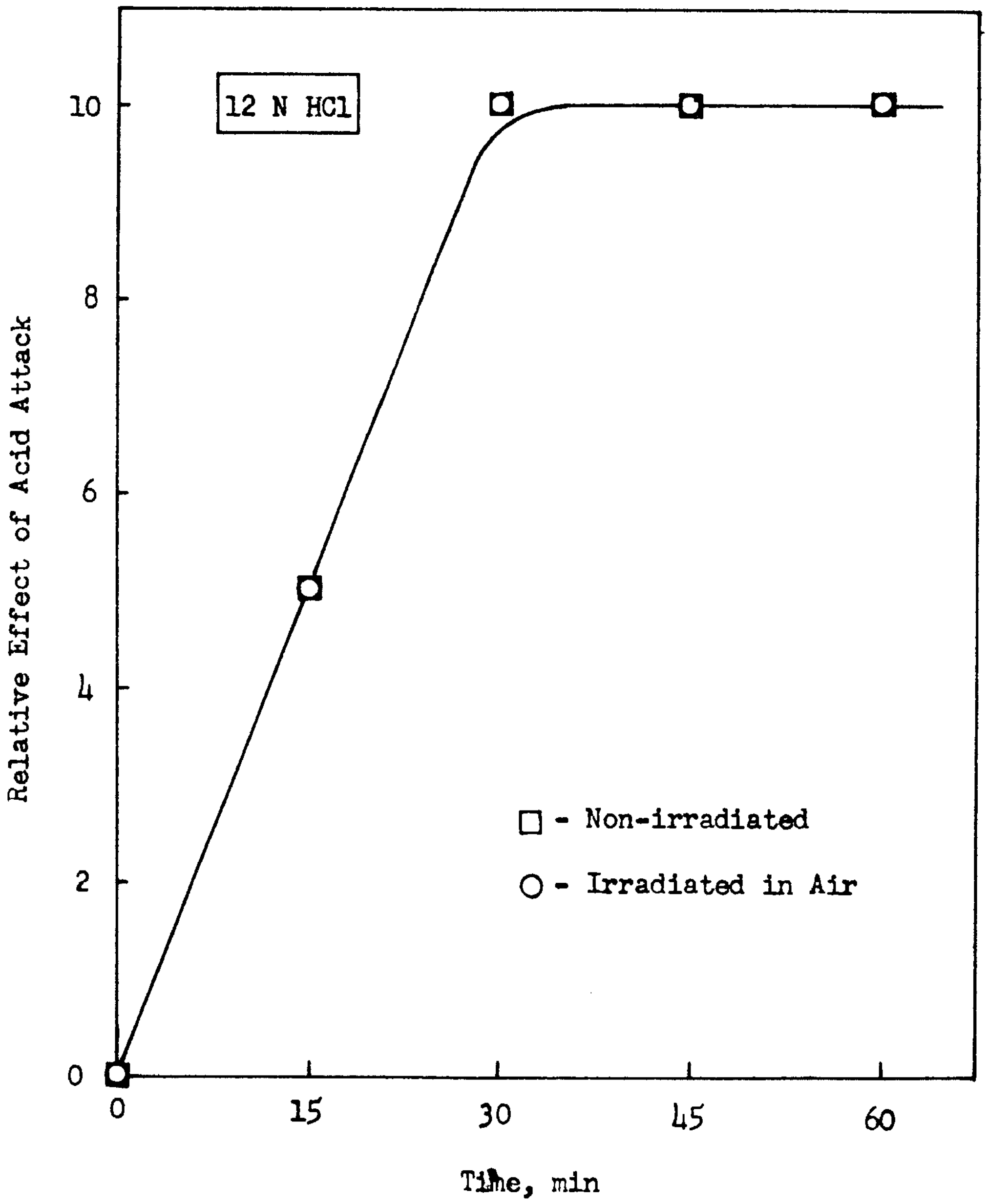

Figure 53. Relative Effect of Acid Attack on Alkyd Enamel Pigmented with Titanium Dioxide.

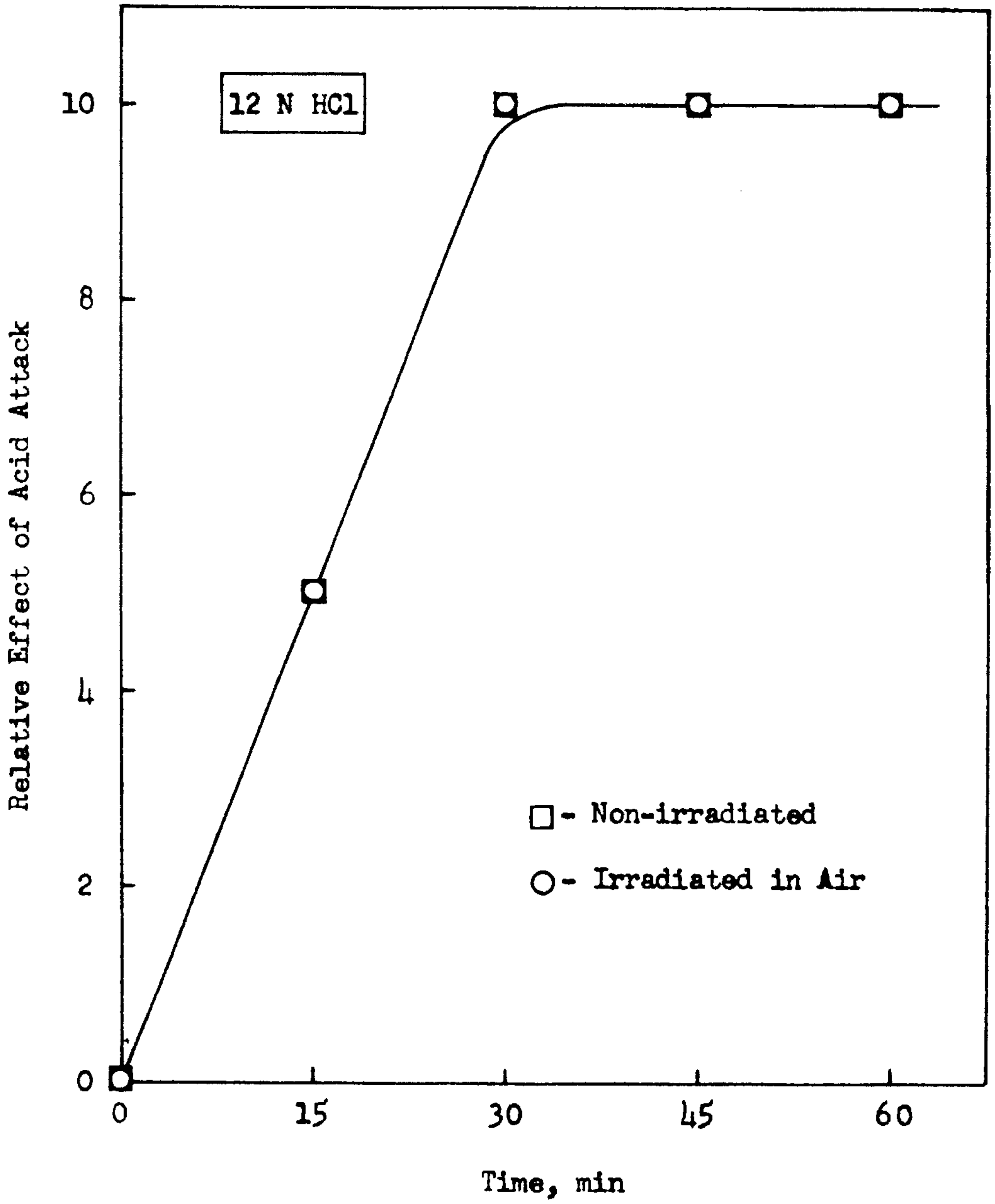

Figure 54. Relative Effect of Acid Attack on Alkyd Enamel Pigmented with Zinc Oxide.

TABLE VIII

Flexibility of Irradiated and Non-Irradiated Coatings

		Minimum Diameter of Bend without Failure of Coating		
Film Former	Pigment	Non-irradiated	Irradiated in Air	Irradiated in Argon
		inch	inch	inch
Vinyl Chloride Copolymer	none	1/8	1/4	1/4
	TiO_2	1/8	3/16	3/16
	ZnO	1/8	1/8	1/8
	$PbCO_3$	1/8	1/8	1/8
Spar Varnish	none	1/8	1/8	
	TiO_2	1/8	1/8	
	ZnO	1/8	1/8	
	$PbCO_3$	1/8	1/8	
	Al	1/8	1/8	
	C	1/8	1/4	
Alkyd Resin	none	1/8	1/8	
	TiO_2	1/8	1/8	
	ZnO	5/16	3/4	
	$PbCO_3$	1/8	1/8	

Abrasion Resistance. The data and results of the abrasion resistance tests are shown in Table IX, page 117.

The weight loss that the panels suffered in the abrasion test has been expressed as milligrams per ten-thousand test cycles. This places all the panels on an equal basis so that the entire group may be compared.

The weight loss per ten-thousand cycles was calculated to be directly proportional to the total weight loss for the total number of test cycles. A sample calculation is shown on page 124.

Scratch Hardness. The results of the scratch hardness tests are shown in Table X, page 118. A constant rider weight on the scratch hardness tester was used to test an entire series of panels.

The effect of the scratching tool was described in Table X by the abbreviations, *N. S.*, *I. S.*, *S. S.*, and *D. S.* The abbreviation *N. S.* (no scratch) was used to describe the condition when the tool slid along the surface of the coating, making a mark or indentation, but not penetrating the surface. The abbreviation *I. S.* (intermitten scratch) was used when the tool made an indentation with an occasional spot being scratched. A shallow, continuous scratch was described by the abbreviation *S. S.* (slight scratch) and a deep continuo scratch by *D. S.* (deep scratch).

Impact Resistance. The results of the impact tests are shown in Table XI, page 119. The maximum impact in inch-pounds that the panels could withstand without failure of the coating is shown opposi the coating on which the test was performed.

TABLE IX

Weight Loss of Irradiated and Non-Irradiated Coatings During Abrasion Test

Film Former	Pigment	Total Number of Test Cycles	-Weight Loss of Coatings-					
			Non-irradiated		Irradiated in Air		Irradiated in Argon	
			Total	Per 10^4 Cycles	Total	Per 10^4 Cycles	Total	Per 10^4 Cycles
			mg	mg	mg	mg	mg	mg
Vinyl Chloride Copolymer	none	15,292	0.9	0.6	2.5	1.6	2.3	1.5
	TiO_2	14,500	0.5	0.3	1.6	1.1	1.2	0.8
	ZnO	14,535	9.8	6.7	9.7	6.7	9.5	6.5
	$PbCO_3$	14,436	10.9	7.6	4.7	3.3	5.6	3.9
Spar Varnish	none	14,500	0.8	0.6	1.5	1.0		
	TiO_2	14,575	2.5	1.7	4.2	2.9		
	ZnO	136,450	8.9	0.65	7.0	0.51		
	$PbCO_3$	18,500	1.3	0.70	1.4	0.76		
	Al	34,700	2.3	0.66	1.6	0.46		
	C	32,850	3.0	0.91	5.1	1.6		
Alkyd Resin	none	14,500	3.2	2.2	4.7	3.2		
	TiO_2	14,700	6.1	4.1	6.3	4.3		
	ZnO	24,443	7.7	3.1	11.3	4.6		
	$PbCO_3$	14,500	7.7	5.3	8.2	5.7		

TABLE X

Scratch Hardness of Irradiated and Non-Irradiated Coatings

			Effect of Scratching Tool on Coatings		
Film Former	Pigment	Load	Non-irradiated	Irradiated in Air	Irradiated in Argon
Vinyl Chloride Copolymer	none	1,000 gm	I. S.[a]	D. S.[b]	D. S.
	TiO_2	1,000	I. S.	I. S.	I. S.
	ZnO	1,000	I. S.	I. S.	I. S.
	$PbCO_3$	1,000	S. S.[c]	D. S.	D. S.
Spar Varnish	none	2,400 gm	S. S.	D. S.	
	TiO_2	2,400	N. S.[d]	D. S.	
	ZnO	2,400	N. S.	I. S.	
	$PbCO_3$	2,400	S. S.	S. S.	
	Al	2,400	D. S.	D. S.	
	C	2,400	D. S.	D. S.	
Alkyd Resin	none	1,200 gm	I. S.	S. S.	
	TiO_2	1,200	N. S.	I. S.	
	ZnO	1,200	I. S.	D. S.	
	$PbCO_3$	1,200	N. S.	N. S.	

[a] I. S.--Intermittent scratch.
[b] D. S.--Deep scratch.
[c] S. S.--Slight scratch.
[d] N. S.--No scratch.

TABLE XI

Impact Resistance and Adhesion of Irradiated and Non-Irradiated Coatings

		- - - - - - - - - -Maximum Impact without Failure- - - - - - - -					
		Non-irradiated		Irradiated in Air		Irradiated in Argon	
Film Former	Pigment	Impact	Adhesion	Impact	Adhesion	Impact	Adhesion
		in.-lb		in.-lb		in.-lb	
Vinyl Chloride Copolymer	none	1	Good	<1	Very Poor	<1	Very Poor
	TiO_2	16	Good	2	Poor	2	Poor
	ZnO	12	Poor	1	Very Poor	1	Very Poor
	$PbCO_3$	10	Good	2	Good	4	Good
Spar Varnish	none	>30	Good	>30	Good		
	TiO_2	>30	Fair	>30	Fair		
	ZnO	10	Poor	14	Poor		
	$PbCO_3$	>30	Poor	>30	Poor		
	Al	6	Poor	6	Fair		
	C	4	Poor	4	Poor		
Alkyd Resin	none	>30	Good	>30	Good		
	TiO_2	>30	Fair	>30	Fair		
	ZnO	< 1	Poor	< 1	Very Poor		
	$PbCO_3$	>30	Fair	>30	Fair		

The upper limit of the testing range was thirty inch-pounds. A number of the panels tested withstood the maximum impact without failure. They are indicated in Table XI, page 119, by the symbol " >30".

It was not possible to determine impact resistances less than one inch-pound. Panels which failed for the one inch-pound impact are indicated in Table XI by the symbol " <1".

The panels subjected to the impact test were also used to determine a very qualitative measure of the adhesion of the coating to the metal panel. The area that had been bulged out from an impact test was used for the determination. The adhesion ratings were: (1) Good--coating was adhered to the panel so that it had to be cut or scraped with a pen knife for removal; (2) fair--coating was adhered to the panel, but small areas could be removed by running the point of a knife under the film; (3) poor--the coating was not adhered to the area of the impact test, but was adhered to the panel around this area; and (4) very poor--the coating was not adhered to the area of the impact test and had been pulled away from the panel in the immediate area around the impact test.

Sample Calculations

Calculation of Weight of Pigment for Enamels. The calculations to determine the weight of pigment for the three series of enamels are presented separately.

Vinyl Chloride Copolymer Enamel. The calculation shown here is for the enamel pigmented with titanium dioxide. The calculations for the amount of pigment in the other enamels in this series were made in a similar manner.

Basis: 253.9 gm vinyl chloride copolymer varnish. (page 35).

$$\frac{\text{Weight resin solids}}{\text{Weight pigment}} = \frac{19}{12} \qquad \text{(page 35)}$$

$$\frac{\text{gm resin}}{\text{gm varnish}} = \frac{200}{600} \qquad \text{(page 32)}$$

$$\text{gm pigment} = \text{gm varnish} \times \frac{\text{gm resin}}{\text{gm varnish}} \times \frac{\text{gm pigment}}{\text{gm resin}}$$

$$= 253.9 \times \frac{200}{600} \times \frac{12}{19}$$

$$= 53.5$$

Spar Enamel. The calculation shown here is for the enamel pigmented with titanium dioxide. The calculations for the amount of pigment in the other enamels in this series were made in a similar manner.

Basis: 500 gm spar varnish (page 37)

$$\frac{\text{Weight resin solids}}{\text{Weight pigment}} = \frac{19}{12} \qquad \text{(page 37)}$$

Per cent solids = 55 (page 27)

$$\text{gm pigment} = \text{gm varnish} \times \frac{\text{gm solid}}{\text{gm varnish}} \times \frac{\text{gm pigment}}{\text{gm solids}}$$

$$= 500 \times \frac{55}{100} \times \frac{12}{19}$$

$$= 173.7$$

Alkyd Enamel. The calculation shown here is for the enamel pigmented with titanium dioxide. The calculations for the amount of pigment in the other enamels in this series were made in a similar manner.

Basis: 250 ml alkyd resin solution (page 40)

$$\frac{\text{Weight resin solids}}{\text{Weight pigment}} = \frac{19}{12} \qquad \text{(page 40)}$$

$$\text{Density of solution} = 8.00 \text{ lbs/gal} \qquad \text{(page 26)}$$

$$= 0.960 \text{ gm/ml}$$

$$\text{Per cent solids} = 70 \qquad \text{(page 26)}$$

$$\text{gm pigment} = \text{ml solution} \times \frac{\text{gm solution}}{\text{ml solution}} \times \frac{\text{gm resin}}{\text{gm solution}} \times \frac{\text{gm pigment}}{\text{gm resin}}$$

$$= 250 \times \frac{0.960}{1} \times \frac{70}{100} \times \frac{12}{19}$$

$$= 106$$

Calculation of Film Thickness on Panels. The following equation was used to calculate the film thickness of the coatings on the test panels.

$$T = \frac{394\,W}{D\,A} \qquad (8)$$

where, T = film thickness of coating, mils.

W = weight of coatings on panel, gm.

D = density of coating, gm/cc.

A = area of panel coated, sq cm.

394 = factor to convert cm to mils.

Coatings were applied to the same area on all the test panels used in this investigation. The area may be calculated by referring to the dimensions of the panels, page 30, and the portion of the panels that was coated, page 43.

$$\begin{aligned} A &= 2 \text{ x area of one side of coated panel} \\ &= 2 \times 6 \times 2\text{-}3/8 \\ &= 28.5 \text{ sq in.} \\ &= 184 \text{ sq cm} \end{aligned}$$

Substituting this value in equation (8), page 122, gives,

$$T = \frac{2.14\ W}{D}. \tag{9}$$

The weight (W) of the coating for use in the above equation was obtained by weighing the test panels before and after the coating was applied.

The density (D) of the coatings was calculated from the density of the resin solids and pigment. A sample calculation is shown for the vinyl chloride copolymer enamel pigmented with titanium dioxide.

Specific gravity of TiO_2 = 4.2 (page 26)

Specific gravity of resin = 1.31 (page 26)

Specific gravity = Density, gm/cc

$$\frac{\text{Weight resin}}{\text{Weight pigment}} = \frac{19}{12} \qquad \text{(page 35)}$$

$$D = (\text{weight-fraction resin x density resin}) + (\text{weight-fraction pigment x density pigment})$$

$$= \frac{19}{31}(1.31) + \frac{12}{31}(4.2)$$

$$= 2.43$$

The densities of the other coatings were calculated in a similar manner. The calculated values are shown in Table XII, page 125.

Calculation of the Weight Loss per Ten Thousand Cycles for Abrasion Resistance Test. The weight loss per ten thousand cycles was calculated to be directly proportional to the total weight loss for the total number of cycles. The following example is shown for the vinyl chloride copolymer varnish taken from Table IX, page 117.

$$\text{Weight loss per } 10^4 \text{ cycles} = \text{weight loss per cycle x } 10^4$$

$$= \frac{\text{total weight loss}}{\text{total cycles}} \times 10^4$$

$$= \frac{0.9 \text{ mg}}{15{,}292} \times 10^4$$

$$= 0.6 \text{ mg}$$

TABLE XII

Densities of Dry Coatings Calculated for Determining Film Thickness

Film Former	Density of Coating with Indicated Pigment					
	none	TiO_2	ZnO	$PbCO_3$	Al	C
	gm/cc	gm/cc	gm/cc	gm/cc	gm/cc	gm/cc
Vinyl Chloride Copolymer	1.31	2.43	2.97	3.18		
Spar Varnish	0.97	2.22	2.77	2.97	1.02	1.19
Alkyd Resin	0.960	2.26	2.80	2.99		

IV. DISCUSSION

The discussion is presented in three sections; (1) Discussion of results, (2) recommendations, and (3) limitations.

Discussion of Results

The results of this investigation are discussed in the same order as the data and results were presented in the preceding section.

Light Reflectivity. An observation of the light reflectance tests, Figures 4 to 35, pages 60 to 91, show that a definite change has taken place in some of the irradiated coatings. The magnitude of these changes as compared with the change of the corresponding non-irradiated coating is such that the irradiation was undoubtedly responsible for the change.

Several interesting points may be noted about the tests on the individual coatings. These are discussed in the following sections under the film former series to which the coating belonged.

Vinyl Chloride Copolymer Series. The reflectance tests for this series are shown in Figures 4 to 15, pages 60 to 71. All of the irradiated panels in this series showed a definite change in light reflectance. When compared with the non-irradiated films, a slight color change could be readily detected by eye in the clear and lead carbonate pigmented films.

Figures 11, 12, 14, and 15, tests for the films pigmented with zinc oxide and lead carbonate, show the change in reflectance was about the same for the coatings irradiated in air and in argon. Figures 5, 6, 8, and 9, indicate a greater change in the light reflectance of the clear and titanium dioxide pigmented films irradiated in argon than those irradiated in air. These tests would give reason to believe that the saran wrap around the panels did not completely isolate them from the atmosphere in which they were irradiated.

The reflectivity of the clear film may not have been changed as much as was indicated by the test. The polished surface of the tin substrate was badly tarnished or etched when the panels were returned from irradiation. This would undoubtedly affect the reflectance of the clear film. The tin was probably tarnished by the release of hydrogen chloride from the vinyl chloride resin by the radiation. This reaction has been reported by Sisman and Bopp.(38) All the irradiated panels in the vinyl chloride copolymer series were tarnished while the panels in the other series were not.

The light reflectance of the lead carbonate pigmented film probably changed the most if the effect of the tarnished panel is taken into account for the clear film. This would be expected from the discussion of the mass absorption coefficient on page eight. Here, it was pointed out that the linear absorption coefficient divided by the density was approximately constant for most materials. Reference to Table XII, page 125, shows the density of the lead carbonate pigmented film is greater than the others, therefore, its absorption coefficient should be greater and more gamma rays should be absorbed in the film.

An interesting point that may be noticed in this and the following series is the maximum and minimum in the reflectance curve for the irradiated lead carbonate pigmented films. These appear in the reflectance tests for light with wavelengths of 600 to 650 millimicrons.

Spar Varnish Series. The reflectance tests for this series are shown in Figures 16 to 27, pages 72 to 83. A slight color change was noticeable by eye in the films pigmented with titanium dioxide, zinc oxide, and lead carbonate when compared with the non-irradiated films. The reflectance tests show these were the only films for which there was a significant change in the reflectance. A very slight change was indicated for the clear and aluminum pigmented film, but no change at all was indicated for the film pigmented with lampblack.

The lead carbonate and titanium dioxide pigmented films appear to have the largest change in reflectance due to irradiation.

Figure 20 shows that the reflectance of the non-irradiated zinc oxide pigmented film changed significantly upon aging.

A slight maximum and minimum may be noted in the lead carbonate pigmented film in this series at the previously mentioned wavelength.

Alkyd Resin Series. The reflectance tests for this series are shown in Figures 28 to 35, pages 84 to 91.

Figure 35 shows that the coating pigmented with lead carbonate is the only one that suffered much of a change in reflectance. The color change in this panel was detectable by eye when compared to a non-irradiated panel.

The other figures show the clear, titanium dioxide pigmented, and zinc oxide pigmented films had small changes of the reflectance in certain wavelength ranges.

The maximum and minimum may again be noted in the reflectance curve for the film pigmented with lead carbonate.

The reflectance tests have shown that the gamma radiation caused changes in some of the coatings which could be detected by the change in the light reflectance of the coating. However, this does not necessarily mean the coatings which did not show a reflectance change was not affected by the radiation. This will be shown by later tests.

Alkali Resistance. The overall results of the alkali tests are shown in Table VI, page 93. It may be noticed that the alkali tests gave positive results in the spar varnish and alkyd resin series.

It was this group in which some of the reflectance tests failed to show any effect of the radiation. The results of the alkali tests on each of the three coating series are discussed separately.

Vinyl Chloride Copolymer Series. This series of coatings showed the best overall resistance to alkali attack. It may be seen from Table VI, page 93, that both the irradiated and non-irradiated films pigmented with titanium dioxide and zinc oxide showed no effect of alkali attack during the eight-hour tests. The table also shows that discolored spots were the only effect of the alkali on the clear and lead carbonate pigmented films.

The discoloration developed on the irradiated clear coatings during the test was very slight. Figure 36, page 95, shows that the discoloration was developed and reached its maximum intensity during the fourth hour of the test. No difference was noted in the alkali resistance of the coatings which were irradiated in air and in argon.

The discolorations were developed at different times on the irradiated and non-irradiated lead carbonate pigmented coatings. This is shown by Figure 37, page 96. In this case, the irradiated coatings were attacked first, with the coating irradiated in air showing the least resistance.

Spar Varnish Series. It may be seen from Table VI, that all of the coatings in the spar series failed during the alkali test. Discoloration of the films was usually a precursor of the failure. The relative effects of the alkali on the irradiated and non-irradiated coatings are shown in Figures 38 to 43, pages 97 to 102. These figures show that the alkali resistance of the coatings was increased by the irradiation in every case. The effect of the alkali on the coatings was usually the same, but the non-irradiated coatings always failed first.

The increase of the alkali resistance was more pronounced in the clear, titanium pigmented, and lampblack pigmented coatings. Figures 38, 39, and 43 show that the initial rate of the alkali effect on these coatings was decreased.

Alkyd Resin Series. Table VI, page 93, shows the alkali test caused discoloration and failure of the alkyd resin films. Discoloration was usually a forerunner to failure. Figures 44 to 47, pages 103 to 106, show the relative effect of the alkali on the irradiated and non-irradiated coatings.

No difference was noted in the alkali resistance of the clear coatings. Both the irradiated and non-irradiated films failed through to the metal substrate in a very short time.

A very striking difference was noted in the alkali resistance of the pigmented coatings. The rates of the alkali attack on the irradiated coatings were much lower than on the non-irradiated coatings. The effects of the alkali on the coatings at the end of the test were also less.

Acid Resistance. The overall results of the acid test are shown in Table VII, page 107. It may be seen that none of the vinyl chloride copolymer coatings were affected by the tests. The results of the acid tests on the spar varnish and alkyd resin series are discussed separately in the following sections.

Spar Varnish Series. Table VII shows that the films pigmented with lead carbonate and lampblack gave no reaction with the acid. The other films were discolored slightly by the acid, but did not fail.

Figures 48 to 51, pages 108 to 111, show the relative effect of the acid on the irradiated and non-irradiated films that were affected by the acid. The general effect of the irradiation was to decrease the acid resistance.

Figure 48 shows that the initial time for the acid to react with the clear spar coating was decreased by the irradiation. Figures 49, 50, and 51 indicate that the rate of the acid effect on the films pigmented with titanium dioxide, zinc oxide, and aluminum paste was increased by the irradiation. However, the film pigmented with zinc oxide was the only one in which the effect of the acid at the end of the test was greater for the irradiated panel.

Alkyd Resin Series. The films in this series pigmented with lead carbonate were not affected by the acid. The other films were slightly discolored, but only the clear film showed any difference in the effect on the irradiated and non-irradiated film. The rate of the effect was increased for the irradiated film.

The relative effect of the acid on the coatings in this series may be seen in Figures 52 to 54, pages 112 to 114.

Flexibility. The flexibility tests were not very useful in making a comparison between the irradiated and non-irradiated panels. Positive results were obtained for only four coatings in the entire group.

Table VIII, page 115, shows the results of the flexibility tests. The coatings that gave positive results were: (1) The vinyl chloride copolymer varnish, (2) the vinyl chloride copolymer enamel pigmented with titanium dioxide, (3) the spar varnish pigmented with lampblack, and (4) the alkyd resin pigmented with zinc oxide. In each case, the flexibility of the irradiated film was less than that of the corresponding non-irradiated film. The decrease in flexibility would suggest that the irradiated film had become more brittle. The increase in brittleness would be associated with increased crosslinking in the film. It may also be noted that the different atmospheres made no apparent difference in the flexibility of the irradiated films.

The remainder of the coatings did not fail when tested with the smallest (one-eighth inch) mandrel. This indicated they all had good flexibility, but gave no indication of a difference in flexibility of the irradiated and non-irradiated films.

Abrasion Resistance. The results of the abrasion tests are shown in Table IX, page 117.

High abrasion resistance is generally associated with flexible, rubber-like films and low abrasion resistance with hard, brittle films. The results of this test should generally substantiate the results of a flexibility test. This was found to be true in this investigation for the cases where the flexibility tests gave positive results. However, the abrasion tests indicated differences in the flexibilities of some of the coatings that were not indicated by the flexibility test.

The results of the individual abrasion tests are grouped according to the film former series for discussion.

> Vinyl Chloride Copolymer Series. A very noticeable decrease in abrasion resistance was shown by the clear and titanium dioxide pigmented films that were irradiated. This would indicate that the irradiation caused the films to become harder or more brittle. This was also indicated by the flexibility tests.
>
> Very little change in abrasion resistance was shown by the film pigmented with zinc oxide. The lead carbonate pigmented film showed a distinct increase in abrasion resistance. This apparent decrease in hardness was not detected by the flexibility test.

No marked difference was noted in the abrasion resistances of the panels irradiated in the different atmospheres.

The film pigmented with titanium dioxide had the highest abrasion resistance of the series.

Spar Varnish Series. The irradiated films pigmented with titanium dioxide and lampblack showed the largest decrease in abrasion resistance in this series. (The film pigmented with lampblack showed a decrease in flexibility in the preceding test.) The clear and lead carbonate pigmented films showed a slight decrease in abrasion resistance.

The films pigmented with aluminum paste and zinc oxide showed a slight increase in abrasion resistance. These films had the best overall abrasion resistance in the series.

Alkyd Resin Series. All of the irradiated films in this series showed a decrease in abrasion resistance. The decrease was slight in the films pigmented with titanium dioxide and lead carbonate, but more pronounced in the clear and zinc oxide pigmented films.

The clear film had the best overall abrasion resistance, even though its resistance was decreased by the irradiation.

The abrasion resistance of the three series may be compared by using Table IX, page 117. It shows that the spar varnish series ha the best overall resistance. The initial resistances were high, an they were not changed much by the irradiation.

The vinyl chloride copolymer series contained two coatings (c and pigmented with titanium dioxide) that had high initial abrasic resistances, but they were decreased considerably by the irradiati

The alkyd resin series, as a group, had low initial abrasion resistance, and they were decreased even more by the irradiation.

Scratch Hardness. The results of the scratch hardness are sh in Table X, page 118.

The tests on the individual coatings were very difficult to e uate, and probably should be used only as a comparison between the series rather than between the individual coating.

Considering the load and effect, the spar varnish series had best scratch resistance. There was not much difference in the scr resistance of the alkyd resin and vinyl chloride copolymer series, the alkyd resin series appeared slightly the better.

Table X indicates a general decrease in the scratch resistanc of the coatings after irradiation.

Impact Resistance. The results of the impact tests are shown in Table XI, page 119. The results are grouped according to film former series for discussion.

Vinyl Chloride Copolymer Series. The impact resistance of all the pigmented coatings in this series was decreased considerably by the irradiation. The clear film had very poor impact resistance initially, and it was decreased even further by the irradiation.

The adhesion of the films was decreased, except for the coating pigmented with lead carbonate. The adhesion of the clear film was affected the most.

The atmosphere during the irradiation seemed to have a very slight, if any, effect on the impact resistance and adhesion.

Spar Varnish Series. The impact resistance of this series was very good. The initial impact resistance of the clear, titanium dioxide pigmented, and lead carbonate pigmented films was high, and was not decreased by irradiation to a point where it could be measured. The initial impact resistance of the film pigmented with zinc oxide compared favorably with the corresponding film of the vinyl chloride copolymer series. The resistance of this film was increased by the irradiation.

The adhesion of the coatings to the panels was not affected by the radiation enough to be noticeable.

The impact resistance of the films pigmented with aluminum paste and lampblack was lower than the other coatings, but was not affected by the radiation.

Alkyd Resin Series. Table XI, page 119, shows all the alkyd resin coatings had good impact resistance except the one pigmented with zinc oxide. This coating was the only one in which the adhesion was affected by the irradiation. The impact resistances of the alkyd coatings were not affected by the irradiation to an extent that was detectable with the impact tester.

Recommendations

There are numerous ways in which this investigation could be extended. The first way would be to study the effect of different radiation doses on the coatings. Coatings are undergoing constant changes when they are radiated. A study confined to a constant radiation dose, such as this one, will reveal only the effect present at that dose. Totally different effects might be found for larger or smaller doses.

Another possible extension would be to study coatings that have been given constant doses of radiation at different rates. It is possible that rate of irradiation would be a determining factor in the effect on the coating.

A third extension might be to test mixtures of film formers or pigments to see if desirable qualities of the different components could be incorporated into one film.

It would also be an item of interest to check the irradiated coatings pigmented with lead carbonate to determine if the pigment was changed by the irradiation. A change in the pigment was indicated in the reflectance curves.

Many variations of this study could be made that would be useful in specifying coatings for use in radioactive areas.

Limitations

There were four limitations in this investigation that could have an important bearing on the results. They are as follows:

Control of Film Thickness. The panels for this investigation were coated by brushing. It was the only means available. There may have been enough variation in film thickness to give different results in the tests.

Rate of Irradiation. An observation of Table IV, page 49, will show that the groups of panels were irradiated at different rates. This could have made a difference in the effect of the radiation on the coatings. The variation was caused by the radioactive decay of the gamma-ray source and could not be kept constant.

Time Delay Between Coating, Irradiation, and Testing. Newly applied protective coatings are usually in a changing state due to curing and may exhibit different properties at different stages of the curing. The irradiation and testing should have been done at specified times after the coating was applied. This was not possible due to varying lengths of time for transportation to and from the irradiation facility, the irradiation schedule set up by the facility, and the varying time needed to test the panels when they were returned.

Control of Relative Humidity. Some of the performance tests that were made on the coatings are very sensitive to the relative humidity of the atmosphere when they are tested. No facilities were available with which to control this factor.

V. CONCLUSIONS

Protective coatings for this investigation were made with three film formers, (1) a vinyl chloride copolymer resin, (2) a spar varnish, and (3) an alkyd resin. Each film former was used to make a clear varnish, and to make enamels pigmented with (1) titanium dioxide, (2) zinc oxide, and (3) lead carbonate. Enamels pigmented with aluminum paste and lampblack were also made with the spar varnish. Test panels were prepared with these coatings and irradiated with gamma rays whose average energy was about 0.75 Mev. The dosage received by the panels was eight million rads. Tests were performed on the coated panels. A comparison of the test results from the irradiated and non-irradiated test panels led to the following conclusions:

A. Vinyl chloride copolymer coatings.

1. Light reflectance (400 to 700 millimicron wavelength).
    - a. The light reflectance of the pigmented and non-pigmented coatings was decreased by the irradiation.
    - b. The clear and lead carbonate pigmented films experienced the greatest decrease in reflectance by the irradiation.
2. Alkali resistance (5% NaOH).
    - a. The non-irradiated coatings had good alkali resistance.

   b. The initial rate of alkali attack on the coating pigmented with lead carbonate was increased by the irradiation.
   c. The alkali resistance of the clear, titanium dioxide pigmented, and zinc oxide pigmented coatings was not appreciably affected by the irradiation.

3. Acid resistance (12 N HCl).
   a. The non-irradiated coatings were not affected by an eight-hour exposure to the acid.
   b. The irradiated coatings were not affected by an eight-hour exposure to the acid.
4. Flexibility.
   a. The irradiated and non-irradiated coatings pigmented with zinc oxide and lead carbonate did not fail when bent over a one-eighth inch mandrel.
   b. The flexibility of the clear and titanium dioxide pigmented coatings was decreased by the irradiation.
5. Abrasion resistance.
   a. The abrasion resistance of the clear and titanium dioxide pigmented coatings was decreased by the irradiation.
   b. The abrasion resistance of the coating pigmented with zinc oxide was not affected by the irradiation.
   c. The abrasion resistance of the coating pigmented with lead carbonate was increased by the irradiation.

6. Scratch hardness.
    a. The scratch hardness of the clear and lead carbonate pigmented coatings was decreased by the irradiation.
    b. The scratch hardness of the coatings pigmented with titanium dioxide and zinc oxide was not appreciably affected by the irradiation.
7. Impact resistance and adhesion.
    a. The impact resistance of the pigmented and non-pigmented coatings was decreased by the irradiation.
    b. The adhesion of the clear, titanium dioxide pigmented, and zinc oxide pigmented coatings was decreased by the irradiation.
    c. The adhesion of the coating pigmented with lead carbonate was not appreciably affected by the irradiatior

B. Spar varnish coatings.

1. Light reflectance (400 to 700 millimicron wavelength).
    a. The light reflectance of the coating pigmented with lampblack was not changed by the irradiation.
    b. The light reflectance of the clear and aluminum pigmented coatings was decreased slightly by the irradiation.
    c. The irradiated coatings pigmented with titanium dioxide, zinc oxide, and lead carbonate experienced a definite change in light reflectance.

2. Alkali resistance (5% NaOH).
    a. The non-irradiated coatings had poor alkali resistance.
    b. The alkali resistance of the coatings was slightly improved by the irradiation.
3. Acid resistance (12 N HCl).
    a. The irradiated and non-irradiated coatings pigmented with lead carbonate and lampblack were not affected by a three-hour exposure to the acid.
    b. The acid resistance of the clear, titanium dioxide pigmented, zinc oxide pigmented, and aluminum paste pigmented coatings was decreased by the irradiation.
4. Flexibility.
    a. The irradiated and non-irradiated clear coating did not fail when bent over a one-eighth inch mandrel.
    b. The irradiated and non-irradiated coatings pigmented with titanium dioxide, zinc oxide, lead carbonate, and aluminum paste did not fail when bent over a one-eighth inch mandrel.
    c. The flexibility of the coating pigmented with lampblack was decreased by the irradiation.
5. Abrasion resistance.
    a. The abrasion resistance of the clear, titanium dioxide pigmented, and lampblack pigmented coatings was significantly decreased by the irradiation.

   b. The abrasion resistance of the coating pigmented with lead carbonate was slightly decreased by the irradiation.

   c. The irradiation increased the abrasion resistance of the coatings pigmented with zinc oxide and aluminum paste.

6. Scratch hardness.

   a. The scratch hardness of the clear, titanium dioxide pigmented, and zinc oxide pigmented coatings was decreased by the irradiation.

   b. The scratch hardness of the coatings pigmented with lead carbonate, aluminum paste, and lampblack was not appreciably affected by the irradiation.

7. Impact resistance.

   a. The irradiated and non-irradiated clear coatings withstood an impact of thirty inch-pounds without failure.

   b. The irradiated and non-irradiated coatings pigmented with titanium dioxide and lead carbonate withstood an impact of thirty inch-pounds without failure.

   c. The impact resistance of the coatings pigmented with aluminum paste and lampblack was not affected by the irradiation.

   d. The impact resistance of the coating pigmented with zinc oxide was increased by the irradiation.

    e. The adhesion of the coatings was not appreciably affected by the irradiation.

C. Alkyd resin coatings.

1. Light reflectance (400 to 700 millimicron wavelength).
    a. The light reflectance of the clear, titanium dioxide pigmented, and zinc oxide pigmented coatings was very slightly changed, if affected at all, by the irradiation.
    b. The light reflectance of the coating pigmented with lead carbonate was definitely decreased by the irradiation.
2. Alkali resistance (5% NaOH).
    a. The non-irradiated coatings had very poor alkali resistance.
    b. The non-irradiated pigmented coatings had superior alkali resistance to the clear coating.
    c. The alkali resistance of the clear coating was not apparently affected by the irradiation.
    d. The alkali resistance of the pigmented coatings showed a marked increase after irradiation.
3. Acid resistance (12 N HCl).
    a. The irradiated and non-irradiated coatings pigmented with lead carbonate were not affected by a two and one-half hour exposure to the acid.

   b. The acid resistance of the clear coating was decrease by the irradiation.
   c. The acid resistance of the coatings pigmented with titanium dioxide and zinc oxide were not affected by the irradiation.

4. Flexibility.
   a. The irradiated and non-irradiated clear coatings did not fail when bent over a one-eighth inch mandrel.
   b. The irradiated and non-irradiated coatings pigmented with titanium dioxide and lead carbonate did not fail when bent over a one-eighth inch mandrel.
   c. The flexibility of the coating pigmented with zinc oxide was decreased by the irradiation.
5. Abrasion resistance.
   a. The abrasion resistance of the clear and zinc oxide pigmented coatings was significantly decreased by the irradiation.
   b. The abrasion resistance of the coatings pigmented with titanium dioxide and lead carbonate was slightly decreased by the irradiation.
6. Scratch hardness.
   a. The scratch hardness of the clear, titanium dioxide pigmented, and zinc oxide pigmented coatings was decreased by the irradiation.

   b. The scratch hardness of the coating pigmented with lead carbonate was not affected by the irradiation.

7. Impact resistance and adhesion.
   a. The irradiated and non-irradiated clear, titanium dioxide pigmented, and lead carbonate pigmented coatings withstood an impact of thirty inch-pounds without failure.
   b. The irradiated and non-irradiated coatings pigmented with zinc oxide failed for an impact of one inch-pound.

A few general conclusions for the coating series may be drawn from the preceding itemized conclusions. They are as follows:

A. The alkali and acid resistance of the vinyl chloride copolymer coatings was superior to either the spar or alkyd resin coatings.

B. The vinyl chloride copolymer coatings retained their superior alkali and acid resistance after receiving an eight million rad dose of gamma radiation.

C. The impact resistance and adhesion of the spar and alkyd resin coatings (with the exception of the coating pigmented with zinc oxide) were superior to the vinyl chloride copolymer coatings.

D. The spar and alkyd resin coatings retained their superior impact resistance and adhesion after receiving an eight million rad dose of gamma radiation.

E. The following items were noted about the various coatings pigmented with lead carbonate. It was the only pigment used in this investigation that showed consistant behavior throughout a film former series.

   1. The irradiated coatings pigmented with lead carbonate exhibited a greater change in light reflectance than the clear, titanium dioxide pigmented, or zinc oxide pigmented coatings.
   2. The lead carbonate pigment improved the alkali and acid resistance of the irradiated and non-irradiated spar and alkyd resin coatings.
   3. The lead carbonate pigment decreased the alkali resistance of the irradiated and non-irradiated vinyl chloride copolymer coatings.

VI. SUMMARY

The purpose of this investigation was to prepare pigmented and non-pigmented organic protective coatings and to irradiate them with gamma rays. The irradiation dose received by the coatings was eight million rads. Tests were performed on the irradiated coatings and on similar non-irradiated coatings. The results of the tests were tabulated so comparisons could be made to determine the effect of the radiation on the coatings.

The film formers used in the coatings were: (1) A vinyl chloride copolymer resin, (2) a phenolic resin modified with tung and linseed oil, and (3) an alkyd resin modified with soya oil. The modified phenolic resin was obtained as a spar varnish.

The coatings prepared with the film formers were varnishes and enamels. The enamels were pigmented with a single pigment. The pigments used were: (1) Titanium dioxide, (2) zinc oxide, and (3) lead carbonate. Aluminum paste and lampblack were also included as pigments with the spar varnish film former.

The tests performed on the irradiated and non-irradiated coatings were: (1) Light reflectance before and after irradiation, (2) alkali resistance, (3) acid resistance, (4) flexibility, (5) abrasion resistance, (6) scratch hardness, and (7) impact resistance.

The investigation led to the following conclusions about the coatings:

1. The vinyl chloride copolymer coatings had superior alkali and acid resistance to either the spar or alkyd resin coatings and retained the superior resistance after receiving an eight million rad dose of gamma radiation.
2. The alkali resistance of the vinyl chloride copolymer coatings tended to be slightly decreased by the irradiation. It was slightly increased for the spar and alkyd resin coatings.
3. The acid resistance of the vinyl chloride copolymer coatings was not affected by the irradiation, but it was slightly lowered in the spar and alkyd resin coatings.
4. The spar and alkyd resin coatings (with the exception of the coating pigmented with zinc oxide) had superior impact resistance and adhesion to the vinyl chloride copolymer coatings. They retained this superior impact resistance and adhesion after receiving an eight million rad dose of gamma radiation.
5. The impact resistance and adhesion of the vinyl chloride copolymer coatings were drastically reduced by the irradiation. They were not measurably affected in the spar and alkyd resin coatings.

The lead carbonate was the only pigment that was observed to show a consistant behavior in the coating series. The following ite were noted:

1. The irradiated coatings pigmented with lead carbonate consistantly exhibited a large change in light reflectance. The change was much greater than in the other coatings.
2. The lead carbonate pigment improved the alkali and acid resistance of the irradiated and non-irradiated spar and alkyd resin coatings.
3. The lead carbonate pigment decreased the alkali resistance of the irradiated and non-irradiated vinyl chloride copolymer coatings.

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IX. VITA

James Winston Johnson, son of Fred M. and Lois Johnson, was born May 25, 1930, in Quinton, Oklahoma.

He attended public school at Clayton, Oklahoma, and graduated from high school in May, 1947.

He attended Southeastern State College, Durant, Oklahoma, during the school year of 1947-48. Following this, he worked for the Oklahoma State Highway Department as a rodman in a highway construction surveying crew.

In 1950, he returned to college, and in May, 1953, received the degree of Bachelor of Science in Education with majors in mathematics and chemistry from Southeastern State College.

He was inducted into the United States Army for two years on July 13, 1953. At the time of his release, his principle duty was Operations Sergeant with the 333rd Engineer Pipeline Company at Fort Leonard Wood, Missouri.

On October 10, 1953, he was married to Vera Mae Hamman of Nashoba, Oklahoma.

He enrolled at the Missouri School of Mines and Metallurgy, in September, 1955, and received the degree of Bachelor of Science in Chemical Engineering in June, 1957.

He was employed by the Production Department, Shell Oil Company, during the summers of 1956 and 1957.

In September, 1957, he entered the Graduate School at the Missouri School of Mines and Metallurgy, where he began a course of study under a one-year fellowship in Nuclear Technology granted by the United States Atomic Energy Commission.

James R. Johnson