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REPORT ON RAIN REPELLENT COATINGS

By
O. Davis Shreve

Report No. P-2319

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NAVY DEPARTMENT

Report on

Rain Repellent Coatings

Naval Research Laboratory
Anacostia Station
Washington, D.C.

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ABSTRACT

Due to the hydrophilic nature of a glass (or plexiglas) surface water spreads in a sheet on the windshield of an airplane flying under rainstorm conditions. Non-uniformity of light refraction through this sheet results in distortion of vision.

The theory underlying the distinction between wettable and non-wettable substances is discussed, and it is shown that:

(a) elimination of this distortion involves the transformation of the hydrophilic glass (or plexiglas) surface to a hydrophobic, or water repellent, surface and;

(b) application of a thin transparent water repellent coating offers the most practical means of effecting this transformation. Water striking a windshield thus treated is resolved into discrete droplets which are immediately removed by the windstream and distortion of vision is eliminated or greatly diminished.

Consideration of the few known facts bearing on the problem, together with the chief properties required of such a coating, point to a lacquer type of mixture as the most promising.

The history of the development of a lacquer type repellent (N.R.L. No. 199) is outlined with composition and test data for 199 mixtures. Various test methods and apparatus designed during the course of the investigation are described.

Photographs and laboratory and service test data indicate the merit of various repellents and establish the superiority of N.R.L. No. 199.

 [redacted]

AUTHORIZATION

1. This study was authorized by BuAer Project No. 176/44.

STATEMENT OF PROBLEM

2. A quantity of a rain repellent preparation for improving visibility through airplane windshields was purchased from Lorr Laboratories, Inc., under experimental contract with the Navy. As a result of this purchase, the Bureau of Aeronautics requested that (a) this laboratory develop specifications for a rain repellent material similar to the Lorr product and (b) that test methods be devised for determining the comparative efficacy of such preparations in improving vision through windshields under rainstorm conditions.

THEORETICAL CONSIDERATIONS

3. Suppose a liquid L (Fig. 1, Plate 9) rests on a solid surface S and comes to equilibrium with the "angle of contact" θ, which is measured in the liquid phase. G is the gas, usually air, in contact with liquid and solid. At equilibrium the forces acting at the interfaces must balance and, if these forces can be represented by surface tensions acting in the direction of the surfaces, it follows that:

$$\gamma_{gs} = \gamma_{ls} + \gamma_{gl} \cos \theta \qquad \text{(Eq. 1)}$$

where γ_{gs}, γ_{ls} and γ_{gl} are the surface tensions at the gas-solid, liquid-solid and gas-liquid interfaces respectively.

4. The contact angle, θ, thus depends on the three interfacial tensions but whether it is greater or less than 90^o depends on the relative magnitudes of γ_{gs} and γ_{ls}. If the gas-solid tension (γ_{gs}), is greater than the liquid-solid tension (γ_{ls}), cos θ is positive and $\theta < 90^o$; if the reverse is true, then θ lies between 90^o and 180^o. In the former instance, e.g., water on glass, the liquid is regarded as wetting the solid. In the latter case, e.g., water on paraffin, the liquid is said not to wet the solid. The difference is a matter of degree but it is conventional to say that wetting occurs when the contact angle is below 90^o and no wetting if it is greater than 90^o.

5. Since the contact angle for water on glass is low (ca. 30^o), water spreads on such a surface. Under non-ideal conditions the distribution of the liquid over the surface is not uniform, with the result that the degree of refraction of light passing through the water layer varies from point to point. This variation causes distortion of vision when the objects from which the light was reflected are viewed through the glass-water combination. In the case of the windshield of an airplane in flight under rainstorm conditions the sheet of water on the glass is distorted by the air stream thus adding to the distortion of vision.

6. Consideration of the theory indicates two possible approaches to the problem of minimizing or eliminating this distortion.

7. From equation (1) it is seen that a decrease in γ_{ls} (surface tension at the water-glass interface) will result in a decreased contact angle, while an increase in this quantity will increase the contact angle between the two phases.

 [redacted]

A decrease of surface tension (and thus contact angle) to the point of perfect wetting should theoretically produce uniform spreading and thus eliminate distortion. Such a decrease in the surface tension of water can be achieved through the use of wetting agents (compounds containing long carbon chains and hydrophilic groups.) Such materials, however, being water soluble cannot be durable nor can such a method eliminate the air stream distortion of the water sheet. Wetting agents are therefore eliminated from consideration.

8. The alternative approach based on methods of increasing the surface tension seems to offer greater possibilities. As pointed out above, a surface on which the contact angle of a liquid exceeds 90° is not wet by the liquid. If a stream of water strikes an inclined surface on which its contact angle exceeds 90°, the water will not spread in a continuous sheet but will be resolved into discrete droplets and these droplets will roll freely down the inclined surface.

9. A consideration of wetting and contact angle from the point of view of work of adhesion and cohesion may be useful at this point. If the surface between L and S (Fig. 1) is diminished by 1 sq. cm. the interfaces between G and S and between G and L are both increased by 1 sq. cm. Since surface tension may be defined as the work done in extending the surface by 1 sq. cm., the resultant work done is $\gamma_{gs} + \gamma_{gl} - \gamma_{ls}$. This work is a measure of the energy required to separate the liquid from the solid and is called the work of adhesion (W_{ls}), thus:

$$W_{ls} = \gamma_{gs} + \gamma_{gl} - \gamma_{ls} \qquad \text{(Eq. 2)}$$

10. If the liquid is imagined to be in the form of a column 1 sq. cm. in cross section and the two ends are pulled so that the liquid is divided into two parts, then two new liquid gas surfaces each of 1 sq. cm. area will be formed. The work required is $2\gamma_{gl}$ and since the work must be done against the cohesive forces within the liquid, it is called the work of cohesion of the liquid. If (1) and (2) are combined we obtain:

$$W_{ls} = \gamma_{gl} \; (1 + \cos \theta) \qquad \text{(Eq. 3)}$$

11. From equation (3) it is seen that the contact angle increases as the ratio of work of cohesion to work of adhesion increases. In other words, if the attraction between molecules of the liquid themselves greatly exceeds that between molecules of solid and liquid, then the contact angle will be large, and the surface of liquid-solid contact will be small. As a result droplets will roll on the surface very easily and will be removed immediately if a stream of air plays on the surface as in the case of the windshield of an airplane in flight.

12. The problem then of improving visibility through an airplane windshield under rain conditions becomes a problem of transforming the hydrophilic glass surface into a hydrophobic or water repelling surface.

13. If this surface is to be realized by means of a coating on the glass, it must meet certain requirements in addition to water repellency. It must be transparent, durable and adhesive to glass. It should be easily applied and easily removed unless it is to be permanent. Its nature should be such that the water striking it is resolved into very <u>small</u> droplets since the motion of large droplets causes eye fatigue.

 [redacted]

14. When we consider what types of materials are most likely to present surfaces highly repellent to water, the high molecular weight, water insoluble organic solids are immediately indicated. Nietz (2) measured contact angles and calculated works of adhesion for a number of organic compounds. His data are reproduced in Table I.

15. This data shows that within any given chemical class there is a wide variation in contact angle and work of adhesion and that there is no tendency for any class to assume a particular value. This indicates that factors other than the mere presence of a given functional group influence the degree of water repellence exhibited by a substance. Probably the most important of these is the molecular orientation in the surface. Nietz made measurements on several samples of stearic acid crystallized under different conditions and obtained widely varying values for the contact angle and work of adhesion. Thus a knowledge of the chemical structure must be supplemented by information on molecular orientation in the surface if a prediction as to the water repellent properties of a compound is to be made. In view of the absence of sufficient data on which to base such predictions it seems best to select for experimentation those solid compounds whose contact angles have been measured and found to exceed 90°. Twenty such compounds appear in Table I and are indicated by asterisks.

16. Once a water repellent compound has been selected for trial, attention must be directed to methods of securing on glass a thin coating of the compound or a coating of which it is a constituent. If the compound alone does not possess, in addition to water repellence, the other requisite characteristics indicated in paragraph 13 above, there remains the possibility of mixing it with other materials capable of contributing these required properties to the final film.

17. Three possible methods of obtaining water repellent films on glass will be considered:

(a) The water repellent compound along with a film forming constituent and an agent designed to impart adhesion to glass may be dissolved in a volatile solvent. This is a lacquer type of preparation and evaporation of the solvent will leave a thin uniform film of the dissolved material on the glass surface. The "film-forming" and "adhesion-imparting" materials will be found among the natural and synthetic resins which are usually esters of high molecular weight organic acids. They contain no functional groups and many have good water resistant properties.

(b) The repellent compound together with any other necessary substances may be incorporated into a paste and applied by a rubbing and polishing technique.

(c) The glass may be exposed to the vapors of a member of a special class of compounds which hydrolyze to produce solid water repellent products integrally combined with the glass surface.

18. The lacquer type of coating requires a drying period and must be applied in a dry dust-free environment. Acutal application however is simple and high reproducibility of coatings is obtainable. Also the great majority of materials possessing the properties required in a rain repellent coating are organic compounds readily soluble in the common organic solvents and thus suitable for incorporation in a lacquer type of preparation.

[redacted]

19. The paste type is easiest and simplets to apply and can be applied under rain conditions but since the films must be very thin and uniform, the rubbing and polishing involved makes it very difficult to get an effective coating.

20. Production of an integrally combined repellent surface can be accomplished through the application of volatile chlorosilicomethane compounds which hydrolyze and produce a film of polymerized hydroxy compounds with water repellent properties. Trimethylmonochlorsilicomethane reacts as follows:

$$(CH_3)_3\ SiCl + H_2O \rightarrow (CH_3)_3\ SiOH + HCl$$

This type of coating has possibilities but requires a specialized application technique. (Some compounds of this type may be applied in a solvent and thus may be considered similar to the lacquer type.)

KNOWN FACTS BEARING ON THE PROBLEM

21. Most research in industry on water repellent coatings has been directed toward the problem of waterproofing fabrics and similar materials. Usual preparations developed specifically for waterproofing are ineffective in improving visibility through optical surfaces. They are designed to render materials impermeable but not non-wettable. They are in general not transparent.

22. The Lorr preparation, previously mentioned, is one of the few commercial preparations developed for the specific purpose of improving optical visibility and its use in limited quantities by the Navy gave rise to the investigations embodied in this report. It is a lacquer type of repellent consisting of a wax (to impart high contact angle), a plasticizing agent and a film forming agent (resin) dissolved in a mixture of amyl acetate and a hydrocarbon solvent. The material is applied by means of a paper squeegee and after drying 30 minutes produces a film on which water has a contact angle of 96° to 98°.

23. The National Research Council has developed a paste type repellent, to be applied by rubbing and polishing, and requiring no drying time. It consists of a mixture of paraffin and zinc stearate brought to the desired consistency by the addition of a hydrocarbon solvent.

24. The substituted methyl silico methanes have been developed by the General Electric Company for producing water repellent coatings on insulated parts of electrical equipment. Recently these coatings, (produced, by hydrolytic action, as explained under Theory above), have received some attention in connection with the improvement of visibility through airplane windshields in rainstorms.

METHODS

25. Materials: The selection of materials for experimentation was governed by the general considerations brought out in the theoretical discussion. Since a lacquer type of formulation seemed most promising, materials were selected from those chemical classes most likely to contribute one or more of the chief characteristics desired in such a mixture, viz: a high contact angle in the final film; film forming properties in the mixture; plasticizing and adhesive properties.

[redacted]

26. Of the twenty compounds in Table I possessing contact angles over 90°, seventeen are high molecular weight organic acids or esters of such acids; one (paraffin) is a mixture of high molecular weight aliphatic hydrocarbons; one (anthracene) is an aromatic hydrocarbon and one (trichlorobenzene) is a substituted aromatic. Zinc stearate is insoluble in organic solvents and is therefore eliminated. Many waxes are mixtures of these high molecular weight organic acids and their esters, and others contain aliphatic and aromatic hydrocarbons of high molecular weight. Accordingly the natural and synthetic waxes were selected as the classes most likely to yield a material capable of contributing high contact angle to the repellent film.

27. Substances to be tried as film forming agents were selected from the types commonly used for this purpose in waterproof lacquers, varnishes, etc. These include the nitrocelluloses and cellulose acetates, the acryloid resins, the glyptals, the bakelite resins and others.

28. Rosin and its condensation products with higher alcohols (ester gums) were selected as materials likely to impart adhesion as well as water resistance and hardness to the films.

29. The more commonly known materials used in the investigation were representative samples of average purity and exact specifications are not given except in the case of ingredients in the finally accepted mixture. Several of the materials used however, are less common and appear in the data under commercial names. These are listed alphabetically, together with identifying information, in Table V.

30. Several commercial rain repellents were received at the Laboratory during this investigation and all such materials are listed in Table X with pertinent comments regarding the merits of each as judged by laboratory tests.

31. <u>Description of Experiments</u>: Stock solutions of the selected waxes and resins in suitable organic solvents were prepared, and samples applied to 3" x 8" glass plates with the aid of folded "Kleenex" tissue squeegees and a small metal trough. After evaporation of the solvent the contact angle of a drop of distilled water on the solid film was measured inside the drop by means of a simple telescope arrangement. The principle of the method is illustrated diagrammatically in Fig. 2, Plate 9. The axis of the telescope is lined up with the surface of the glass plate G and the instrument focused until a magnified image D of the water drop d is obtained. The cross hair AB in the eyepiece of the telescope is adjusted until parallel to the edge MN. Cross hair CK which is attached to the indicator P is then adjusted until it forms a tangent to the curvature of the drop at its point of contact with the surface. The contact angle θ may now be read on the protractor scale which is graduated in degrees. Waxes having contact angles under 90° were eliminated from further consideration. Materials selected primarily for film forming or adhesive properties, however, were not eliminated on the basis of this test.

32. The next test applied was the water immersion test. The solutions were applied to the glass plates as described and these, after evaporation of the solvent, were immersed in water at room temperature and examined at frequent intervals to determine the general behavior of the films thus exposed. In some cases the change of contact angle with time of immersion was determined. Materials which stood up well in this immersion test were retained for further experimentation.

[redacted]

33. After the elimination of waxes with low contact angles and resinous materials with poor water resistance, the stock solutions of the more promising materials were used in the preparation of mixtures. Those mixtures usually (but not always) included at least one wax, one film-forming agent and one adhesion-contributing substance. The stock solutions of any such group selected for study were mixed in varying proportions, applied to glass and subjected to one or more of the tests listed below:

(a) Rough estimation of drying time and examination of the film for compatibility.
(b) Measurement of contact angle as described above.
(c) Exposure to water in the immersion test described above.
(d) <u>Spray Test</u>. In this test a sample of the mixture was applied to one-half of one surface of a soda glass plate one foot square and the plate mounted in the "windshield" of a special apparatus designed to simulate rainstorm conditions. In this apparatus (see Plate 1) a spray of tap water from a spray nozzle was directed at the film by means of a 3" blower placed 20" from the center of the glass plate, the latter being inclined at an angle of 30° with the normal to the windstream. This arrangement produced a windstream of 40 miles per hour at the windshield surface. Water passed through the apparatus at a rate corresponding approximately to 3.6 inches of rain per hour. Visual observation and photographs of distant objects through the coated and uncoated portions of the glass provided an excellent means of judging the effectiveness of the various films in improving visibility. Change in visibility with time of exposure, appearance of flaws in the film, clouding, size and dispersion of droplets and adhesive qualities were noted.
(e) <u>Determination of Minimum Rolling Angle</u>. Of considerable value in comparing water repellence of films is the determination of the minimum angle of inclination at which a water drop will just roll down the surface of a coated glass plate. Plate 2 shows an apparatus especially designed to carry out such measurements. The mixture under test is applied to a 3" x 8" glass plate and, after solvent evaporation, the plate is mounted in the frame of the apparatus. With the frame in a horizontal position a water drop of known size and weight is placed on the surface. The plate is then gradually inclined by means of the hand crank and gear and the angle at which the drop just begins to roll is read to the nearest degree on the graduated scale. Plate 3 shows a weatherometer device designed for use in conjunction with the rolling angle apparatus. It is similar in principle to the spray machine already described. In this apparatus a rectangular extension with a 30° inclined frame for the small glass plates at one end and a spray nozzle near the other is mounted on the 3" blower. The blower furnishes a 60 miles per hour wind and directs a spray of water at the plate, the center of which is 20" from the spray nozzle. The water passes through the apparatus at a rate corresponding to 3.6 inches of rain per hour and escapes through the drain as shown. After exposure in this device for a given time, the sample plate may be removed and its rolling angle and contact angle determined. The exposure time versus contact angle or versus rolling angle data thus obtained reflect the durability and effective life of the film.
(f) <u>Service Tests</u>. Samples of the best mixture, as judged by the above laboratory tests, were submitted to various naval aviation establishments for flight tests under rainstorm conditions.

[redacted]

DATA

34. The compositions and test data for the mixtures investigated are recorded in Tables II, III and IV. The mixtures are numbered from 1 to 199, the numerical order corresponding to the chronological order in which they were prepared and studied. Mixtures 1 to 59 inclusive and 154 through 199 inclusive, in which F-10 Acryloid resin is the film forming constituent, are listed in Table II. Mixtures 60 through 153 inclusive in which nitrocellulose is the film forming constituent are listed in Table III. Table IV gives data on miscellaneous mixtures (not numbered) which were studied from time to time but do not fit into the main sequence of the investigation. As mentioned under materials, the less commonly known substances which appear in the tables under trade names are listed alphabetically in Table V, together with descriptive information.

35. Mixture No. 199 (Table II) is the most effective film developed in this work and the remainder of the tables, figures and plates give data on this film (named N.R.L. No. 199), the Lorr material and other commercial products.

36. Table VI summarizes the composition and lists specifications for the constituents of 199 and a cleaning solvent for removing spent films. Table VII gives data on change of contact angle with time of exposure in water immersion test for No. 199, the Lorr preparation and "Anti-Rain Compound 1-A", the latter a paste type of repellent developed by the National Research Corporation. These data are plotted in Figure 3. Rolling angles as a function of drying time for No. 199 are recorded in Table VIII and are plotted in Figure 4. Table IX gives rolling angles before and after a 10 hour exposure in the weatherometer for plates coated with No. 199, Lorr and N.R.C. 1-A. Table X summarizes the good and bad points of several commercial repellents.

37. Plates 4 to 8 show photographically the effectiveness of N.R.L. No. 199 in improving visibility through the windshield of the spray machine in a 40 mile windstream and 3.6 inches rain per hour as follows:

Plate 4: Comparison of Treated Glass with Normal Glass.
Plate 5: General Vision Through Glass Windshield Coated with N.R.L. 199.
Plate 6: General Vision Through Uncoated Glass Windshield.
Plate 7: General Vision Through Plexiglas Coated with N.R.L. 199.
Plate 8: General Vision Through Uncoated Plexiglas Windshield.

38. Service test data on N.R.L. 199 are included in Appendix 2 in the form of copies of all reports thus far received and relevant to performance under actual service conditions.

DISCUSSION

39. The first 59 mixtures containing F-10 Acryloid Resin as the film forming agent (Table II) were studied before the spray test was designed. Many of these gave clear compatible films with high contact angles and good water resistance in the immersion test. Almost invariably, however, these films became fogged in the water bath, and accordingly the F-10 type was temporarily abandoned when it was found that nitrocellulose base films lacked the fogging characteristic.

 [redacted]

40. Nitrocellulose films (Table III Nos. 60 to 153), showed promise but when extensive experimentation failed to produce a film which would perform satisfactorily, one of the F-10 films was subjected to the spray test and the fogging noticed in the water bath was completely absent. Study of the F-10 type was then resumed (mixtures 154 to 199 - Table II) using the spray test as the primary criterion of performance and a satisfactory film (No. 199) was developed.*

41. The considerations which determined the course of the investigation from mixture 1 through to the final acceptable mixture, 199 (Tables II, III, IV), may be chronologically summarized.

42. Since the contact angle of water on film 1 (F-10 alone) was too low, the addition of paraffin in amounts varying from .5% to 4% solids was studied (films 2 - 6). More than 3% paraffin caused incompatibility and all these films fogged on immersion. The presence of about 75% rosin (films 7 - 10) in the F-10 paraffin mixtures, decreased the contact angle, gave powdery films, and did not impart adhesion.

43. Staybelite 10 (a glycerol ester of hydrogenated rosin) was next substituted for rosin (films 11 - 15). These films were clear and adhesion good but contact angles were considerably lower than in films 2 - 6 and they fogged on immersion.

44. A film of Vulkamine (polyisobutylene) plasticizer alone gave a contact angle of 95° which is much higher than that of rosin or staybelite 10 alone. For this reason it was next tried (films 16 and 17) as adhesive and plasticizing agent in the F-10 paraffin films. However, it was not sufficiently effective in the proportions employed.

45. Vistanex (film 18), Petrolatum (films 19 and 20), Camphor (film 21) and shellac (film 22), were next tried with F-10 but with unsatisfactory results.

46. At this point waxes other than paraffin were tried with F-10 (films 23 - 32) in an attempt to eliminate the fogging phenomenon. Films containing these new waxes still fogged but in the case of ceresin wax (film 32) and Japan wax (film 30) the fogging cleared almost immediately on removal from the bath. Japan wax being unavailable in quantity, F-10-ceresin films were next studied extensively (films 33 - 59) with the results indicated in the data.

47. On the theory that the water bath fogging might be a characteristic of the F-10, it was decided at this point that another film former should be sought. The wide use of nitrocellulose in waterproof preparations suggested this type of material as a possibility. Collodion (a lower nitrated cellulose dissolved in an ether-alcohol mixture) blushed because of rapid evaporation (film 60, Table III). By evaporating the solvent from collodion and dissolving the solid (Nitrocellulose C) in butyl acetate, a clear film (61) was obtained but it peeled on immersion. Addition of ceresin (films 62 - 64) raised the contact angle to around 100 but poly-pale resin and Flexalyn (films 65 - 69) proved unsatisfactory as adhesives.

*NOTE: Spray data recorded in Table II for F-10 films below No. 59 were obtained by remixing and testing after the spray test was designed.

40% staybelite 10 ester, however, did impart adhesion (film 70)* without serious lowering of contact angle and the film did not fog in the immersion test as did the F-10 films.

48. The effect of varying staybelite concentration in nitrocellulose C - staybelite - paraffin films was next studied (films 74 - 81).** Since 40% staybelite did not impart adhesion to nitrocellulose (½ and ¼ sec.), as shown by films 71 and 72, the effect of increasing staybelite to 75% on films containing these was tried. The increased staybelite imparted adhesion but resulted in a considerable lowering of the contact angle (films 82 and 83).

49. As already observed Vulkamine has a high contact angle and is used as a plasticizer. It accomplished nothing in the F-10 films but gave promising behavior with nitrocellulose. Film 88 containing only nitrocellulose and Vulkamine had a contact angle of 95°, the highest obtained in a film not containing a wax. It peeled on immersion but the addition of staybelite 10 imparted the necessary adhesion (film 91).

50. Beginning with No. 94 the remainder of the mixtures in Table III (94 - 153) contain only those three constituents, i.e., nitrocellulose as film former, Vulkamine for contact angle and plasticizing and staybelite 10 for adhesion, all in butyl acetate as solvent. The proportions were varied over a wide range in an attempt to develop a satisfactory film.

51. In films 94 to 98, Vulkamine was varied from 5% to 44%. All these were streaked (due to some incompatibility) except No. 97 (containing only 5% Vulkamine) which was clear. Dilution with the solvent down to 15% solids eliminated the streaking in the others but the 5% Vulkamine film still looked best in the immersion test. This film was also subjected to the newly designed spray test; the first, chronologically speaking, to be so exposed.

52. In films 99 to 106 Vulkamine was kept constant at 5% and staybelite 10 varied from 5% to 40%, nitrocellulose C going from 90% to 55%. It is seen from the data that 20% staybelite or over is necessary to prevent peeling on immersion.

53. As already pointed out in connection with films 82 and 83, if nitrocellulose ½ or ¼ sec. is used instead of nitrocellulose C, the staybelite 10 content must be greatly increased to avoid peeling on immersion and this increase involves an undesirable lowering of contact angle. Since it seemed desirable to use a standard nitrocellulose rather than the ill-defined residue from the evaporation of collodion, several common plasticizing materials were tried at this point in place of staybelite 10 in mixtures containing nitrocellulose ¼ or ½ second and Vulkamine. These included castor oil, dimethyl phthalate, tricresyl phosphate and several staybelites other than No. 10. Also several materials were tried in place

NOTES: *Paraffin replaces ceresin in film 70 since it was found to be more compatible with nitrocellulose.

**It should be noted at this point that while nitrocellulose-staybelite-paraffin films do not fog on immersion as do the F-10 type they lose contact angle more rapidly (compare water bath data on films 74 and 75 with that for 199 in Table VI.)

 [redacted]

of nitrocellulose with the staybelite 10 and Vulkamine. These included cellulose acetates, cellulose acetate butyrate and ethyl cellulose.

54. When neither of these approaches was successful it seemed apparent that the only hope of solving the problem with a nitrocellulose film lay in quantitative experimentation with nitrocellulose $\frac{1}{2}$ (or $\frac{1}{4}$) sec., staybelite 10 and Vulkamine. Accordingly in films 107 to 123 Vulkamine was varied from 5% to 20% for each of 4 different staybelite 10 contents between 70% and 90%, nitrocellulose $\frac{1}{2}$ sec. being used as film former.

55. Addition of a trace of paraffin to film 123 seemed to improve spray test performance (see film 126) but further experimentation without the wax was pursued.

56. In films 127 - 131 Vulkamine was cut below 5% with improved spray test performance as noted. The lower Vulkamine content suggested the possibility that less staybelite 10 might now suffice for adhesion. Accordingly Vulkamine was held constant at 3% and staybelite 10 varied from 40% to 70% in films 132 - 137. With the decreased Vulkamine 60% to 65% staybelite seemed sufficient, 60% being the lowest allowable.

57. In films 138 - 143 the nitrocellulose content was cut to very low percentages but with no improvement.

58. The remainder of the nitrocellulose group films (144 - 153) represent an attempt to obtain a higher contact angle by increasing Vulkamine, staybelite 10 being correspondingly increased to avoid streaking of the film. Film 144 containing 15% Vulkamine and 80% staybelite 10 had the highest contact angle of any adherent and completely compatible nitrocellulose type film. It was considered that 144 represented about the limit in performance attainable with the nitrocellulose-Vulkamine-staybelite 10 combination. This film greatly improved visibility but was not considered an acceptable answer to the problem since its effectiveness diminished rapidly, the water droplets were too large and the film became misty soon after exposure.

59. As previously noted, the F-10 type of film had been abandoned because of the fogging behavior in the immersion test. At this point a few mixtures of the F-10-staybelite-10-wax type, were again prepared and the films subjected to the spray test. The absence of fogging under these more revealing and rigorous conditions seemed to warrant further development work on this type.

60. In films 154 - 162, containing F-10, staybelite 10 and ceresin, the ceresin was kept constant at 2% and staybelite 10 varied from 5% to 70%. Those containing less than 30% staybelite peeled and No. 162 containing 70% staybelite was powdery indicating too much staybelite. Nos. 159, 160 and 161, though very slightly incompatible, performed well as indicated in the data. Thus the optimum staybelite for this type seemed to lie somewhere between 30% and 70%.

61. Substitution of paraffin for ceresin diminished the slight incompatibility and increased the contact angle somewhat. Addition of 2% to 20% nitrocellulose $\frac{1}{2}$ sec. (with the idea of increasing hardness) to the F-10-staybellite-paraffin films caused some incompatibility, and peeling in the spray test where nitrocellulose was above 2% (films 163 - 169).

 [redacted]

62. Films 170 and 171 containing around 40% staybelite gave very good performance in the spray test but the films were still not absolutely free from some incompatibility on drying. Substitution of a mixture of mineral spirits and butyl acetate for straight butyl acetate as solvent (No. 172) gave complete transparency in the dry film.

63. The performance of 172 in the spray test was excellent and from this point the work consisted for the most part of experimentation with solvents in an attempt to reduce the drying time, together with some further variation of the constituents over the narrow ranges already established. This final study resulted in the substitution of ethyl acetate for the less volatile butyl acetate and the final establishment of optimum proportions for the three solid constituents (<u>film 199</u>).

64. Examination of the data in Table VII (plotted in Fig. 3) shows that the original contact angle of water on 199 is considerably higher than that of the Lorr or N.R.C. paste. The angle for 199 drops off more rapidly during the first 1½ hours of immersion but levels off at a higher value than is the case with either of the others, and maintains this advantage during the first eight hours of immersion. Between the 8 and 50 hour marks the Lorr and N.R.C. maintain average angles slightly higher than does 199. The N.R.C. outlasts 199 and Lorr in the immersion test but was eliminated due to very unsatisfactory spray test performance. The data in Table VIII indicates that between one and two hours is required for complete drying of a 199 application. In practice however one hour has been found to be sufficient.

65. The rolling angle data before and after spray exposure (Table IX) leaves little choice between 199 and Lorr but shows the inferiority of the N.R.C. paste in this more rigorous test. Rolling angles for all films decrease as drop size increases, a fact brought out in Fig. 3.

66. The effective life of 199 in 3.5% salt spray is approximately 30% of that in fresh water as determined by substitution of salt water for tap water in the usual spray test. The life of the Lorr material under these conditions is reduced to about the same extent.

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CONCLUSIONS AND RECOMMENDATIONS

67. On the basis of the experimental work involved in the development of N.R.L. 199 and test results on this and several commercial repellents, the following general conclusions are drawn:

(a) A paste type repellent has the advantage of requiring no drying time but satisfactory films are almost impossible to obtain by the rubbing and polishing technique involved, the films are relatively ineffective and durability is poor.

(b) The only stick form of preparation tested requires no drying time, can be applied in rain, and performs fairly well when a good film is obtained. Obtaining a satisfactory film, however, is even more difficult than is the case with the paste type.

(c) The silicomethane type requires a special application technique, is only moderately effective, and is very difficult to remove after breakdown. These compounds readily release hydrochloric acid before and during application. This is corrosive and may be hazardous to personnel.

(d) The lacquer type of preparation requires a drying period and must be applied in a dry, dust-free atmosphere at a temperature above 40°F (somewhat lower for 199) but from the standpoint of reproducibility of films, effectiveness in improving vision, durability and ease of removal it is clearly superior to the other types.

(e) N.R.L. 199 and the Lorr product (both lacquer types) are the most effective rain repellents developed to date. A comparison of these two based on laboratory tests alone indicates little choice between them as far as effectiveness and durability are concerned but the application of 199 is less critical and its effectiveness extends to somewhat lower temperatures (about 30°F as compared to 40°F for the Lorr). Comparative tests in the field, however, under actual service conditions (see appendix) clearly establish the superiority of 199 as a rain repellent coating.

68. It is the opinion of this laboratory that 199 probably represents the optimum in performance attainable with this type of repellent and further work with such mixtures is therefore not recommended except attempts to reduce the tackiness of No. 199. If additional research in this field is contemplated, it is recommended that it envisage the possible development of a permanent, transparent hydrophobic plastic to be substituted for glass or plexiglas in airplane windshields.

69. Pending such possible development it is recommended that since N.R.L. 199 has been proved superior to the Lorr rain repellent, it be substituted for the latter and used wherever conditions permit application in a dry, dust-free atmosphere above 30°F, and a drying time of at least one hour before exposure.

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TABLE I

Contact Angles of Water on Solid Organic Compounds in Air

Compound	Contact Angle (Degrees)	Work of Adhesion Ergs/Sq. Cm.
Hydrocarbons		
Dibenzyl	50	118.8
Stilbene	72	94.7
Diphenyl	68	99.6
*Paraffin	108	49.9
Benzene	105	43.5
Naphthalene	62	106.3
*Anthracene	92	68.6
Diphenyl methane	62	103.6
Triphenyl methane	45	123.4
Tetraphenyl methane	15	135.0
Ethers		
Glyceryl phenyl	51	97.6
Diphenyl	88	75.7
Alcohols		
Myristyl	60	54.2
Cetyl	46	61.3
Menthol	73	71.8
Trichloro tert. butyl	65	102.9
Triphenyl carbinol	57	111.7
Aldehydes		
Trioxymethylene	122	34.5
p-iso-butyraldehyde	70	75.6
Ketones		
Acetophenone	65	83.4
Benzophenone	65	100.0
Phenols		
B-naphthol	35	128.5
Resorcinol	25	137.8
Thymol	85	65.6
Amines and Amides		
Diphenyl amine	80	85.4
2-4-6 trichloro aniline	55	114.5
Acetamide	15	143.0
Halide Substitutions		
*1-2-4 trichloro benzene	98	62.6
Nitrogen Cpds.		
Azobenzene	64	104.5
Hydrazobenzene	52	118.7

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TABLE I (Cont.)

Compound	Contact Angle (Degrees)	Work of Adhesion Ergs/Sq. Cm.
Acids		
Caproic	45	87.0
Caprylic	60	66.2
Pelargonic a	49	66.1
Pelargonic b	69	54.0
Capric	85	45.1
Undecylic	79	50.8
*Lauric	111	32.6
Tridecylic a	75	50.4
*Tridecylic b	95	36.5
*Myristic	115	32.0
Pentadecylic a	73	65.0
*Pentadecylic b	110	37.8
*Palmitic	111	42.8
Margaric a	77	89.0
*Margaric b	105	54.2
*Stearic	106	56.8
*Arachidic	111	46.7
*Behenic	110	47.9
*Cerotic	116	40.8
*Erucic	93	46.2
Cinnamic	40	127.7
Hydrocinnamic	63	87.2
Undecylenic	53	70.0
Benzoic	65	93.6
Esters		
Methyl cinnamate	45	111.0
Ethylene glycol dicaprylate	84	68.8
*Ethylene glycol dicaprate	96	59.5
*Ethylene glycol dilaurate	123	32.8
*Ethylene glycol dimyristate	110	47.6
*Ethylene glycol dipalmitate	98	62.2
Ethylene glycol distearate	75	91.1
Butyl carbamate	40	95.1
*Tristearin	110	47.0
Miscellaneous		
Benzyl sulfide	90	72.8
*Zinc stearate	135	21.3
Castile soap powdered	68	-

* Contact angle above 90°

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MIXTURES IN WHICH F-10 (ACRYLOID RESIN) IS THE FILM FORMING AGENT.

TABLE II

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
1.	15% F-10 Resin 65% Mineral Spts. 20% F-10 Thinner.	100% F-10	50	83	Fogs but clears in 3 min. on removal.	Peels off almost immediately. Droplets large due to low contact angle.	Film clear and smooth.
2.	12.02% F-10 .64% Paraffin 67.34% Min. Spts. 20% F-10 Thinner	96% F-10 4% Paraffin	60	103	Fogs, clears in 5 min. on removal.	Good repellent, fine droplets but peels almost immediately.	Film a little cloudy.
3.	12.22% F-10 .48% Paraffin 67.34% Min. Spts. 20% F-10 Thinner	97.% F-10 3% Paraffin	60	102	Same as No. 2	Same as No. 2	Film slightly streaked.
4.	12.33% F-10 .32% Paraffin 67.34% Min. Spts. 20% F-10 Thinner	98% F-10 2% Paraffin	60	100	Same as No. 2 except clears more quickly on removal.	Same as No. 2.	Film clear.
5.	12.5% F-10 .16% Paraffin 67.34% Min. Spts. 20% F-10 Thinner	99% F-10 1% Paraffin	60	92	Same as No. 4.	Same as No. 2.	Film clear.
	12.58% F-10 .08% Paraffin 67.34% Min. Spts. 20% F-10 Thinner	99.5% F-10 .5% Paraffin	60	86	Same as No. 4.	Same as No. 2.	Film clear.
	2.3% F-10 7.4% Rosin .3% Paraffin 17% Min. Spts. 67% Amyl acetate 6% F-10 Thinner	24% F-10 72% Rosin 4% Paraffin	50	90	Film disintegrates.	Not tested.	Film powdery and rough on drying.
	2.3% F-10 7.5% Rosin .2% Paraffin 17% Min. Spts. 67% Amyl acetate 6% F-10 Thinner	25% F-10 72% Rosin 3% Paraffin	50	85	Film disintegrates.	Not tested.	Film powdery and rough on drying.

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TABLE II (Cont.)

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
9.	2.4% F-10 7.58% Rosin .12% Paraffin 17% Min. Spts. 67% Amyl acetate 6% F-10 Thinner	25% F-10 2% Paraffin 73% Rosin	50	72	Not immersed; contact angle too low.	Not tested.	Film powdery and rough on drying.
10.	2.4% F-10 7.53% Rosin .07% Paraffin 67% Amyl acetate 17% Min. Spts. 6% F-10 Thinner	26% F-10 73% Rosin 1% Paraffin	50	70	Not immersed; contact angle too low.	Not tested.	Film powdery.
11.	2.5% F-10 7.5% Staybellite 10 17% Min. Spts. 67% Butyl acetate 6% F-10 Thinner	25% F-10 75% Staybellite 10	50	70	Not immersed; contact angle too low.	Not tested.	Clear film.
12.	2.3% F-10 7.4% Staybellite 10 .3% Paraffin 17% Min. Spts. 67% Butyl acetate 6% F-10 Thinner	24% F-10 72% Staybellite 10 4% Paraffin	50	92	Film looks good for 1 hour, then fogs and appears glazed.	Not tested.	Clear film.
13.	2.3% F-10 7.5% Staybellite 10 .2% Paraffin 17% Min. Spts. 67% Butyl acetate 6% F-10 Thinner	25% F-10 72% Staybellite 10 3% Paraffin	50	90	Fogging not as bad as 12 but glazed appearance.	Not tested.	Clear film.
14.	24% F-10 7.58% Staybellite 10 .12% Paraffin 17% Min. Spts. 67% Butyl acetate 6% F-10 Thinner	25% F-10 73% Staybellite 10 2% Paraffin	50	90	Fogging little less than 13. Glazed.	Not tested.	Clear film.
15.	2.4% F-10 7.53% "taybellite 10 .07% .araffin 17% Min. Spts. 67% Butyl acetate 6% F-10 Thinner	26% F-10 73% Staybellite 10 1% Paraffin	50	90	Fogging little less than 13. Glazed.	Not tested.	Clear film.

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TABLE II (Cont.)

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
16.	12% F-10 2% Vulkamine .07% Paraffin 55% Min. Spts. 12% Butyl acetate 18% F-10 Thinner	84% F-10 15% Vulkamine 1% Paraffin	50	90	Clouds badly but film stands up overnight.	Not tested.	Streaked on drying.
17.	12% F-10 2% Vulkamine 15% Paraffin 55% Min. Spts. 12.8% Butyl acetate 18% F-10 Thinner	84% F-10 14% Vulkamine 2% Paraffin	50	90	Fogs badly.	Not tested.	Streaks on drying.
18.	9% F-10 1% Vistanex 10% Solvesso 60% Min. Spts. 20% F-10 Thinner	90% F-10 10% Vistanex	60	80	Film Disintegrates.	Not tested.	Film clear.
19.	10% F-10 2% Liquid Petro-latum 66% Min. Spts. 22% F-10 Thinner	80% F-10 20% Liquid Petro-latum	60	80	Not immersed.	Not tested.	Film very sticky.
20.	9% F-10 .1% Liquid Petro-latum 67.9% Min. Spts. 23% F-10 Thinner	99% F-10 1% Liquid Petrolatum	60	90	Becomes glazed - vision bad.	Not tested.	Petrolatum is too sticky and soft.
21.	9% F-10 1% Camphor 68% Min. Spts. 22% F-10 Thinner	90% F-10 10% Camphor	60	85	Fogs badly and disintegrates after 2 hours.	Not tested.	
22.	13% F-10 2% Shellac 54% Min. Spts. 13% Butyl acetate 18% F-10 Thinner	78% F-10 22% Shellac	60	90	Stands up well but fogs.	Not tested.	Clear film.

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TABLE II (Cont.)

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
38.	10% F-10 .1% ceresin 66.9% Min.Spts. 22% F-10 Thinner	99% F-10 1% ceresin	60	94			
39.	10% F-10 .05% ceresin 68% Min. Spts. 22% F-10 Thinner	99.5% F-10 .5% ceresin	60	90			
40.	Same as 32 except ceresin in butyl acetate.	Same as 32.	60	105	Films 40 - 47 same comment as on 32 - 39.	Films 40 - 47 same comment as on 32 - 39.	Perfectly clear on drying while films 32 - 39 slightly cloudy.
41.	Same as 33 except ceresin in butyl acetate.	Same as 33.	60	105			
42.	Same as 34 except ceresin in butyl acetate.	Same as 34.	60	105			
43.	Same as 35 except ceresin in butyl acetate.	Same as 35.	60	105			
44.	Same as 36 except ceresin in butyl acetate.	Same as 36.	60	102			
45.	Same as 37 except ceresin in butyl acetate.	Same as 37.	60	100			
46.	Same as 38 except ceresin in butyl acetate.	Same as 38.	60	94			
47.	Same as 39 except ceresin in butyl acetate.	Same as 39.	60	90			

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TABLE II (Cont.)

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
48.	7.7% F-10 2% Rosin 48% Min.Spt. 26.3% Butyl acetate 16% F-10 Thinner	78% F-10 20% Rosin 2% Ceresin	60	87	Disintegrates.	No test.	Film powdery on drying.
49.	9.9% F-10 .02% Rosin .08% Ceresin 65% Min.Spts. 23% Butyl Acetate	99% F-10 .2% Rosin .8% Ceresin	60	92	Contact angle 87 after 20 hours. Contact angle 80 after 44 hours. Clouds and clears on removal.	Not tested	Films No. 49 - 55 - All fog and clear up in 2 to 4 minutes after removal.
50.	9.9% F-10 .04% Rosin .06% Ceresin 65% Min.Spts. 23% Butyl Acetate	99% F-10 .4% Rosin .6% Ceresin	60	90	Contact angle 84 after 20 hours. Contact angle 76 after 44 hours.	Not tested.	Small amounts of rosin seem to cause films to last longer in H_2O bath, since F-10 ceresin films are completely gone at end of 44 hours.
51.	9.9% F-10 .06% Rosin .04% Ceresin 65% Min.Spts. 3% Butyl Acetate 22% F-10 Thinner	99% F-10 .6% Rosin .4% Ceresin	60	87	Contact angle 82 after 20 hours. Contact angle 70 after 44 hours.	Not tested.	These plates (49 - 55) however show a tendency to spot where H_2O droplets have been drying.
	9.9% F-10 .08% Rosin .02% Ceresin 65% Min.Spts. 3% Butyl Acetate 22% F-10 Thinner	99% F-10 .8% Rosin .2% Ceresin	60	85	Contact angle 80 after 20 hours. Contact angle 80 after 44 hours.	Not tested.	Plates 48 to 5[illegible] were repeated usinga [illegible]ew improved wood rosin. [illegible]esults same except [illegible] rosin less sticky. Also 32 to 39 repeated using $CHCl_3$ as solvent for the ceresin. [illegible]ts no different except drying time somewhat diminished.
	9.9% F-10 .1% Rosin 0%Ceresin 65% Min. Spts. 3% Butyl acetate 22% F-10 Thinner	99% F-10 1% Rosin 0% Ceresin	60	85	Contact angle 84 after 20 hours. Contact angle 72 after 44 hours.	Not tested.	
	9.9% F-10 .06% Rosin 0% Ceresin 65% Min.Spts. 3% Butyl Acetate 22% F-10 Thinner	99% F-10 .6% Rosin 0% Ceresin	60	83	Contact angle 83 after 20 hours. Contact angle 83 after 44 hours.	Not tested.	

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TABLE II (Cont.)

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
55.	9.9% F-10 .02% Rosin 0% Ceresin 65% Min.Spts. 3% Butyl Acetate 22% F-10 Thinner	99% F-10 .2% Rosin 0% Ceresin	60	83	Contact angle 79 after 20 hours. Contact angle 70 after 44 hours.	Not tested.	
56.	12% F-10 3% Poly Pale Resin 51% Min. Spts. 17% Butyl Acetate 17% F-10 Thinner	80% F-10 20% Poly Pale Resin	60	83	Not immersed - angle low.	Not tested.	
57.	11% F-10 3% Poly Pale Resin 45% Min.Spts. 26% Butyl Acetate .2% Ceresin 15% F-10 Thinner	78% F-10 20% Poly Pale Resin 2% Ceresin	60	104	Broke down in 2 hours. Film gone.	Not tested.	
58.	11% F-10 3% Poly Pale Resin 48% Min. Spts. 22% Butyl Acetate .1% Ceresin 16% F-10 Thinner	78% F-10 21% Poly Pale Resin 1% Ceresin	60	100	Broke down in 2 hours - film gone.	Not tested.	
59.	11% F-10 3% Poly Pale Resin .1% Paraffin 48% Min.Spts. 16% F-10 Thinner 22% Butyl Acetate	78% F-10 21% Poly Pale Resin 1% Paraffin	60	102	Broke down in 2 hours.	Not testéd.	
NOTE: See discussion for explanation of discontinuity in numbers at this point.							
154.	14% Solids 63% Butyl Acetate 23% F-10 Thinner	93% F-10 5% Staybellite 10 2% Ceresin	75	100		No. 161 looks best in spray test.	
155.	14% Solids 66% Butyl Acetate 20% F-10 Thinner	88% F-10 10% Staybellite 10 2% Ceresin	75	99		154, 155, 156, 157 and 158 peeled, due evidently to not enough staybellite. No. 162 is somewhat powdery in appearance due to too much staybellite.	

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TABLE II (Cont.)

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
166.	14% Solids 86% Butyl Acetate	38% F-10 10% Nitrocell.1/2 Sec. 50% Staybellite 10 2% Paraffin	75	100		Drops large, vision not good. Peels.	
167.	14% Solids 86% Butyl Acetate	33% F-10 15% Nitrocell.1/2Sec. 50% Staybellite 10 2% Paraffin	75	100		Peels.	Streaked a little on drying.
168.	14% Solids 86% Butyl Acetate	20% F-10 20% Nitrocell.1/2 Sec. 58% Staybellite 10 2% Paraffin	75	100		Peels.	Streaked on drying.
169.	14% Solids 86% Butyl Acetate	46% F-10 2% Nitrocell.1/2 Sec. 50% Styabellite 10 2% Paraffin	75	100		Looks good and does not peel.	
170.	14% Solids 86% Butyl Acetate	58% F-10 0% Nitrocell.1/2Sec. 40% Staybellite 10 2% Paraffin	75	100		170 and 171 both look very good. Drops still very small and vision not much diminished after 3-1/2 hours. Fogging and misting pretty evident after 4 hours - no difference between 170 and 171.	No distinction between 2% nitrocell. and no nitrocell.
71.	14% Solids 86% Butyl Acetate	58% F-10 0% Nitrocell.1/2Sec. 41% Staybellite 10 1% Paraffin	75	100			
72.	6% F-10 5% Staybellite 10 .3% Paraffin 28% Min.Spts. 60.7% Butyl Acetate	58% F-10 40% Staybellite 10 2.5% Paraffin	60	101		Vision after 3-1/2 hours spraying approximated to be 3/4 as good as at start. At end of 7 hours spray vision still good. Photographs taken.	A few clinging drops appeared during course of spray indicating bad spots in film.
73.	6% F-10 5% Staybellite #1 .3% Paraffin 28% Butyl Acetate 60.7% Min. Spts.	47.5% F-10 50% Staybellite #1 2.5% Paraffin	75	98		Inferior to those containing staybellite 10. More spray.	

[redacted]

TABLE II (Cont.)

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
174.	9% F-10 6% Staybellite 10 .05% Paraffin 65.5% Ethyl Acetate 7% Min. Spts. 12% F-10 Thinner	59.5% F-10 40% Staybellite 10 .5% Paraffin	45 - 60	90			
175.	9% F-10 6% Staybellite 1p 60% Ethyl Acetate .1% Paraffin 12.5% Min. Spts. 12% F-10 Thinner	59% F-10 40% Staybellite 10 1% Paraffin	45 - 60	92			
176.	9% F-10 6% Staybellite 10 .15% Paraffin 55% Ethyl Acetate 17.5 % Min. Spts. 12% F-10 Thinner	58.5% F-10 40% Staybellite 10 1.5% Paraffin	45 - 60	92			
177.	9% F-10 6% Staybellite 10 .2% Paraffin 50% Ethyl Acetate 22.5% Min. Spts. 12% F-10 Thinner	58% F-10 40% Staybellite 10 2% Paraffin	45 - 60	100		Looks good - stands up about like 172 above.	Film nice and clear.
178.	9% F-10 6% Staybellite 1p .25% Paraffin 45% Ethyl Acetate 27.5% Min. Spts. 12% F-10 Thinner	57.5% F-10 40% Staybellite 10 2.5% Paraffin	45 - 60	100		Not quite as good as 172.	Film very slightly cloudy.
179.	11% F-10 4% Staybellite 10 .2% Paraffin 60% Ethyl Acetate 12.8% Min. Spts. 12% F-10 Thinner	61% F-10 37% Staybellite 10 2% Paraffin	45 - 60	102		Shows a little tendency to peel. Staybellite a little low.	
180.	5.5% F-10 6% Staybellite 10 .3% Paraffin 45% Ethyl Acetate 34.2% Min. Spts. 10% F-10 Thinner	47% F-10 50% Staybellite 10 3% Paraffin	45 - 60	100		Looks good. Vision practically unimpaired after 2 hours.	A little streaked on drying. Paraffin high.

[redacted]

TABLE II (Cont.)

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
181.	5.5% F-10 6% Staybellite 10 .3% Ceresin 34.2% Butyl Acetate 45% Ethyl Acetate 10% F-10 Thinner	47% F-10 50% Staybellite 10 3% Ceresin	45 - 60	102		Not quite as good as 180 above and clouding worse.	Somewhat clouded.
182.	9% F-10 6% Staybellite 10 .25% Paraffin 27.5% Butyl Acetate 45% Ethyl Acetate 12% F-10 Thinner	58% F-10 40% Staybellite 10 2.5% Paraffin	45 - 60	100		Not good in spray test. This solvent mixture does not seem to work so well. Mist bad.	
183.	6.5% F-10 6% Staybellite 10 .3% Paraffin 14% Benzene 64.5% Min.Spts. 8.7% F-10 Thinner	58% F-10 40% Staybellite 10 2.5% Paraffin	45 - 60	100		Film clear but on exposure to spray clouds badly right away.	
184.	6% F-10 6% Staybellite 10 .3% Paraffin 75% Benzene 5% Toluene 7.7% F-10 Thinner	58% F-10 40% Staybellite 10 2.5% Paraffin	45 - 60	101		No good. Spray bad and clouds.	
185.	6.5% F-10 6% Staybellite 10 .3% Paraffin 40% Ethyl Acetate 2.2% Toluene 40% Butyl Acetate 5% F-10 Thinner	58% F-10 40% Staybellite 10 2.5% Paraffin	45 - 60	100		Mist bad in spray test.	
186.	6.5% F-10 6% Staybellite 10 .3% Paraffin 40% Ethyl Acetate 42.2% Butyl Acetate 5% F-10 Thinner	58% F-10 40% Staybellite 10 2.5% Paraffin	45 - 60	100		Mist bad in spray test.	

[redacted]

TABLE II (Cont.)

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
187.	6% F-10 6% Staybellite 10 .3% Paraffin 20.7% Butyl Acetate 40% Ethyl Acetate 20% Toluene 7% F-10 Thinner	58% F-10 40% Staybellite 10 2.5% Paraffin	45 - 60	100		No good - clouds badly.	
188.	7.5% F-10 7% Staybellite 10 .3% Paraffin 80.2% Toluene 7% F-10 Thinner	58% F-10 40% Staybellite 10 2.5% Paraffin	75	100			Streaks on drying.
189.	5% F-10 5% Staybellite 10 .4% Paraffin 83.6% Toluene 6% F-10 Thinner	48% F-10 48% Staybellite 10 4% Paraffin	75	100		Looks very good. Vision good after 4 hours. Not enough clouding to hinder vision, few slight flaws in film however.	Film very slightly clouded on drying.
190.	.3% Paraffin 5% F-10 5% Staybellite 10 83.7% Toluene 6% F-10 Thinner	48% F-10 47% Staybellite 10 3% Paraffin	75	100		About same as 189. Few slight flaws.	
191.	6.5% F-10 6% Staybellite 10 .4% Paraffin 81.6% Toluene 6% F-10 Thinner	48% F-10 48% Staybellite 10 4% Paraffin	75	103			Film a little streaked.
192.	5% F-10 5% Staybellite 10 .55% Paraffin 70% Min. Spts. 13.45% Butyl Acetate 6% F-10 Thinner	55% F-10 40% Staybellite 10 5% Paraffin	75	103		Clouds pretty badly.	Clouds, too much pasaffin.

[redacted]

TABLE II (Cont.)

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
193.	5% F-10 5% Staybellite 10 .2% Paraffin 4.8% Min. Spts. 70% Butyl Acetate 5% F-10 Thinner	58% F-10 40% Staybellite 10 2% Paraffin	75	100		Looks very good. Vision good after 4 hours. No flaws.	
194.	4.94% F-10 3.97% Staybellite 10 .5% Paraffin 9% Butyl Acetate 74.66% Min. Spts. 7% F-10 Thinner	56% F-10 40% Staybellite 10 2% Paraffin	75	99			Clouds on drying.
195.	7.4% F-10 7.4% Staybellite 10 .3% Paraffin 25% Ethyl Acetate 50% Min. Spts. 9.9% F-10 Thinner	48.5% F-10 48.5% Staybellite 10 3% Paraffin	45 - 60	100			Clouds on drying.
196.	6% F-10 6.84% Staybellite 10 .26% Paraffin 49% Ethyl Acetate 29% Min. Spts. 9% F-10 Thinner	49% F-10 49% Staybellite 10 2% Paraffin	45 - 60	100		Looks good in spray except for a few clinging drops. Vision good after 5 hours.	
197.	6% F-10 4.6% Staybellite 10 .3% Paraffin 54% Min. Spts. 27% Ethyl Acetate 8.1% F-10 Thinner	55.6% F-10 41.66% Staybellite 10 2.73% Paraffin	45 - 60	100		Looks good - about like 196.	
98.	6.6% F-10 5% Staybellite 10 .33% Paraffin 49.6% Min.Spts. 29% Ethyl Acetate 9.28% F-10 Thinner	55.6% F-10 41.6% Staybellite 10 2.7% Paraffin	45 - 60	100		Looks very good - no flaws. Stands up. Vision good after 8 hours.	

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T A B L E II (Cont.)

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
197.	5.58% F-10 4.29% Staybellite 10 .29% Paraffin 7.77% F-10 Thinner 53.79% Ethyl acetate 28.28% Min. Spts.	54.93% F-10 42.25% Staybellite 10 2.82% Paraffin	60	103	See Table VI and Figure IV.	Most effective film developed; vision excellent. Stands up well 8 to 10 hours.	It is considered that this film probably represents the optimum in performance attainable with a lacquer type preparation.

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MIXTURES IN WHICH NITROCELLULOSE IS THE FILM FORMING AGENT.

TABLE III

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
60.	Collodion (alc. and ether not removed.)	100% Nitro-cellulose C	-	-	-	-	Film badly clouded. Solvent too volatile.
61.	Collodion C 15% Butyl Acetate 85%	100% Nitro-cellulose C	60	70	Peels off right away.		Drops move easily on dry surface even though contact angle low.
62.	12.02% Nitrocell.C .64% Ceresin 87.34% Butyl Acetate	96% Nitrocell.C 4% Ceresin	60	103	Peels in short time.		Film streaked.
63.	12.05% Nitrocell.C .32% Ceresin 87.66% Butyl Acetate	98% Nitrocell.C 2% Ceresin	60	100	Peels in short time.		Film very slightly streaked.
64.	12.02% Nitrocell.C .16% Ceresin 87.82% Butyl Acetate	99% Nitrocell.C 1% Ceresin	60	96	Peels in short time.		Film clear.
65.	7% Nitrocell. C. 7.84% Rosin .16% Ceresin 85% Butyl Acetate	50% Nitrocell.C 49% Rosin 1% Ceresin	60	80	Not immersed - angle too low.		
66.	6% Nitrocell. C 6% Poly Pale resin .16% Ceresin 87.84% Butyl Acetate	50% Nitrocell. C 49% Poly pale resin 1% Ceresin	60	78	Film adheres but angle too low. Drops leave spots.		
67.	11% Nitrocell. C 1% Pale Resin .16% Ceresin 87.84% Butyl Acetate	90% Nitrocell. C 9% Poly pale resin 1% Ceresin	60	76	Film adheres but angle too low. Drops leave spots.		
68.	6% Nitrocell. C 6% Flexalyn Resin .16% Ceresin 87.84% Butyl Acetate	50% Nitrocell. C 49% Flexalyn 1% Ceresin	180 plus	98			Film sticky - will not dry under 3 hours.
69.	6% Nitrocell. C 6% Flexalyn .16% Ceresin 87.84% Butyl Acetate	90% Nitrocell. C 9% Flexalyn 1% Ceresin	180-plus	100			Film sticky - will not dry under 3 hours.

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TABLE III (Cont.)

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
70.	10% Nitrocell. C 4% Staybellite 10 .32% Paraffin 85.68% Butyl Acetate	58% Nitrocell.C 40% Staybellite 10 2% Paraffin	60	97	Looks good. Film clear on removal and adheres overnight.	Spray data on similar films obtained later.	Staybellite 10 imparts adherence to film and also has good water resistance.
71.	Same as 70 except Nitrocellulose ½ sec. used.	Same as 70 except Nitrocellulose ½ sec. used.	60	96	Peels immediately.		40% Staybellite caused films containing nitrocellulose from collodion to adhere but not cellulose ½ sec.
72.	Same as 71 except nitrocellulcse ½ sec. used.	Same as 71 except nitrocellulose ½ sec. used.	60	96	Peels immediately.		40% staybellite caused films containing nitrocellulose from collodion to adhere but not cellulose ½ sec.
73.	12.02% Nitrocell. C .32% Paraffin 87.66% Butyl Acetate	98% Nitrocell. C 2% Ceresin	60	100	Peels immediately		Plate slightly clouded.
74.	7.4% Nitrocell. C 2.5% Staybellite 10 .1% Paraffin 90% Butyl acetate	74% Nitrocell. C 25% Staybellite 10 1% Paraffin	60	100	Angle - 70 after 20 hours, no clouding.		
75.	85% Nitrocell. C 1.4% Staybellite 10 .1% Paraffin 90% Butyl Acetate	85% Nitrocell.C 14% Staybellite 10 1% Paraffin	60	95	Angle - 70 after 20 hours, no clouding.		
76.	9.3% Nitrocell. C .6% Staybellite 10 .1% Paraffin 90% Butyl Acetate	93% Nitrocell C 6% Staybellite 10 1% Paraffin	60	78	Not immersed - angle too low.		
77.	9.6% Nitrocell.C .3% Staybellite 10 .1% Paraffin 90% Butyl Acetate	96% Nitrocell. C 3% Staybellite 10 1% Paraffin	60	70	Not immersed - angle too low.		
78.	9.3% Nitrocell. C .7% Staybellite 10 90% Butyl Acetate	93% Nitrocell. C 7% Staybellite 10	60	72	Not immersed - angle too low.		

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TABLE III (Cont.)

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
79.	9.65% Nitrocell.C .35% Staybellite 10 90% Butyl Acetate	96.5% Nitrocell.C 35% Staybellite 10	60	70	Not immersed - angle too low.		
80.	9.9% Nitrocell.C .1% Staybellite 10 90% Butyl Acetate	99% Nitrocell.C 1% Staybellite 10	60	70	Not immersed - angle too low.		
81.	9.9% Nitrocell.C .05% Staybellite 10 90.05% Butyl Acetate	99.5% Nitrocell.C .5% Staybellite 10	60	68	Not immersed - angle too low.		
82.	2.3% Nitrocell. ¼ sec. 7.5% Staybellite 10 .2% Paraffin 90% Butyl Acetate	23% Nitrocell.¼ sec. 75% Staybellite 10 2% Paraffin	60	86	Adheres - does not fog.		Adheres but angle lowered by large amount staybellite. Film blushes slightly.
83.	.8% Nitrocell.¼ sec. 9% Staybellite 10 .2% Paraffin 90% Butyl Acetate	8% Nitrocell.¼ sec. 90% Staybellite 10 2% Paraffin	60	85	Adheres but spots some.		Film somewhat powdery. Angle lowered by large amount staybellite.
	8% Nitrocell. ¼ sec. 2% Camphor .2% Paraffin 89.8% Butyl Acetate	80% Nitrocell. ¼ sec. 20% Camphor 2% Paraffin	60	98	Peels off.		Camphor accomplishes nothing.
	Same as 84 except nitrocell. ½ sec. used.	Same as 84 except nitrocell. ½ sec. used.	60	97	Peels off.		Camphor accomplishes nothing.
	Same as 82 except Rosin (new wood) replaces staybellite 10.	Same as 82 except Rosin replaces staybellite 10.	60	75	Not immersed - angle too low.		
	Same as 82 except poly pale clear resin replaces staybellite 10.	Same as 82 except poly pale resin replaces staybellite 10.	60	70	Not immersed - angle too low.		

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TABLE III (Cont.)

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
88.	9.6% Nitrocell.½ sec. 2.4% Vulkamine 88% Butyl Acetate	80% Nitrocell.C 20% Vulkamine	60	95	Peels in bath.		Highest contact angle yet obtained without using a wax. Film slightly streaked.
89.	7% Nitrocell.½ sec. 1% Vulkamine 2% Rosin 90% Butyl Acetate	70% Nitrocell.½ sec. 10% Vulkamine 20% Rosin	60	87	Adheres but not immersed - angle too low.		Powdery.
90.	8% Nitrocell. C 2% Vulkamine 90% Butyl Acetate	80% Nitrocell.C 20% Vulkamine.	60	95	Peels off.		
91.	4% Nitrocell.C 2% Vulkamine 4% Staybellite 10 90% Butyl Acetate	40% Nitrocell. C 20% Vulkamine 40% Staybellite 10	60	90	Adheres. Stands up well overnight - no fogging. Drops lie flat on removal.		
92.	4% Nitrocell.C 2% Vulkamine 3.8% Staybellite 10 90% Butyl Acetate 2% Paraffin	40% Nitrocell.C 20% Vulkamine 38% Staybellite 10 2% Paraffin	60	95	Stands up well in bath. Drops more spherical on removal. No fogging.		Film cloudy on drying.
93.	8% Nitrocell.C 1.8% Vulkamine .2% Paraffin 90% Butyl Acetate	80% Nitrocell.C 18% Vulkamine 2% Paraffin	60	95	Peeled off.		
94.	12% Nitrocell.C 4% Vulkamine 4% Staybellite 10 80% Butyl Acetate	56% Nitrocell.C 22% Vulkamine 22% Staybellite 10	60	94	After 20 hours, not fogged but somewhat hazy. Haziness disappears shortly on removal. Drops lie rather flat. Angle - 89 after 20 hours.		Film streaky.
95.	132% Nitrocell.C 3.4% Vulkamine 3.4% Staybellite 10 80% Butyl Acetate	66% Nitrocell.C 17% Vulkamine 17% Staybellite 10	60	91	Same remarks as for 94. Angle - 88 after 20 hours.		film streaky.
96.	14.6% Nitrocell.C 1.8% Vulkamine 3.6% Staybellite 10 80% Butyl Acetate	73% Nitrocell.C 9% Vulkamine 18% Staybellite 10	60	90	A little tendency to peel, otherwise about same as above. Angle 87 after 20 hours.		Film streaky.

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TABLE III (Cont.)

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
97.	15% Nitrocell.C 1% Vulkamine 4% Staybellite 10 80% Butyl Acetate	75% Nitrocell.C 5% Vulkamine 20% Staybellite 10	60	90	Looks best at this group. Angle - 88 after 20 hours.	First film (chronologically) exposed to spray test. Vision good for 15 minutes, then mists badly.	Film clear.
98.	8.8% Nitrocell. C 8.8% Vulkamine 2.4% Staybellite 10 80% Butyl Acetate	44% Nitrocell. C 44% Vulkamine 12% Staybellite 10	60	90	Fair		Film very streaky.
99.	13.5% Nitrocell. C .75% Staybellite 10 .75% Vulkamine 85% Butyl Acetate	90% Nitrocell.C 5% Staybellite 10 5% Vulkamine	60	95	Peeled off.		Not enough staybellite for adhesion.
100.	12.75% Nitrocell.C 1.5% Staybellite 10 .75% Vulkamine 85% Butyl Acetate	85% Nitrocell.C 10% Staybellite 10 5% Vulkamine	60	93	Peeled off.		
101.	12% Nitrocell.C 2.25% Staybellite 10 .75% Vulkamine 85% Butyl Acetate	80% Nitrocell.C 15% Staybellite 10 5% Vulkamine	60	91	Peeled off.		
102.	11.25% Nitrocell.C 3% Staybellite 10 .75% Vulkamine 85% Butyl Acetate	75% Nitrocell.C 20% Staybellite 10 5% Vulkamine	60	92	Contact angle of all these around [illegible] - 90 after 20 hours. Plates don't fog. Drops stick rather flat - interspersed with air bubbles - these blow off easily on removal.		Staybellite must be over 20% to secure adhesion. 20% seems as effective as larger amount. Addition of paraffin or ceresin causes blushing.
103.	10.5% Nitrocell.C 3.75% Staybellite 10 .75% Vulkamine 85% Butyl Acetate	70% Nitrocell.C 25% Staybellite 10 5% Vulkamine	60	90			
104.	9.75% Nitrocell.C 4.5% Staybellite 10 .75% Vulkamine 85% Butyl Acetate	65% Nitrocell.C 30% Staybellite 10 5% Vulkamine	60	90			
105.	9% Nitrocell.C 5.25% Staybellite 10 .75% Vulkamine 85% Butyl Acetate	60% Nitrocell.C 35% Staybellite 10 5% Vulkamine	60	90			
106.	8.25% Nitrocell.C 6% Staybellite 10 .75% Vulkamine 85% Butyl Acetate	55% Nitrocell.C 40% Staybellite 10 5% Vulkamine	60	90			

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T A B L E III (Cont.)

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
107.	15% Solids 85% Butyl Acetate	50% Staybellite 10 30% Nitrocell. ½ 20% Vulkamine	75	94	Peeled off.	Spray tests on this group carried out only on Nos. 116, 117 and 119 since they were the 3 which neither streaked nor peeled.	Slightly streaky on drying.
108.	15% Solids 85% Butyl Acetate	50% Staybellite 10 35% Nitrocell. ½ 15% Vulkamine	75	92	Peeled off.	Not much distinction between the 3. Vision excellent for 5 minutes. After that - somewhat misty. This does not impair vision. Vision noticeably worse after ½ hour and only fair at end of 1 hour.	Very streaky on drying.
109.	15% Solids 85% Butyl Acetate	50% Staybellite 10 40% Nitrocell. ½ 10% Vulkamine	75	92	Peeled off.		Streaky on drying.
110.	15% Solids 85% Butyl Acetate	50% Staybellite 10 45% Nitrocell. ½ 5% Vulkamine	75	98	Peeled off.		Very slightly streaky on drying.
111.	15% Solids 85% Butyl Acetate	60% Staybellite 10 20% Nitrocell. ½ 20% Vulkamine	75	93	Stood up overnight. Angle - 93 next morning.	Contact angle drops considerably and is not high enough at start to make droplets as small as they should be.	Slightly streaky on drying.
112.	15% Solids 85% Butyl Acetate	60% Staybellite 10 25% Nitrocell. ½ 15% Vulkamine	75	94	Peeled off.		Very streaky on drying.
113.	15% Solids 85% Butyl Acetate	60% Staybellite 10 30% Nitrocell. ½ 10% Vulkamine	75	100	Partially peeled.		Streaky on drying.
114.	15% Solids 85% Butyl Acetate	60% Staybellite 10 35% Nitrocell. ½ 5% Vulkamine	75	93	Angle - 90 in morning but partially peeled.		Not streaky.
115.	15% Solids 85% Butyl Acetate	70% Staybellite 10 10% Nitrocell. ½ 20% Vulkamine	75	95	Stood overnight - angle - 90 next morning.		Very streaky.
116.	15% Solids 85% Butyl Acetate	70% Staybellite 10 15% Nitrocell. ½ 15% Vulkamine	75	93	Stood overnight angle - 88 next morning.		Not streaky.
117.	15% Solids 85% Butyl Acetate	70% Staybellite 10 20% Nitrocell. ½	75	93	Angle - 90 after overnight immersion.		Not streaky.

[redacted]

TABLE III (Cont.)

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
118.	15% Solids 85% Butyl Acetate	70% Staybellite 10 25% Nitrocell. ½ 5% Vulkamine	75	92	Angle -92 after overnight immersion.		Slightly streaky.
119.	15% Solids 85% Butyl Acetate	80% Staybellite 10 20% Nitrocell. ½ 10% Vulkamine	75	90	Angle - 92 after overnight immersion.		Not streaky.
120.	15% Solids 85% Butyl Acetate	80% Staybellite 10 15% Nitrocell. ½ 5% Vulkamine	75	95	Angle - 90 after overnight immersion.		Not streaky but a little powdery.
121.	15% Solids 85%Butyl Acetate	90% Staybellite 10 1% Nitrocell. ½ 9% Vulkamine	75	95	Angle - 90 after overnight immersion.		Not streaky but a little powdery.
122.	15% Solids 85% Butyl Acetate	90% Staybellite 10 5% Nitrocell. ½ 5% Vulkamine.	75	88	Angle - 90 after overnight immersion.		Not streaky but a little powdery.
123.	15% Solids 85% Butyl Acetate	75% Staybellite 10 20% Nitrocell. ½ 5% Vulkamine	75	92	Stands up well.	Spray behavior as described for 116, 117, 119.	123 was made up as result of above study. Film perfectly clear and compatible. 75% seems optimum staybellite content for nitrocellulose ½ sec.
124.	15% Solids 85% Butyl Acetate	75% Staybellite 10 19% Nitrocell. ½ 5% Vulkamine 1% Paraffin	75	92		Spray test looks fair. Clouds pretty badly in one hour.	
125.	15% Solids 85% Butyl Acetate	75% Staybellite 10 19-1/3% Nitrocell. ½ 5% Vulkamine 2/3% Paraffin	75	93		Spray test fair. Clouds good bit after 45 minutes.	
126.	15% Solids 85% Butyl Acetate	75% Staybellite 10 19-2/3% Nitrocell.½ 5% Vulkamine 1/3% Paraffin	75	93		Spray test looks best of nitrocellulose films tried thus far. Vision good after 30 minutes - fair after one hour - bad after 2 hours. Drops rather large.	

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TABLE III (Cont.)

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
127.	15% Total Solids 85% Butyl Acetate	75% Staybellite 10 21% Nitrocell. ½ sec. 4% Vulkamine	75	93		All these films (except 131), look good in spray test, 128 being the best. Vision unimpaired after 15 minutes; only slightly impaired after one hour. Drops, however, rather large but do not interfere too badly with vision of distant objects. Fogged some after one hour but vision through dry film compared to glass practically unimpaired. Fogged good bit after 2 hours and vision bad then.	127 - 131 all clear and compatible. Elimination of Vulkamine (131) cuts contact angle and reduces repellance.
128.	15% Total Solids 85% Butyl Acetate	75% Staybellite 10 22% Nitrocell. ½ sec. 3% Vulkamine	75	92			
129.	15% Total Solids 85% Butyl Acetate	75% Staybellite 10 22.8% Nitrocell. ½ sec. 2.2% Vulkamine	75	90			
130.	15% Total Solids 85% Butyl Acetate	75% Staybellite 10 23.6% Nitrocell. ½ sec. 1.4% Vulkamine	75	90			
131.	15% Total Solids 85% Butyl Acetate	75% Staybellite 10 25% Nitrocell. ½ sec. 0% Vulkamine	75	83			
132.	15% Total Solids 85% Butyl Acetate	70% Staybellite 10 27% Nitrocell. ½ 3% Vulkamine	75	90		Not good in spray test. Drops followed by too much fine spray.	
'33.	Total Solids 15% Butyl Acetate 85%	65% Staybellite 10 32% Nitrocell. ½ 3% Vulkamine	75	91		Looks good - same description as 128 above except little less fogging.	These films won't stick if staybellite much less than 60%.
34.	15% Total Solids 85% Butyl Acetate	60% Staybellite 10 37% Nitrocell. ½ 3% Vulkamine	75	90		Some tendency to peel.	
35.	15% Total Solids 85% Butyl Acetate	55% Staybellite 10 42% Nitrocell. ½ 3% Vulkamine	75	90		Peels.	
36.	15% Total Solids 85% Butyl Acetate	50% Staybellite 10 47% Nitrocell. ½ 3% Vulkamine	75	90		Peels.	
37.	15% Total Solids 85% Butyl Acetate	40% Staybellite 10 57% Nitrocell. ½ 3% Vulkamine	75	90		Peels.	

[redacted]

T A B L E III (Cont.)

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
138.	15% Solids 85% Butyl Acetate	90% Staybellite 10 3% Nitrocell. ½ sec. 7% Vulkamine	75	94		)These do not look good in)spray test. Drops do not)roll well. Too much mist.	
139.	15% Solids 85% Butyl Acetate	90% Staybellite 10 2% Nitrocell. 8% Vulkamine	75	90		)	
140.	15% Solids 85% Butyl Acetate	90% Staybellite 10 1% Nitrocell. 9% Vulkamine	75	90		)Looks good in spray test,)but not as good as 128.	
141.	15% Solids 85% Butyl Acetate	85% Staybellite 10 3% Nitrocell. 12% Vulkamine	75	90		)	
142.	15% Solids 85% Butyl Acetate	85% Staybellite 10 5% Nitrocell. 10% Vulkamine	75	90		)	
143.	15% Solids 85% Butyl Acetate	85% Staybellite 10 8% Nitrocell. 7% Vulkamine	75	90		)	
144.	15% Solids 85% Butyl Acetate	80% Staybellite 10 5% Nitrocell. ½ sec. 15% Vulkamine	75	95		Best yet in spray test. No change in 30 minutes; still looks good after 1 hour; vision fair after 2 hours.	
145.	15% Solids 85% Butyl Acetate	80% Staybellite 10 3% Nitrocell. ½ sec. 17% Vulkamine	75	93		Plates of this final group - 145 - 153, containing more than 15% Vulkamine and do not look good in spray test. Drops run in rivulets and interfere with vision.	
146.	15% Solids 85% Butyl Acetate	80% Staybellite 10 1% Nitrocell. ½ sec. 19% Vulkamine	75	87			
147.	15% Solids 85% Butyl Acetate	76% Staybellite 10 3% Nitrocell. ½ 21% Vulkamine	75	90			
148.	15% Solids 85% Butyl Acetate	75% Staybellite 10 2% Nitrocell. ½ sec. 23% Vulkamine	75	90			
149.	15% Solids 85% Butyl Acetate	75% Staybellite 10 0% Nitrocell. ½ sec. 25% Vulkamine	75	90			

[redacted]

TABLE III (Cont.)

Film No.	I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
150.	15% Solids 85% Butyl Acetate	75% Staybellite 10 5% Nitrocell. ½ 20% Vulkamine	75	94			
151.	15% Solids 85% Butyl Acetate	70% Staybellite 10 5% Nitrocell. ½ 25% Vulkamine	75	93			
152.	15% Solids 85% Butyl Acetate	65% Staybellite 10 5% Nitrocell. ½ 30% Vulkamine	75	95			
.	15% Solids 85% Butyl Acetate	70% Staybellite 10 15% Nitrocell. ½ 15% Vulkamine	75	94			

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MISCELLANEOUS FILMS

TABLE IV

I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
10% Shellac 90% Butyl Acetate	100% Shellac(Brown)	60	78	Glazed and fogged. Disintegrated after 2 hours.		Streaked on drying
9.7% Shellac 45% Butyl Acetate 45% Ethyl Alcohol .3% Ceresin	97% Shellac 3% Ceresin	60	97			Streaked badly on drying.
5% Shellac 50% Butyl Acetate .3% Ceresin 44.7% Ethyl Alcohol	97% Shellac 3% Ceresin	60	98	Disintegrates after one hour.		Film clear.
5% Shellac 50% Butyl Acetate .3% Paraffin 44.7%Ethyl Alcohol	97% Shellac 3% Paraffin	60	95	Glazed and disintegrated after 2 hours.		
9.7% F-10 .3% Shellac 70% Butyl Acetate 20% F-10 Thinner	97% F-10 3% Shellac	60	80	Angle too low - not immersed.		
5% F-10 4.7% Shellac .3% Ceresin 75% Butyl Acetate 15% F-10 Thinner	48% F-10 49% Shellac 3%Ceresin	60	100	Clouds and does not clear up.		
5% F-10 4.7%Shellac 3% Paraffin 75% Butyl Acetate 15% F-10 Thinner	48% F-10 49% Shellac 3% Ceresin	60	100	Clouds and does not clear up.		
9% F-10 1% Napthalene 67% Min. Spts. 23% F-10 Thinner	90% F-10 10% Napthalene	60	83	No different from F-10 alone. (see No. 1 film).		

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TABLE IV (Cont.)

I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
10% Poly Pale Resin 90% Butyl Acetate	100% Poly Pale Resin	60	70	Angle too low.		
9.7% Poly Pale Resin .3% Ceresin	97% Poly Pale Resin 3% Ceresin	60	100	Angle - 90, 4 hours after immersion. Angle - 80, six hours after immersion - no clouding.	No good in spray test. Peels off.	
	Dried F-10 film coated with Johnson's auto wax.		90			Surface bad. Rough film.
	Dried F-10 film coated with Crusader auto wax.		92			Surface bad. Rough film.
Motorboat varnish thinned with min. spts. 1 to 1.	100% solid constituents of motorboat varnish.	See Col.VII.	90	H_2O resistance good. Stood up overnight, but badly fogged. Does not clear up.		Drying time entirely too long - about 5 hours.
Motorboat varnish thinned with butyl acetate 1 : 1.	100% solid constituents of motorboat varnish.		90	H_2O resistance good. Stood up overnight, but badly fogged. Does not clear up.		Drying time entirely too long - about 5 hours.
Spar varnish unthinned.	100% solid constituents of Spar Varnish.		95	About same as above.		Drying time entirely too long - about 5 hours.
Floor varnish unthinned.	100% solid constituents of floor varnish.			Disintegrates shortly.		Drying time entirely too long - about5 hours.
DeKhotinsky cement. Solid (shellac and pine oil).	100% DeKhotinsky cement.		103	Clouds very badly.		Applied to warm glass Too insoluble for any solvent tried.
Swannee Lacquer	100% solid constituents of Swannee Lacquer.			Peels off.		Has a nitrocellulose base.
7% Swannee Lacquer 2.7% Staybellite 3% Vulkamine .3% Paraffin	Unknown			Behavior very similar to nitrocellulose-Vulkamine-Staybellite films.		

[redacted]

TABLE IV (Cont.)

I Composition of Original Mixture (% by Wt.)	II Composition of Dry Film (% by Wt.)	III Approx. Drying Time (Min.)	IV Contact Angle (Degrees)	V Behavior in Water Immersion Test	VI Behavior in Spray Test	VII Remarks
7% Shellac 3% Vulkamine 3% Staybellite .3% Paraffin 83.7% Butyl Acetate	57% Shellac 20% Vulkamine 20% Staybellite 3% Paraffin			Clouds - peels off shortly.		Incompatible.
5% Nitrocell. ½ 4% Vulkamine 1.5% Canada Balsam 89.5% Butyl Acetate	90% Nitrocell. ½ sec. 7% Vulkamine 3% Canada Balsam			Clouds and peels off shortly.		
5% Nitrocell. ½ 3% Vulkamine 3% Canada Balsam 89% Butyl Acetate	90% Nitrocell. ½ 5% Vulkamine 5% Canada Balsam			Clouds and peels off shortly.		
5% Nitrocell. ½ 2% Vulkamine 4% Canada Balsam 89% Butyl Acetate	90% Nitrocell. ½ sec. 3% Vulkamine 7% Canada Balsam			Clouds and peels off shortly.		
5% Nitrocell. ½ sec. .5% Vulkamine 5.5%Canada Balsam 89% Butyl Acetate	90% Nitrocell. ½ sec. 1% Vulkamine 9% Canada Balsam	80	86	Clouds and peels off.		

[redacted]

TABLE V

Description of Commercial Products

Name (As it appears in Tables II, III, IV)	Description
Cenco Tackiwax	Hydrocarbon wax manufactured by Central Scientific Company.
Flexalyn	Diethylene Glycol Diabietate. An adhesive resin manufactured by Hercules Powder Company.
F-10 Resin	An acryloid (methyl methacrylate) resin manufactured by Resinous Products and Chemical Company.
F-10 Thinner	A thinner added to F-10 by the manufacturers. 9 parts mineral spirits, 1 part solvesso.
Halowax	Halowax Corporation product.
Mineral Spirits	A paint thinner (mixture of hydrocarbon solvents). Federal Specification TT-T-291.
Nitrocellulose "C"	Nitrocellulose obtained from collodion by evaporation of the ether-alcohol solvent.
Nitrocellulose ¼ sec.	R.S. Nitrocellulose for general lacquer use. Manufactured by Hercules Powder Company.
Nitrocellulose ½ sec,	R.S. Nitrocellulose, higher viscosity than above.
Petroleum ether	Purified benzine B.P. 30 - 60°C. Federal Specification O-E-751.
Poly Pale Resin	A polymerized rosin of high melting point. Manufactured by Hercules Powder Company.
Staybelites	Esters of hydrogenated rosin. Staybelite 10 is the glycerol ester. Manufactured by Hercules Powder Company.
Solvesso #1	Solvent (mixture of aromatic and aliphatic hydrocarbons). Manufactured by Standard Oil Company of New Jersey.

 [redacted]

TABLE V (Cont.)

Description of Commercial Products

Name (As it appears in Tables II, III, IV)	Description
Vistanex	Isobutene polymer. A synthetic rubber. Manufactured by Standard Oil Company of New Jersey.
Vulkamine	Polymerized isobutylene - a plasticizer manufactured by General Dyestuff Corp.
Wood Rosin	H wood rosin. A refined wood rosin Grade H-U.S. Std. Rosin Types. Manufactured by Hercules Powder Co.

 [redacted]

TABLE VI

A - Composition of Rain Repellent N.R.L. 199

(1)	Acryloid F-10 (41.8% in F-10 Thinner)	13.35% by wt.
(2)	Paraffin wax	.29% " "
(3)	Staybelite 10	4.29% " "
(4)	Mineral Spirits	28.28% " "
(5)	Ethyl Acetate	53.79% " "

****

(1) Manufactured by Resinous Products & Chemical Company
(2) m. p. = 51.7°C.
(3) Glycerol ester of hydrogenated rosin, manufactured by Hercules Powder Company.
(4) Federal Specification #TT-T-291.
(5) Army-Navy Aeronautical Specification AN-O-E 758.

B - Composition of Cleaner #55

(1)	Ethyl acetate	50% by volume
(2)	Mineral Spirits	50% " "

****

(1) Army-Navy Aeronautical Specification AN-O-E 758.
(2) Federal Specification #TT-T-291.

 [redacted]

TABLE VII

Change of Contact Angle with Time of Immersion in Water

NRL No. 199		LORR		N.R.C. Paste	
Time (Hours)	Contact Angle (Degrees)	Time (Hours)	Contact Angle (Degrees)	Time (Hours)	Contact Angle (Degrees)
0	103	0	96	0	100
½	102	½	93	½	100
1	97	1	92	1	93
1½	94	1½	91	1½	93
2	94	2	91	2	93
2½	94	2½	91	2½	93
3	94	3	91	3	93
3½	94	3½	91	3½	93
4	94	4	91	4	93
4½	94	4½	91	4½	93
8	93	8	91	8	93
10	92	10	91	10	93
17	90	17	91	17	90
20	89	20	91	20	89
30	89	30	89	30	88
50	86	50	87	50	88
'eeled after 60 hours.		Peeled off after 60 hours.		Not peeled after 90 hours.	

[redacted]

TABLE VIII

Rolline Angle of Water Droplets on 199 Film

As A Function of Drying Time

Drying Time (Hours)	Rolling Angle (Degrees) *Drop Size #1	*Drop Size #2
½	13	26
1	10	20
2	8	19
3	9	18
4	9	19
5	9	18
6	8	18
8	9	19
12	9	17.5
16	9	17.5
20	8	18.5

*Drop size #1 - .124 g.
*Drop size #2 - .046 g.

Each value represents the average of 25 single determinations to the nearest ½ degree.

[redacted]

TABLE IX

Effect of Weatherometer Exposure on Rolling Angle

Film Tested	Drop Size (Grams)	Rolling Angle Before 10 Hour Exposure (Degrees)	Rolling Angle After 10 Hour Exposure (Degrees)
NRL 199	.012	35	Greater than 90
"	.027	19	60
"	.077	10	26
"	.106	6	24
Lorr	.012	39	Greater than 90
"	.027	20	65
"	.077	13	22
"	.106	5	23
N.R.C. Paste	.012	45	Not measurable -
"	.027	30	films broken down.
"	.077	28	
"	.106	25	

Values represent averages of 10 single determinations on duplicate samples.

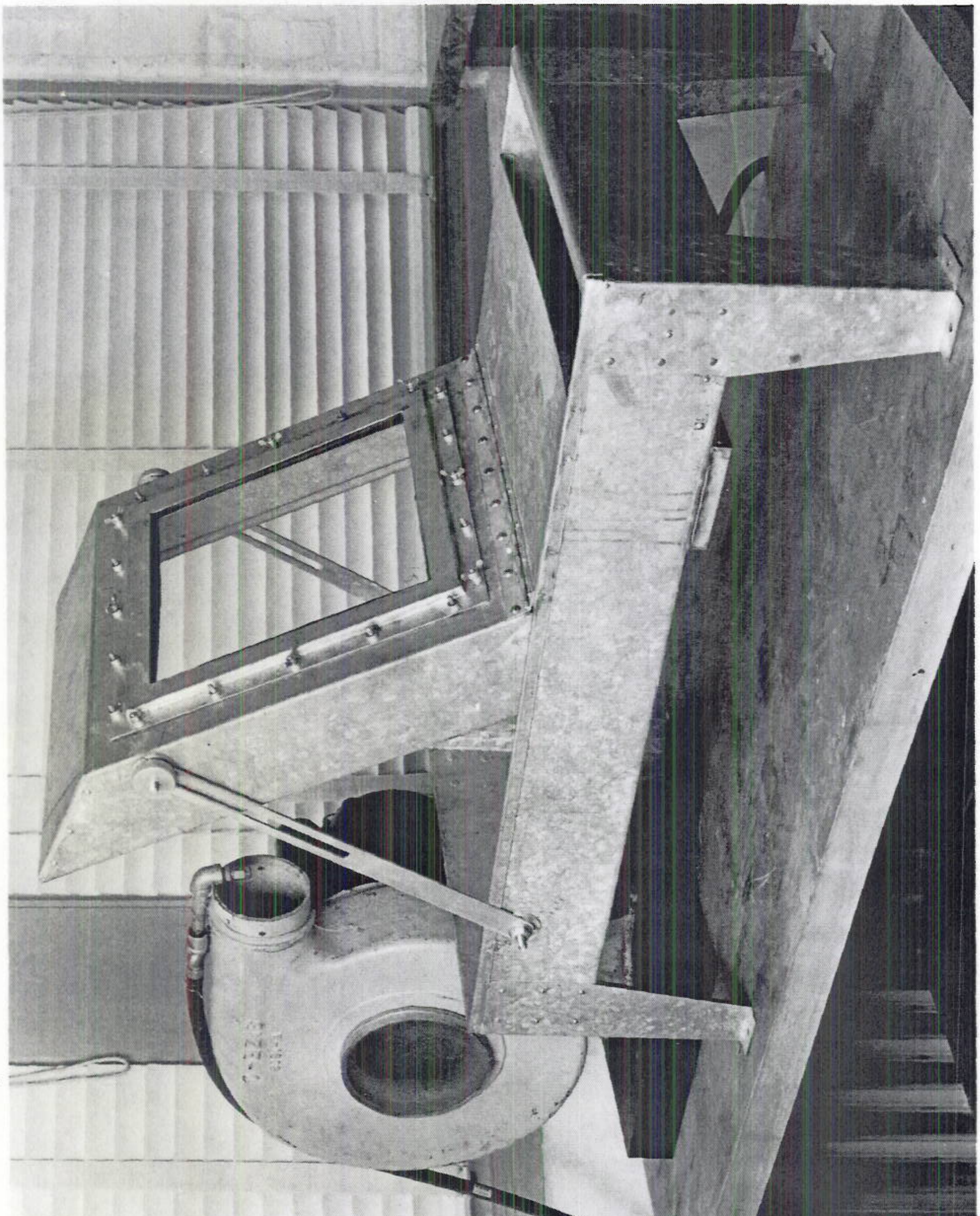
 [redacted]

TABLE X

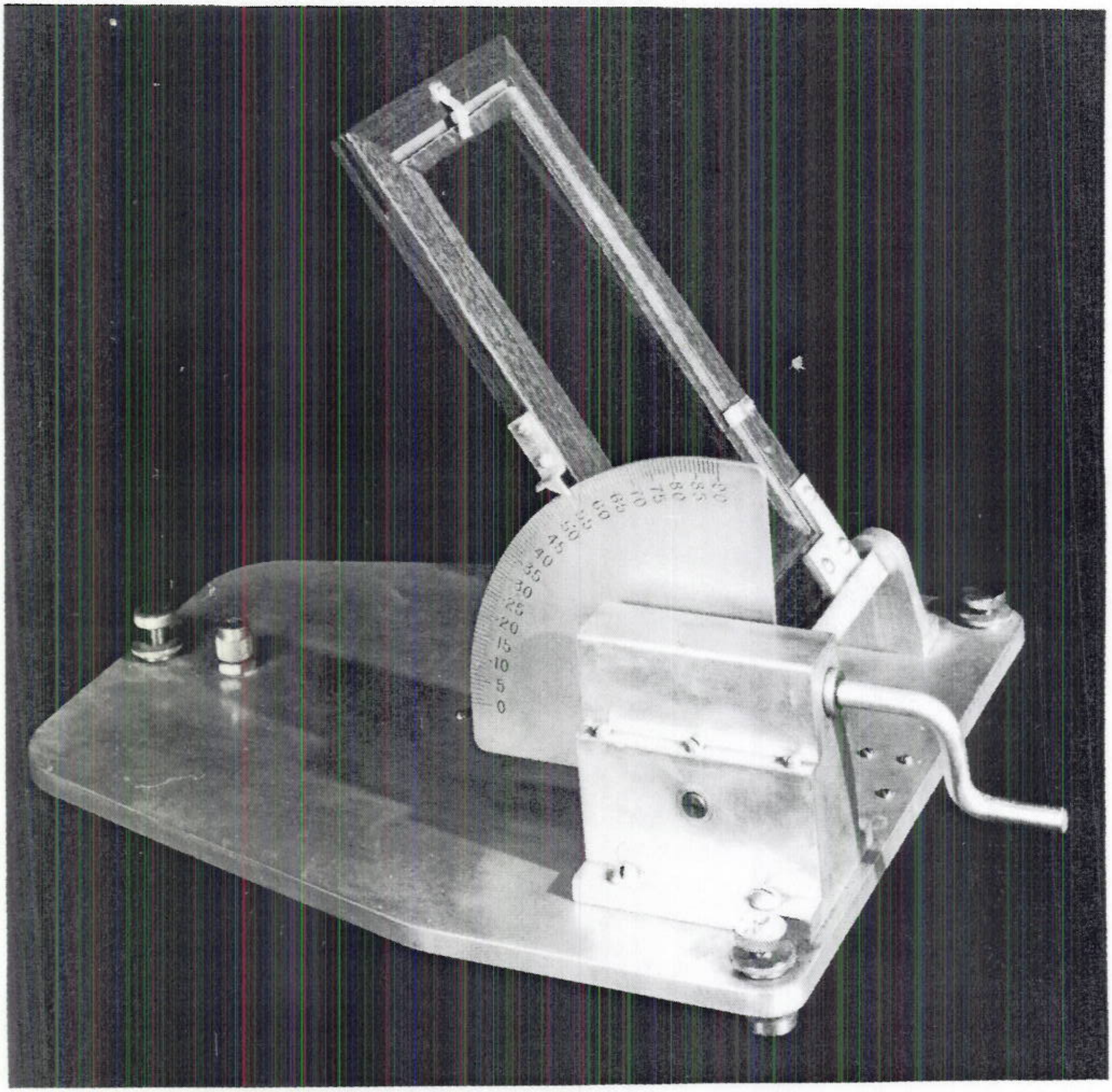
Summary of Comments on Commercial Rain Repellents

Name	Manufacturer	Opinions and Comments
Lorr Rain Repeller	Lorr Laboratories	There is little choice between a Lorr film and a 199 film in effectiveness and durability as judged by the spray test. Application of Lorr however is much more critical and it cannot be used below 40°F. 199 is effective down to 30°F. Field tests establish the definite superiority of 199 over Lorr.
N.R.C. "Anti Rain"	National Research Council	Three N.R.C. products have been tested: two paste types and one stick type. The pastes are easily applied, require no drying time, have good contact angles and outlast 199 and Lorr in the straight immersion test. Under spray however they fail completely within 5 minutes, lacking adhesion and durability. The stick type requires no drying, can be applied in rain and performs well when a satisfactory film is obtained. Only 10% of applications can be expected to yield acceptable films.
Drifilm	General Electric Company	The drifilms are methyl chlorosilicomethanes in CCl_4 as solvent. A film of this material applied by exposing glass to the vapors compares favorably with 199 in performance and durability. Such a method of application however is impractical for a windshield. Application of the liquid by means of a cloth gives films of low contact angle and fair durability.
Tedol	Admiralty Research Laboratory, Teddington, England.	This is a British repellent, same type as drifilm. It is corrosive and difficult to handle - not as effective as 199 but as durable. The material is hygroscopic and releases HCl vapors.

[redacted]



DECLASSIFIED [redacted] PLATE I



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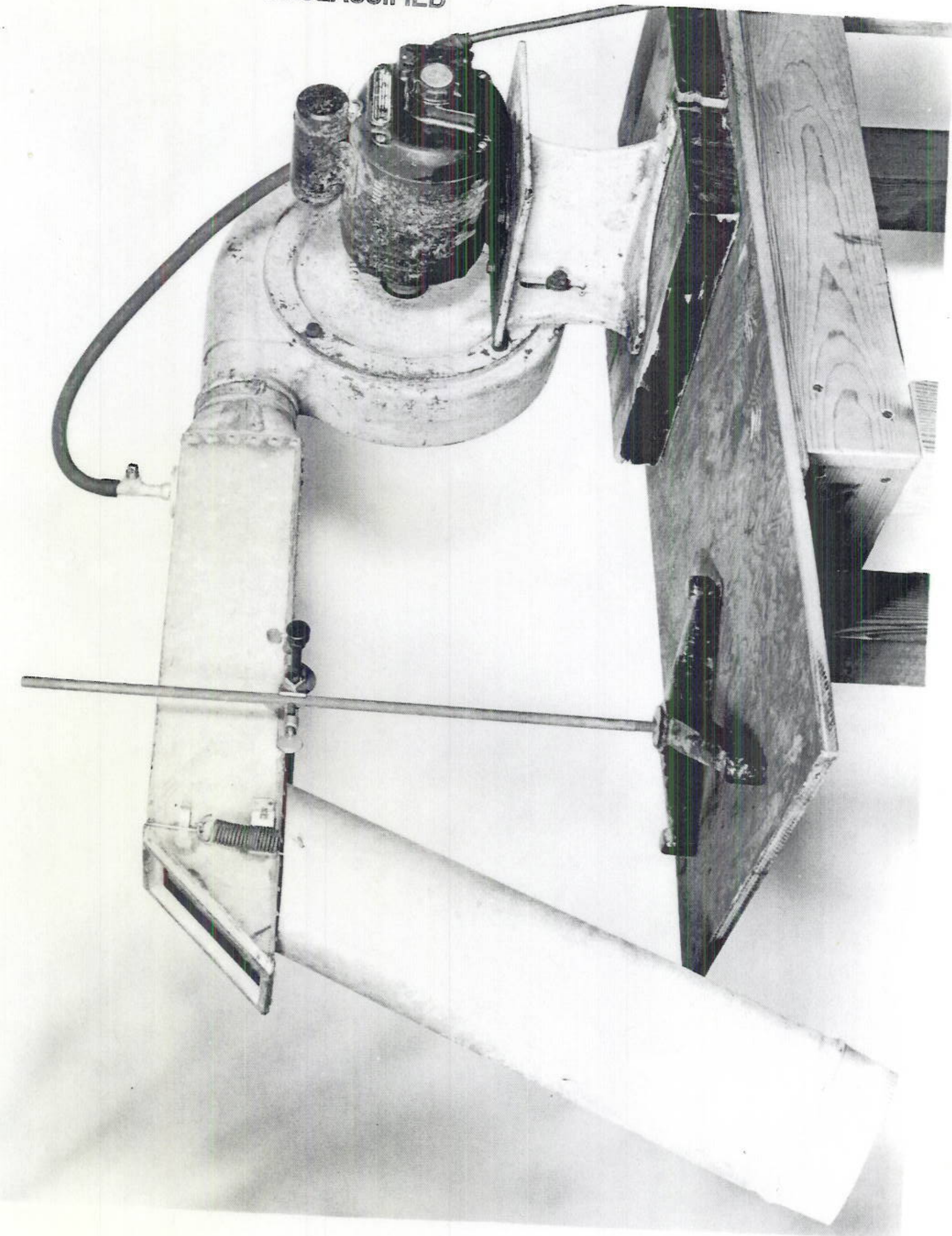

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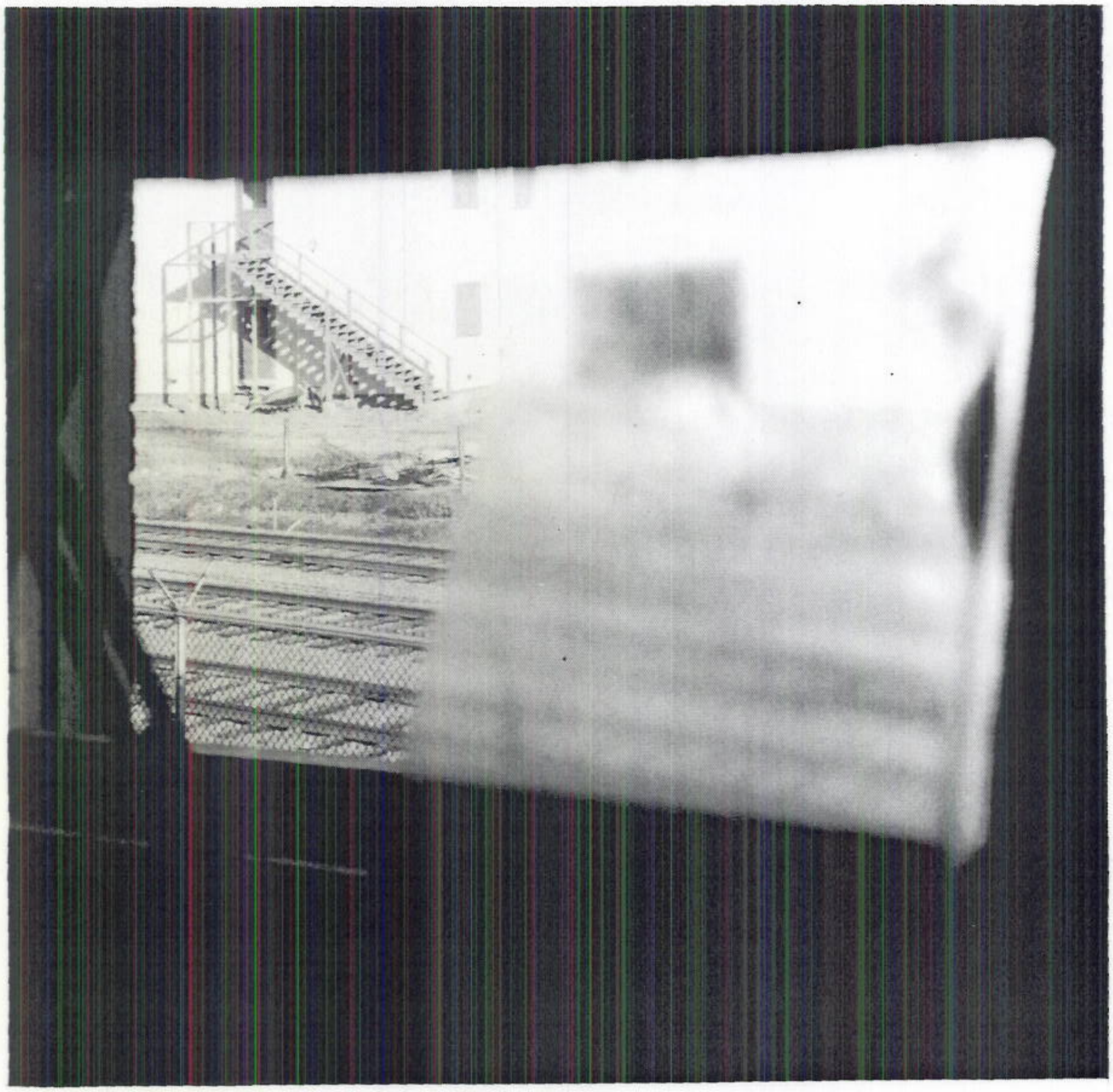

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PLATE 4

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DECLASSIFIED [redacted] PLATE 5

[redacted] PLATE 6

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PLATE 7

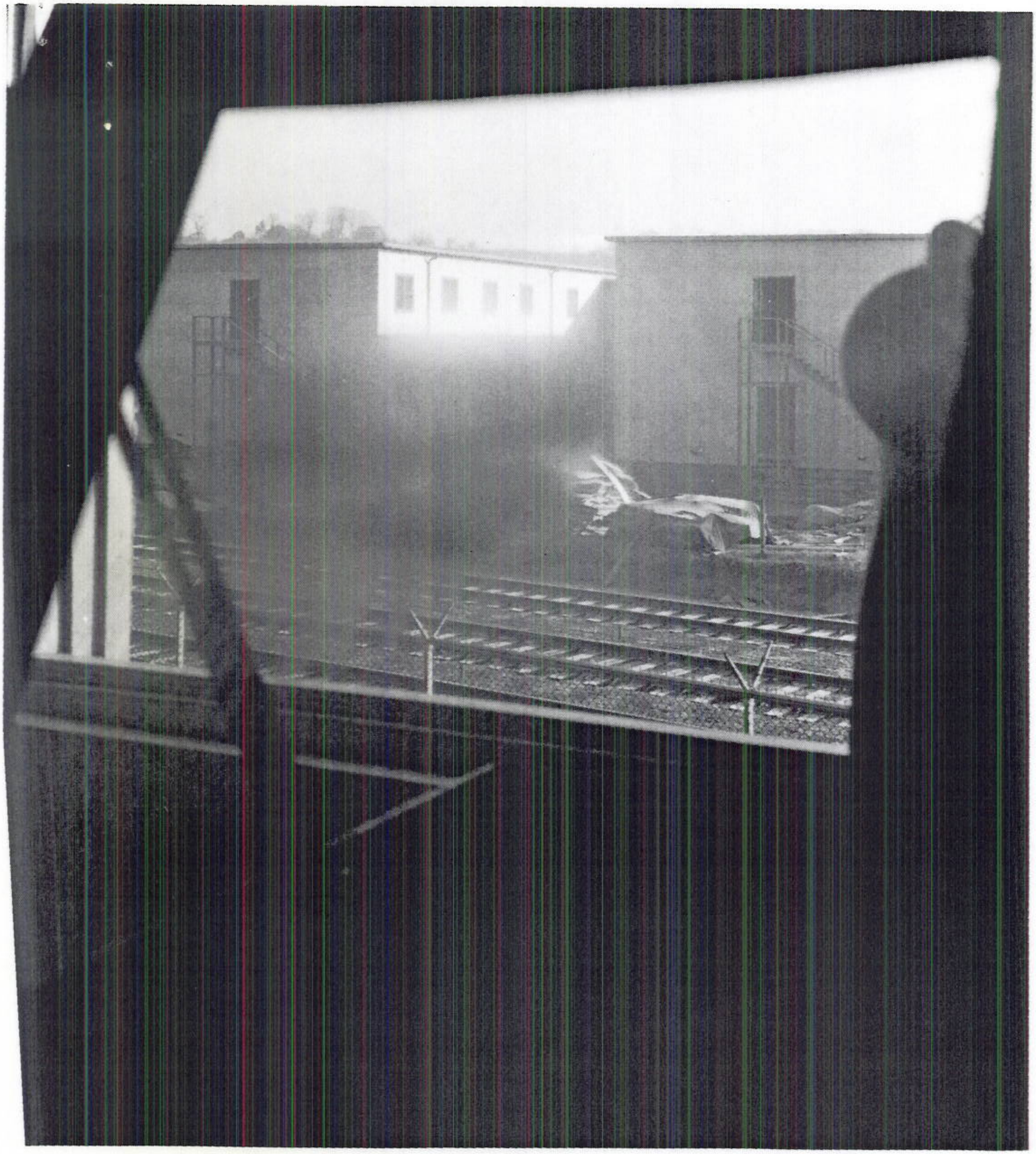

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PLATE 8

FIG. 1

FIG. 2

[redacted]

PLATE 9

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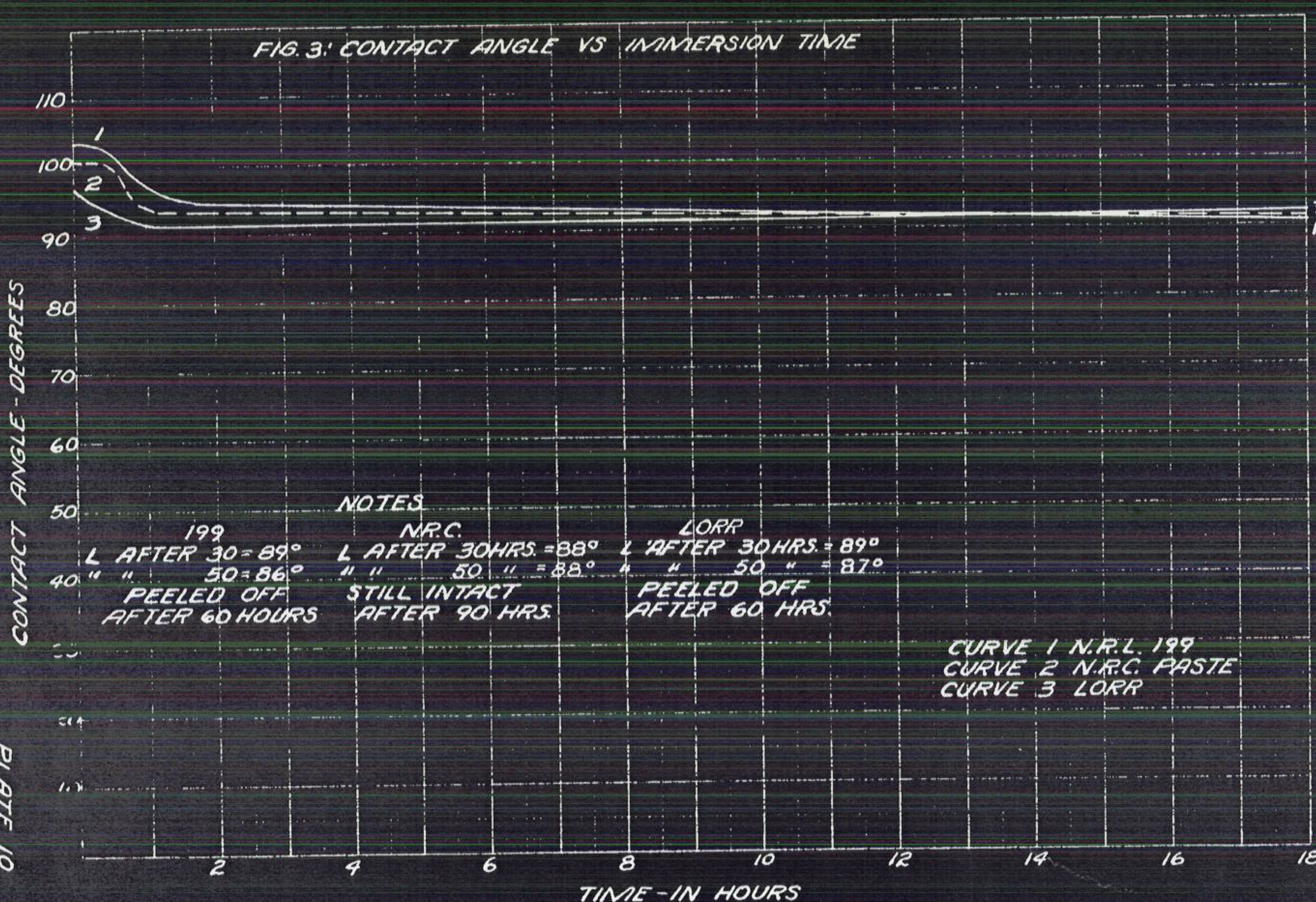
FIG. 3: CONTACT ANGLE VS IMMERSION TIME
CONTACT ANGLE - DEGREES
110
100
90
80
70
60
50
40
1
2
3
NOTES
199
L AFTER 30 = 89°
" " 50 = 86°
PEELED OFF
AFTER 60 HOURS
N.R.C.
L AFTER 30HRS. = 88°
" " 50 " = 88°
STILL INTACT
AFTER 90 HRS.
LORR
L AFTER 30 HRS. = 89°
" " 50 " = 87°
PEELED OFF
AFTER 60 HRS.
CURVE 1 N.R.L. 199
CURVE 2 N.R.C. PASTE
CURVE 3 LORR
2
4
6
8
10
12
14
16
18
TIME - IN HOURS

PLATE 10

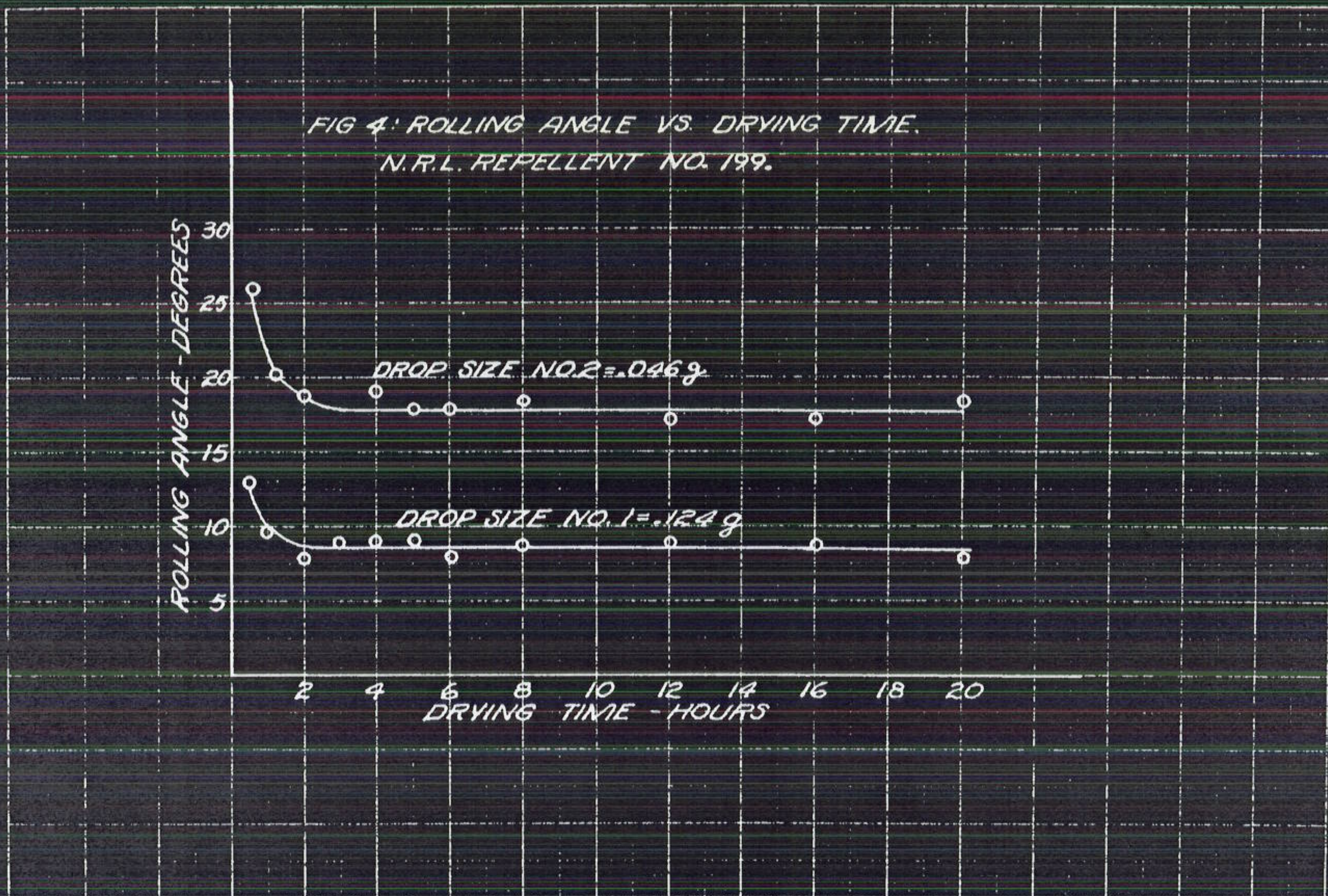

PLATE 11

[redacted]

APPENDIX 1

References

(1) S. Glasstone, "Textbook of Physical Chemistry", pp. 474-475.

(2) A. H. Nietz, J. Phys. Chem. 32,255 (1928.)

APPENDIX 2

Service Test Reports Herewith

(1) NAS, Norfolk, Va. ltr report NA8/F38-2(96-Ml) dated 18 March 1943

(2) NAS, Seattle, Wash., ltr to BuAer NA13/F48-2(2) dated 21 Mar.1943

(3) NAS, Alameda, Calif. ltr to BuAer NA27-30-Cs L5 dated 13 Apr.1943

(4) COMFAW6 ltr to ComFair Seattle, F48 Serial 52 dated 26 April 1943

[redacted]