March 30, 1944 NRL Report No. P-2257

NAVY DEPARTMENT

Report

on

REFLECTION-REDUCING COATINGS FOR OPTICAL SURFACES

Naval Research Laboratory
Anacostia Station
Washington, D. C.

NUMBER OF PAGES: Text - 10 Tables - 4 Plates - 9

AUTHORIZATION: BuOrd ltrs S71/NOrd 451 of 29 March 1943 and NP14 (IRE4e) of 11 February 1944.

Date of Test: October 1943 to March 1944

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Distribution:

BuOrd	(5)
BuShips	(2)
SONRD	(2)
BuAer	(2)

Classification changed from _____
to [redacted]
By authority of C-AL-8 ltr.
File No. C-188-48/47 Dated 4/11/47

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ABSTRACT

A study of the two-coat reflection-reducing film developed by Moulton and Tillyer of the American Optical Co. was made, particularly as regards the method of formation of these films and their properties. The preferred method of application was found to be the addition of portions of Solution #157c to glass disks while rotating at 4750 RPM followed by the application of Solution #50a (containing 5% butanol) at 2300-2500 RPM with a 15-minute baking at 100-105°C after each addition of solution. The films so formed were uniform, clear and hard and very efficient in reducing reflection. A typical disk of 1.525 index glass coated with this system gave a reflection of 0.14% per surface of incident light of 5500 A° wave length.

The use of various silicon esters for the formation of single-coat films was studied and found to give, at best, films capable of reducing reflection of light from glass of 1.52 index to about 50% of its original value. The films prepared from methyl and ethyl silicates were hard, clear, water-repellent, and were the most effective esters in reducing reflection.

Investigation of the use of various silicon compounds (Dow-Corning fluids 400 and 1300, dimethyl silicon dichloride and ethyl silicon trichloride) to produce non-wetting surfaces on reflection-reducing films showed dimethyl silicon dichloride to be the only one capable of giving good non-wetting surfaces without harming the reflection-reducing properties of the films.

INTRODUCTION

A. Authorization

1. This work was originally authorized by Bureau of Ordnance letter S71/NOrd 451 of 29 March 1943 and continued by Bureau of Ordnance letter NP14 (Re4e) of 11 February 1944.

B. Statement of Problem

2. The objects of this investigation were the production of reflection-reducing coatings on glass surfaces by chemical means and the treatment of the coatings to make them water repellent. If the first of these could be accomplished, the process for depositing low reflecting films on optical surfaces would be greatly simplified eliminating the high vacuum technique now generally used.

3. Certain requirements were placed on the coatings, namely, that they reflect only 1.5% per surface of the incident light at some wave length between 5000 and 6000 A°, and that they be clear and hard. These coatings could be produced in one or more applications of the same or different materials, the only limitation being that the result is reproducible following detailed instructions.

C. Known Facts Bearing on the Problem

4. This Laboratory has previously written three reports on reflection-reducing coatings[1,2,3]. Reference (1) deals with evaporated metallic fluoride films. It also contains the theory for the one-coat low reflecting coating. References (2) and (3) describe methods for the application of low reflecting films to transparent plastic surfaces and to glass surfaces. Films[2] produced on plastics by the evaporation of a suspension of finely divided silica are very efficient, but soft and somewhat cloudy. These may be hardened[3] by the addition of ethyl silicate to the suspension without impairing their effectiveness.

5. The American Optical Company has developed a two-coat system for producing coatings of very low reflectivity. This system consists of a high index coat containing a titanium compound and a low index coat of a silicon ester. The method is discussed fully in a patent application[4] of Moulton and Tillyer.

6. In general, the requirements for the type of coating described here are that it be 1/4 wave length thick optically for the incident light and that its index of refraction be the square root of that of the substrate.

7. The use of various organic silicon compounds for the production of water repellent films is well known. Liberal use was made of this fact in applying non-wetting coatings to the low-reflecting films. The hydrophobic properties of other adsorbed

monolayers have also been described in another Laboratory report[5].

EXPERIMENTAL

A. Apparatus

8. The apparatus used in the application of the two-coat reflection-reducing system consisted of a centrifuge whose head had been removed and replaced with a chuck suitable for holding glass disks up to three inches in diameter and for rotating them in a horizontal plane. The speed of rotation was controlled by a built-in rheostat and also by a "Variac" variable voltage transformer. The speed of rotation was measured by use of a "Strobotac".

9. The apparatus used to produce the single-coat films of the various silicon esters consisted of a small 2-RPM synchronous motor which withdrew the glass plates from a beaker of the coating material at a constant speed. The rate of withdrawal was varied by changing the size of the pulley attached to the shaft of the motor.

10. The reflection-reducing efficiency of the various films was determined by allowing parallel light which had passed through a daylight blue filter, Corning #5900, to fall on the coated plate and be reflected onto a "Photox" photoelectric cell at an angle as near to the normal as possible. The intensity of the reflected light was measured by the deflection of a sensitive galvanometer to which the photoelectric cell was attached.

11. The water repellent properties of various films was evaluated by measuring the contact angle of water with the film using a "contact goniometer"[5]. The apparatus consists of an eye-piece (magnifier) and two movable cross-hairs. One cross-hair is rotated until it is parallel to the base of the water droplet and the other rotated until it is tangent to the surface of the droplet at its point of contact with the film - the contact angle being the angle between the two cross-hairs and is read directly from an attached scale.

B. Materials

12. Glass plates, 1-5/8" x 3-3/8" x 1/8", 1.52 index.
Glass disks, 2" dia. x 3/16", 1.525 index, from Semon-Bache & Co., New York.
Solution #115 (Silica suspension) supplied by the American Optical Co.
Solution #50 prepared according to the procedure of the American Optical Co. Ethyl silicate in ethyl alcohol and ethyl acetate (see appendix).

Solution #155, $TiCl_4$ in ethyl alcohol as above, (see appendix).
Solution #157c, mixture of #50 and #155 as above, (see appendix).
Solution #50a, ethyl silicate in ethyl alcohol and ethyl acetate (see appendix for preparation.)
Ethyl silicate (commercial), Carbide & Carbon.
Silicon Tetrachloride, Niagara Smelting Corp.
Titanium Tetrachloride (technical), Eimer & Amend.
Dimethyl silicon dichloride (technical), H. S. Booth.
Ethyl silicon trichloride (technical), H. S. Booth.
Dow-Corning materials identified as follows:

a) DC fluid type 400, 1% Etc SiO in CCl_4
b) DC fluid type 1300, 1% LoMe in CCl_4

Common organic solvents.

C. Preparation of Films

13. Two-coat reflection-reducing systems were prepared by the following procedure: The clean glass disk to be coated was mounted in the chuck of the centrifuge and whirled at 4750 RPM. The surface was flushed with alcohol, whirled until dry and then blown free of dust and lint. A portion of Solution #157c (clear, containing no particles of dust, silica, etc.) was poured upon the center of the spinning disk and spun until dry. It was found that for best results the liquid (approximately 0.5 ml per surface for a 2" disk) should be added in one smooth motion and no extra drops allowed to fall on the surface. After both sides were coated the disk was further dried and the coating hardened by heating in an oven at 100-105°C. for 15 minutes. This film causes an increase in reflection of light from the surface. The glass disks were again mounted in the centrifuge and spun at 2200 RPM. A portion (approximately 0.5ml) of Solution #50 was added and the disk allowed to rotate until the film was dry. After the second surface was coated, the disk was heated at 100-105°C. for 15 minutes to complete the removal of the solvent. The films thus produced appeared deep blue or purple to daylight reflected normally from the surface, indicating the nearly complete extinction of reflection in the green-yellow portion of the spectrum. This low speed used caused an appreciable "edge effect", i.e., an area of high reflectivity was formed around the edges of the disk and extended inward for 1-2 mm.

14. A more satisfactory procedure for the deposition of the low index film was found to be the application of Solution #50a to the disks, as above, but using a higher speed of rotation; namely, 3000-3600 RPM. The "edge effect" was considerably lessened by this procedure. Best results were obtained through use

of Solution #50a to which 5% butanol had been added. For this solution a speed of rotation of 2300-2500 RPM was found to give films of desired thickness. The butanol caused a more even spread of the solution over the surface of the glass resulting in greater uniformity and fewer spots. Not enough edge reflection was present to be troublesome. The reflectivities of a series of plates prepared by the above technique and under normal Winter laboratory conditions (temperature, 75°F; relative humidity, 15%) are listed in Table I. Coatings applied under conditions of high humidity (temperature 75°F, relative humidity 50%) were effective in reducing reflection but were unsatisfactory because of excessive cloudiness.

15. Several single-coat reflection-reducing films were produced using various organic silicon esters; namely, methyl, ethyl, butyl and cellosolve silicates and trimethoxyethyl silicane.

16. A solution of methyl silicate in methanol was prepared by the slow addition of 15 ml of silicon tetrachloride to 50 ml of absolute methanol with cooling. The reaction mixture was allowed to stand two hours and then various quantities of it were added to 50 ml of acetone and 5 ml of butanol. (These acetone solutions decompose on standing.) These solutions were used for the application of films to glass plates of 1.52 index by the withdrawal of the plates from the solutions at various rates. Evaporation of the solvent left a homogeneous film having reflection-reducing properties, clarity, hardness and water-repellency especially after baking at 100°C for one hour. Plate 1 shows graphically the effects of the concentration and the rate of withdrawal on the effectiveness of these films in reducing reflection. The minima of the curves probably indicate that the maximum efficiency of this type of film is in the range of 52% reduction of reflection when applied to glass of 1.52 index. A series of plates was treated as above but in an atmosphere of 48% relative humidity. There was no detectable difference between films prepared under these conditions and those prepared under normal laboratory conditions (relative humidity 10-20%).

17. Plates were coated with ethyl silicate films by withdrawal at various rates from a solution of the following composition: acetone, 50 ml; butanol 5 ml; ethyl silicate, 5 ml; hydrochloric acid, 1 ml. The plates were baked 1.5 hours at 90°C. The effect of the rate of withdrawal on the efficiency of the films is indicated in Plate 2. The effect of additional coatings applied at a withdrawal rate of 11.9 cm/min is indicated in Plate 3. This procedure was found to give inconsistent results as may be noted from the lack of smoothness in the curve. This inconsistency was found on subsequent runs. Plate 4 shows the reflectivity toward light of various wave lengths of a plate coated with an ethyl silicate film. The film shows a fairly uniform reflectivity 55-60% over the range of 4000-6250 A°.

18. A solution of butyl silicate in butanol was prepared by the slow addition of 10 ml of silicon tetrachloride to 50 ml of redistilled butanol and allowing the mixture to stand for two hours. Various amounts of this solution were added to 50 ml portions of acetone and applied to glass plates using the procedure outlined above for methyl silicate films. Plate 5 shows the relation of the reflection-reducing properties of this film to the concentrations used.

19. A cellosolve silicate solution was prepared by the addition of 10 ml of silicon tetrachloride to 50 ml of technical ethyl cellosolve and allowing the mixture to stand one hour. Portions of this solution were added to 50 ml of acetone and 5 ml of butanol and applied to glass plates as above. The relation of concentration to reflection-reducing efficiency is shown in Plate 6.

20. A solution of trimethoxyethyl silicane was prepared by adding 10 ml of ethyl silicon trichloride to 50 ml of methanol and allowing to stand for two hours. This solution was applied to glass plates in a manner similar to the cellosolve silicate procedure. Plate 7 shows the relation between the reflection-reducing efficiency and the concentration.

D. Application of Non-Wetting Films

21. The ability of several reagents to produce non-wetting films was tested by applying them to a wettable reflection-reducing film and then measuring its non-wetting properties and reflection-reducing efficiency. The wettable reflection-reducing film was prepared by withdrawing small glass plates at 11.9 cm/min from a solution of the following composition.

Cellosolve	46 ml
Butanol	35 ml
Ethanol (95%)	340 ml
Solution #115	92 ml
Ethyl Silicate	5 & 10 ml
Hydrochloric Acid	1 ml

The plates were cut in half, one half kept as a control and the other treated with the various solutions capable of producing non-wetting films. The results are listed in Table II.

22. Small quantities of the Dow-Corning fluids (DC 400 and DC 1300) were poured over the surfaces of the above coated plates after they had been rinsed with acetone. The fluids were allowed to drain while held in a vertical position and finally baked for four hours at 150°C. The above process was repeated using dilute solutions of the DC fluids in carbon tetrachloride.

In all cases a sticky residue remained after baking. This was removed by rinsing with ethyl alcohol. The plates so treated were not wet by water but were no longer effective in reducing the reflection of light from the surface. (See Table II.)

23. A solution of dimethyl silicon dichloride in tetrachloroethylene was applied to the coated glass plates by spraying using a hand atomizer. After drying for five minutes at room temperature, the plates were rinsed with alochol. The films were not wet and were still very effective in reducing-reflection.

24. Ethyl silicon trichloride in tetrachloroethylene solution was applied to the plates by spraying and treated as above. The films were non-wetting to some extent but their reflection-reducing efficiency was decreased.

E. Testing of Films

25. A graph of the reflectivity of a disk treated with the two-coat system of reflection-reducing films toward light of various wave lengths is shown in Plate 8. The reflectivity toward light of wave lengths 4900-6500 A° is less than 20% of that of an uncoated piece of glass of the same kind and reached a minimum of 3.4% (0.14% of the incident light per surface) at 5500A°. In Plate 9 is shown a comparison of the transmission of a 5-piece system (ten surfaces) of coated glass disks with a similar system of uncoated disks for various wave lengths of incident light. The maximum efficiency of the coated system is in the range 5250-5500 A° where nearly 50% more light is transmitted than by the uncoated system.

26. The two-coat films are fairly hard and glass so coated may be handled in much the same way as uncoated glass. The films may be scratched by rubbing with metal, grit, or other abrasive material, but may be safely polished by rubbing with a clean cloth. These films are non-wetting, the contact angle of water with the film being 40-50°. Long immersion (one week) in water causes a decrease in the non-wetting properties of the films and also a shift in color from blue to brown with a slight decrease in the reflection-reducing properties of the film. These films are also extremely grease resistant; grease spots do not tend to spread over the surface and may be readily removed by wiping with a clean cloth.

27. A comparison of the properties of the single-coat films produced from the various silicate esters is given in Table III. For the alkyl silicate esters (methyl, ethyl, butyl) the effectiveness of the films in reducing reflection decreases with an increase in the size of the alkyl group, but the water-repellency as measured by the contact angle of water with the film, increases with an increase in chain length of the alkyl groups.

All of the films are clear but those of longer chain length are soft and easily made cloudy by rubbing. The films prepared from methyl and ethyl silicate are very hard and very clear.

28. Typical plates covered with films of methyl, ethyl and butyl silicate were heated in an oven at 200°C for one week in an accelerated weathering test. The reflection-reducing efficiency was greatly decreased in each case but the plates were still clear and non-wetting. An ethyl silicate film on glass was immersed in distilled water at room temperature for 40 hours. At the end of this time the plate was partially wet by water and the film was brown rather than gray-blue but the efficiency in reducing reflection was essentially unchanged.

29. Relatively thick films of methyl silicate, ethyl silicate, butyl silicate, trimethoxyethyl silicane and Solution #155 suitable for infra-red absorption studies were prepared by pouring small quantities of the solutions on to rock salt plates and drying in an oven at 105-110°C for several hours. The infra-red absorption spectra were measured with a rock salt prism spectrometer. The results are indicated in Table IV. Absorption bands at 3μ arise from water or hydroxyl groups and those at 6μ are caused by water alone. Groups containing a carbon-hydrogen bond (C-H) have absorption bands at 3.45μ, while $-CH_2$ and $-CH_3$ groups absorb at 6.8 and 7.2μ. Silicates absorb at 3, 8.4, 8.8, 9.7, 10, 10.5, and 11μ, the number of absorption bands depending on the structure of the silicate. Titanate bands arise at about 13μ. The results listed in Table IV under "Weak Bands" are somewhat doubtful as the absorption was extremely weak. They would indicate that the surface was rich in C-H groups of some sort but that not many were present in the body of the substance. This rich C-H surface substantiates conclusions based upon measurements of the contact angle of water with these surfaces.

30. X-ray studies* of an ethyl silicate coating show no lines of crystalline quartz. The lines would be detectable if the particle size were greater than 50 A° and the amount of quartz present greater than 1%. The percentage detectable would be lower with increasing particle size. In the case of the high index coating no lines of crystalline titanium oxide appear until after the film has been flamed. During the flaming the coating first chars and then becomes white. After this moderate heat treatment anatase lines appear. The sharpness of the lines indicate that fairly large particles are formed immediately.

DISCUSSION OF RESULTS

31. The reflectivities of a series of plates prepared according to the procedure in paragraph 14 (using Solution #50a containing 5% butanol to produce the second film) are listed in

*These studies were made by Dr. H. Friedman of the Physical Optics Division.

Table I. The values obtained indicate that by using a standardized procedure it is possible to produce efficient and nearly identical films consistently. It is to be expected, however, that to produce similar films from different batches of solution, the speeds of rotation must be adjusted to the specific solution used or the concentration adjusted to fit a given speed since there naturally will be small variations in effective concentration due to differences in solution age, concentration, temperature of aging, etc. Presumably the rate of change of concentration of these solutions will decrease as the solution ages and thus after a given period of aging (one month) will undergo only slight changes on further standing. It is therefore suggested that the speeds be adjusted to the solution being used.

32. It has been postulated that the single-coat reflection-reducing films produced on glass by the above processes consist of thin silica (SiO_2) coatings. However, there is much evidence that would indicate this to be untrue. These films are not wet by water while silica films are very readily wet. Variation of the alkyl group present in the silicate ester causes a variation in the wettability of the films, as is evidenced by a change in the contact angle of water with the films, and also a change in the efficiency of the films in regard to reduction of reflection. Thus it would appear that some of the alkyl groups are still present in the film - possibly as a condensed silicate ester. The presence of alkyl groups in the butyl silicate film has been definitely established through infra-red absorption studies. Confirmation, however, is lacking in the case of methyl and ethyl silicate possibly because of the thinness of the films. Infra-red studies also do not show strong evidence for alkyl groups in the trimethoxyethyl silicane film, even though the structure of this compound precludes the possibility of the loss of all of its alkyl groups.

33. The presence of organic groups in the high index coating ($TiCl_4$ in ethylalcohol) is indicated by the high contact angle of water with the surface, and the fact that the material chars on flaming. The absence of an X-ray pattern until after flaming indicates that the titanium is in a non-crystalline compound on the surface.

34. The Dow-Corning fluids gave films having very good non-wetting properties but which also partially destroyed the effectiveness of the reflection-reducing film. The more dilute solutions did not harm the original film to the same extent as the concentrated (1%) solution but were also less effective in producing a non-wetting surface. These fluids were found to be unsatisfactory for giving a non-wetting surface to a reflection-reducing coating. The films produced from ethyl silicon trichloride were fairly non-wetting but decreased the effectiveness

of the reflection-reducing film. The films produced by the treatment with solutions of dimethyl silicon dichloride in tetrachloroethylene were found to give the best results. These films give good water repellency to the reflection-reducing films but have very little effect on the other properties.

CONCLUSIONS AND RECOMMENDATIONS

35. The two-coat system of reflection-reducing films developed by Moulton and Tillyer of the American Optical Company was investigated and found to produce satisfactory reflection-reducing films.

36. Modification of Solution #50 by its preparation in a more concentrated form and also by the addition of 5% butanol before applying to the glass is advocated since more uniform films are thus produced and the "edge effect" is less bothersome.

37. It is suggested that the speed of rotation required to produce efficient films by the above technique may vary with the particular batch of solution used and that it be adjusted for each batch by the preparation of a few test plates.

38. It is recommended that this work be continued and that other methods of application of these films be studied. The method of application by a "withdrawing" procedure should be studied so that films may be applied to objects incapable of being treated by the spinning technique and also that the "edge effect" produced by spinning may be avoided.

39. The application of this two-coat system of reflection-reducing films to materials other than glass; for example, plastic lenses, should be investigated.

40. Films produced from single-coatings of silicate esters have a maximum reflection-reducing efficiency of about 50% on 1.52 index glass. The films produced by methyl and ethyl silicate are the only ones having properties making them worthy of further investigation and possibly then only in conjunction with a high index film.

41. The composition of the films produced by solutions of these esters - and presumably from Solution #50 of the American Optical Company - is still not definitely known, although most evidence points toward the formation of a partially hydrolyzed polymerized silicate ester.

42. The use of Dow-Corning fluids 400 and 1300 and of ethyl silicon trichloride for the application of a non-wetting surface to reflection-reducing films is discouraged because of the adverse effect of the materials on the efficiencies of the films.

43. The use of dimethyl silicon dichloride for producing a non-wetting surface on these films is advocated.

TABLE I

REFLECTIVITY OF DISKS COATED WITH A TWO-COAT REFLECTION-REDUCING FILM*

Disk No.	Reflectivity to White Light	Reflectivity to Daylight Blue	Reflection Surface
1	21%	23%	0.92%
2	20	21	0.84
3	19	21	0.84
4	20	21	0.84
5	20	21	0.84
6	21	21	0.84
7	18	16	0.64

*All films were hard and clear but a little too thick to be the most efficient type.

TABLE II

EVALUATION OF VARIOUS NON-WETTING FILMS APPLIED TO REFLECTION-REDUCING FILMS

Reagent	Concentration	No. Coats	Reflectivity of Film Before	Reflectivity of Film After	Non-Wetting Properties
DC 1300	Undiluted	1	10%	66%	Good
	Undiluted	1	8%	61%	Good
	1:9 CCl_4	1	27	61	Good
	1:25 CCl_4	1	34	51	Good
	1:100 CCl_4	1	34	40	Poor
DC 400	Undiluted	1	10	52	Good
	1:9 CCl_4	1	35	59	Good
	1:25 CCl_4	1	36	45	Good
	1:100 CCl_4	1	34	38	Fair
$EtSiCl_3$	1:20 C_2Cl_4	2	9	30	Fair
	"	4	7-1/2	21	Poor
Me_2SiCl_2	1:15 C_2Cl_4	4	8	9	Good
	"	4	8	9	Good
	1:6.4 C_2Cl_4	2	34	35	Good
	"	2	37	34	Good
	"	1	37	34	Good
	"	2	34	34	Good
	"	1	34	29	Good

TABLE III

COMPARISON OF PROPERTIES OF SINGLE-COAT REFLECTION-REDUCING FILMS PREPARED FROM VARIOUS SILICON ESTERS

	Methyl Silicate	Ethyl Silicate	Butyl Silicate	Cellosolve Silicate	Trimethoxy-ethyl Silicane
Reflectivity	48	58	61	64	60
Hardness	v. hard	v. hard	softer	soft	soft
Clarity	v. clear	v. clear	fairly clear	fairly clear	clear
Non-wetting Properties	+	+	+	+	+
Contact Angle*	55°	60°	70°	45°	85°

*Films had weathered at room temperature for about 3 weeks.

TABLE IV

INFRA-RED ABSORPTION BANDS OF FILMS*

Source of Film	Predominant Bands μ	Weak Bands μ
Methyl silicate	3,6,8.8,9.7,10.5 HOH, Silicate	3.4,6.8,7.2 C-H,-CH_2,-CH_3
Ethyl silicate	3,6,8.8,9.7,10.5 HOH, Silicate	3.4,6.8,7.2 C-H,-CH_2,-CH_3
Trimethoxyethyl silicane	3,4.5,8.8,9.7 -OH, Silicate	3.4,6.8,7.2 C-H,-CH_2,-CH_3
Butyl Silicate	3,8.8,9.7,11,3.4,6.8,7.2 Silicate, -CH, -CH_2,-CH_3	
Solution #50 (Ethyl Silicate)	3,8.8,9.7,10.5 Silicate	6 HOH
Solution #155 (Ethyl Titanate)	3,6,13 HOH, Titanate	

*Data by Mr. P. S. Smith of Physical Optics Division

APPENDIX

PREPARATION OF SOLUTIONS

Solution #155 is prepared by the slow careful addition of ten parts by weight of titanium tetrachloride to ninety parts of 190 proof ethyl alcohol and diluting the resulting solution with an equal volume of 190 proof ethyl alcohol.

Solution #50 is prepared by mixing the following components in the proportions indicated and allowing to stand at least one week:

Ethyl alcohol (190 proof)	45	parts	by	weight
Ethyl silicate	5	"	"	"
Ethyl acetate	45	"	"	"
Hydrochloric acid (d 1.17)	5	"	"	"

Solution #50a is a modification of Solution #50 and is prepared by adding a double portion of ethyl silicate to the above solution. It gives a solution that is essentially twice as concentrated as Solution #50 in regard to film-forming material.

Solution #157c is prepared according to the directions of Moulton and Tillyer[4] by mixing equal parts by weight of Solutions #155 and #50. It is used to produce films having a refractive index somewhat higher than that of glass but less than that produced by Solution #155 alone.

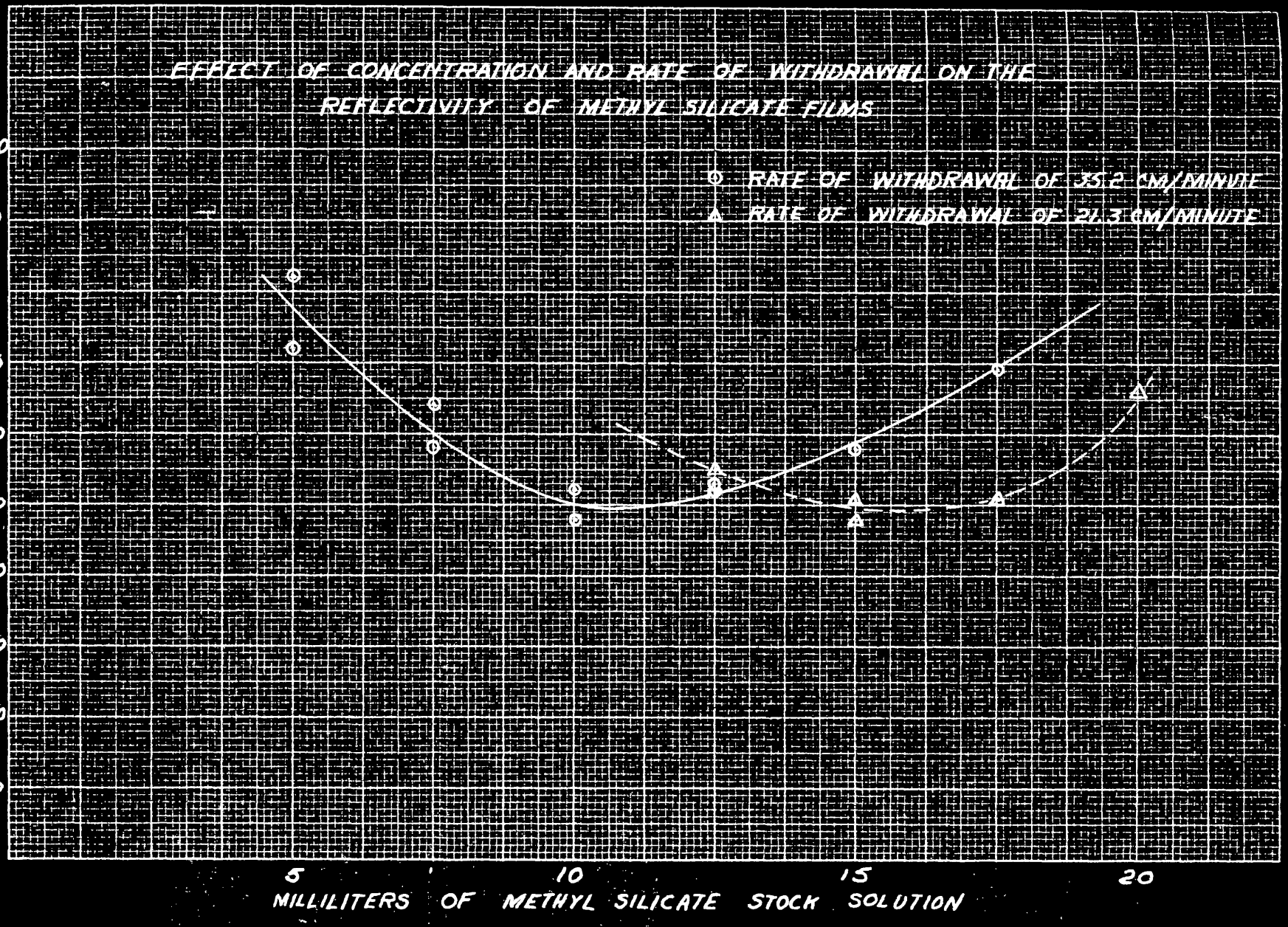
EFFECT OF CONCENTRATION AND RATE OF WITHDRAWAL ON THE REFLECTIVITY OF METHYL SILICATE FILMS
RATE OF WITHDRAWAL OF 35.2 CM/MINUTE
RATE OF WITHDRAWAL OF 21.3 CM/MINUTE
REFLECTIVITY %
100
90
80
70
60
50
40
30
20
10
5
10
15
20
MILLILITERS OF METHYL SILICATE STOCK SOLUTION

PLATE I

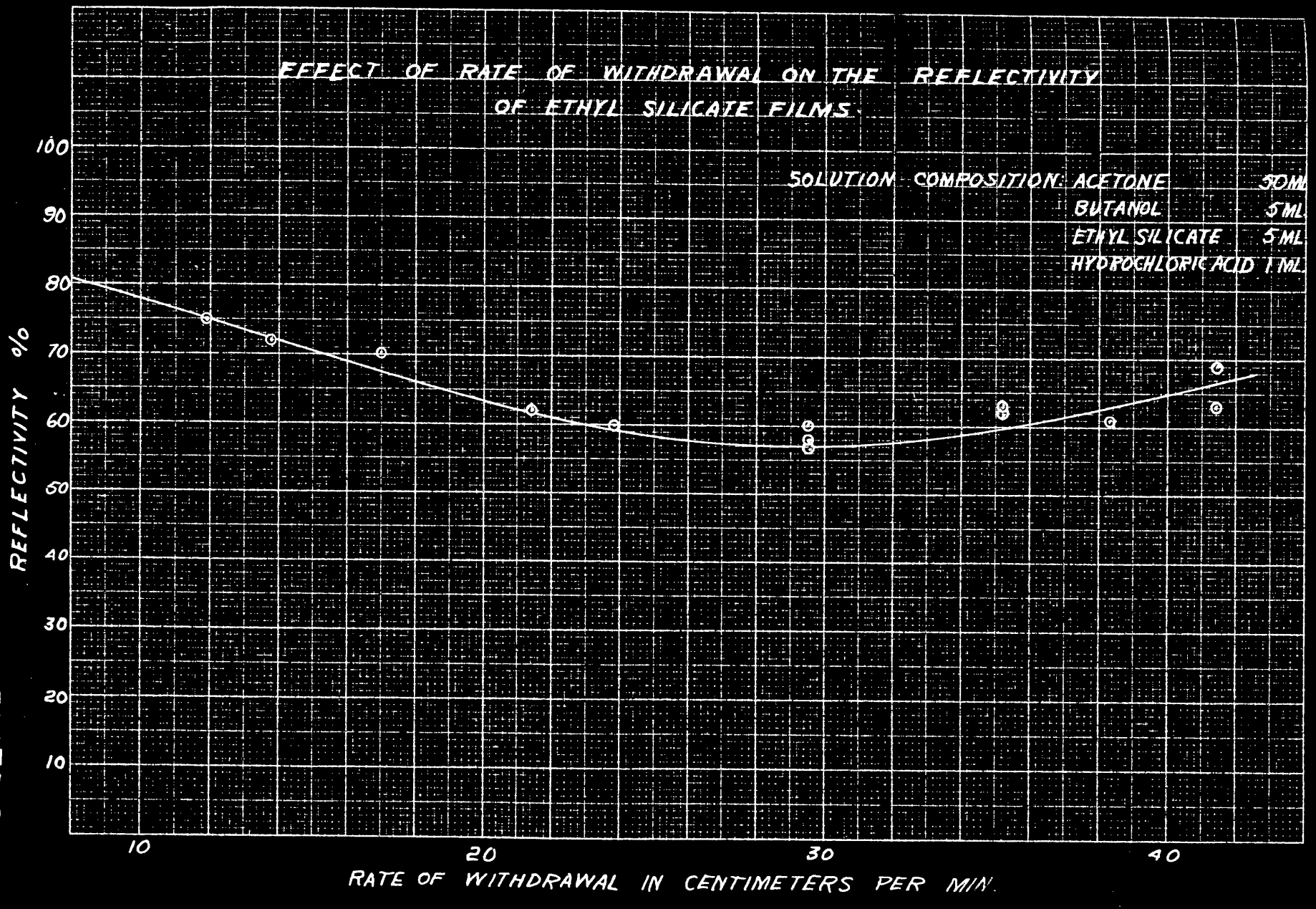
EFFECT OF RATE OF WITHDRAWAL ON THE REFLECTIVITY
OF ETHYL SILICATE FILMS.
SOLUTION COMPOSITION: ACETONE 50ML
BUTANOL 5ML
ETHYL SILICATE 5ML
HYDROCHLORIC ACID 1ML
REFLECTIVITY %
100
90
80
70
60
50
40
30
20
10
10
20
30
40
RATE OF WITHDRAWAL IN CENTIMETERS PER MIN.

PLATE 2

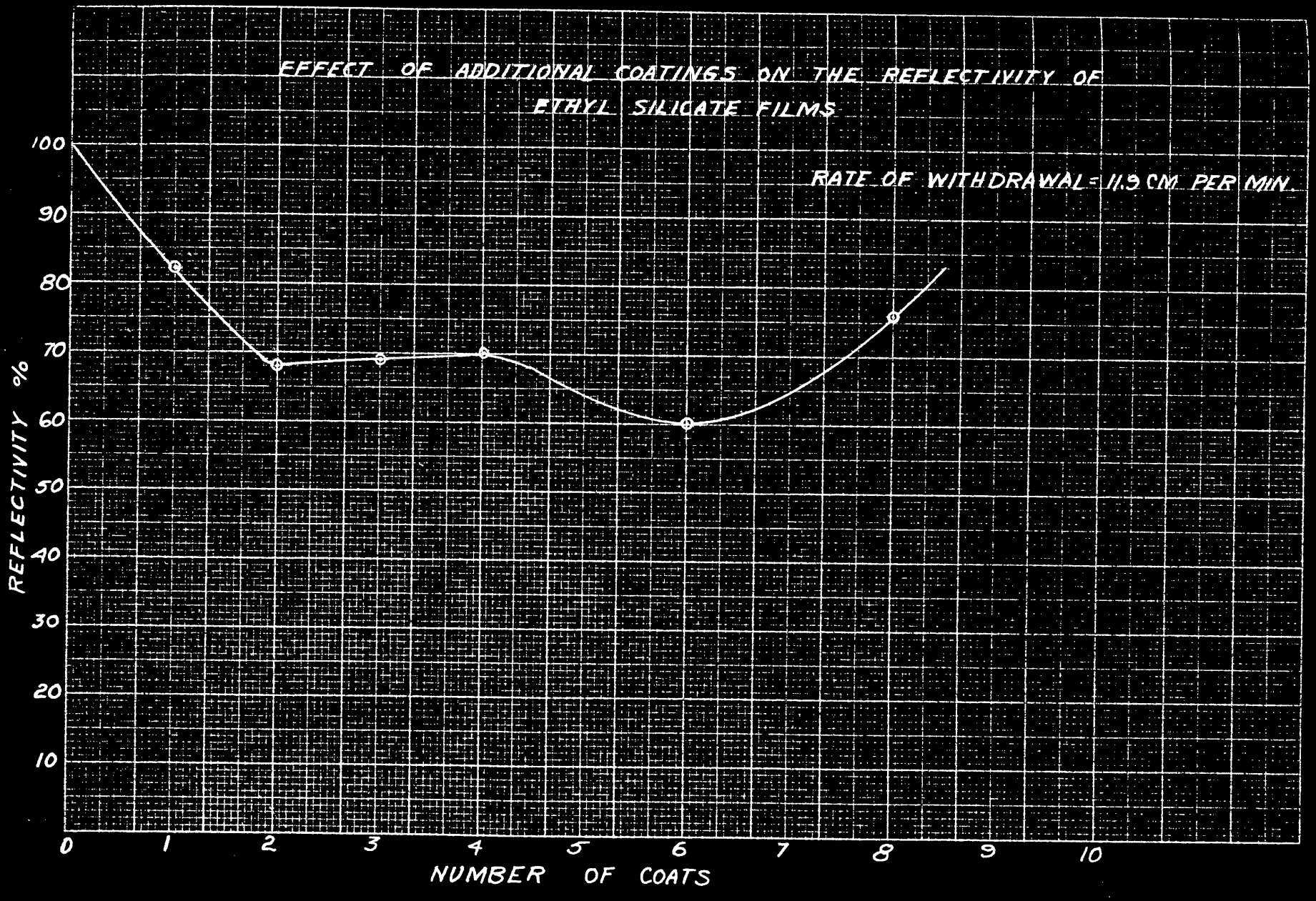
EFFECT OF ADDITIONAL COATINGS ON THE REFLECTIVITY OF
ETHYL SILICATE FILMS
RATE OF WITHDRAWAL = 11.9 CM. PER MIN.
REFLECTIVITY %
100
90
80
70
60
50
40
30
20
10
0
1
2
3
4
5
6
7
8
9
10
NUMBER OF COATS

PLATE 3

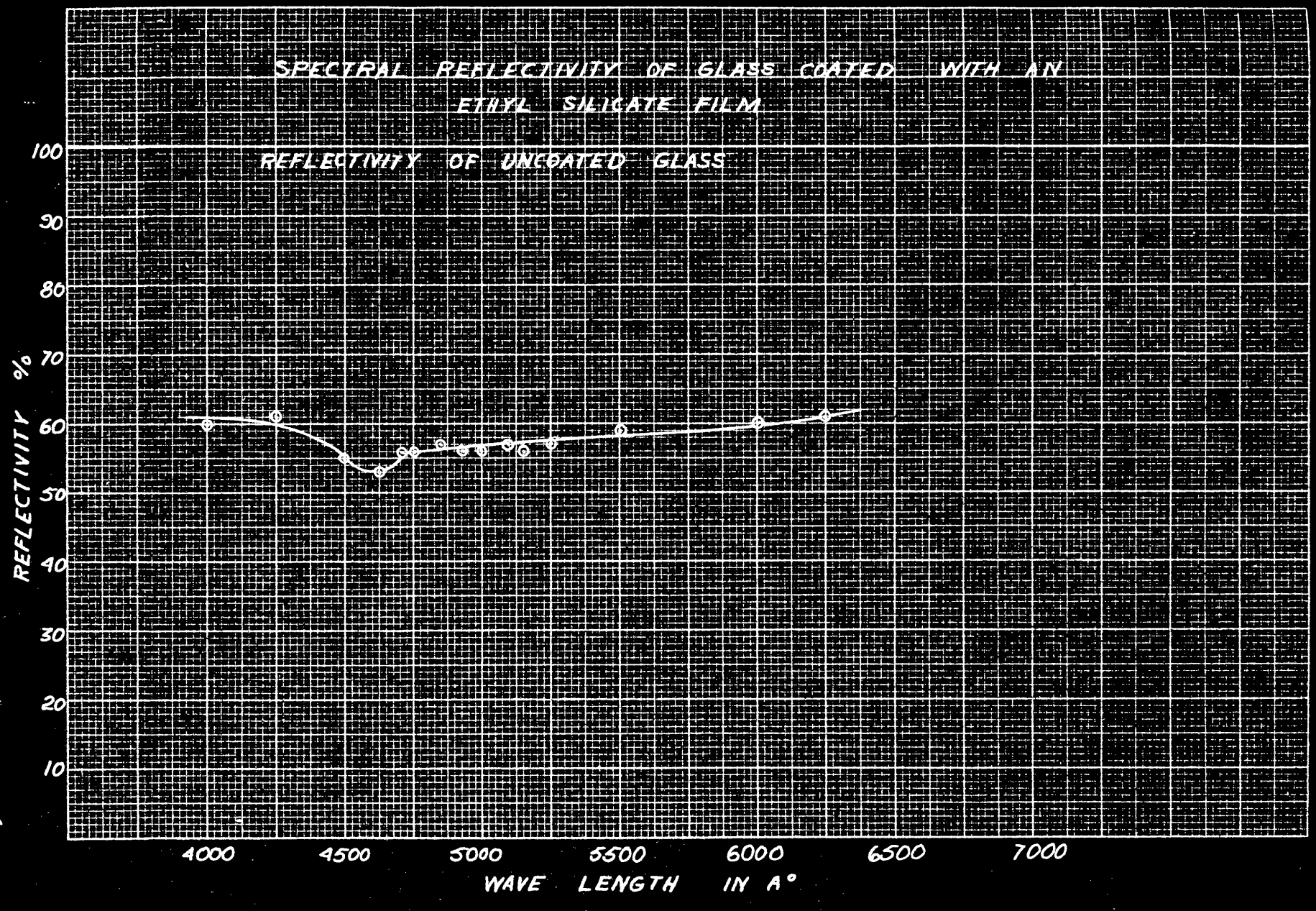
SPECTRAL REFLECTIVITY OF GLASS COATED WITH AN ETHYL SILICATE FILM
REFLECTIVITY OF UNCOATED GLASS
REFLECTIVITY %
100
90
80
70
60
50
40
30
20
10
4000
4500
5000
5500
6000
6500
7000
WAVE LENGTH IN A°

PLATE 4

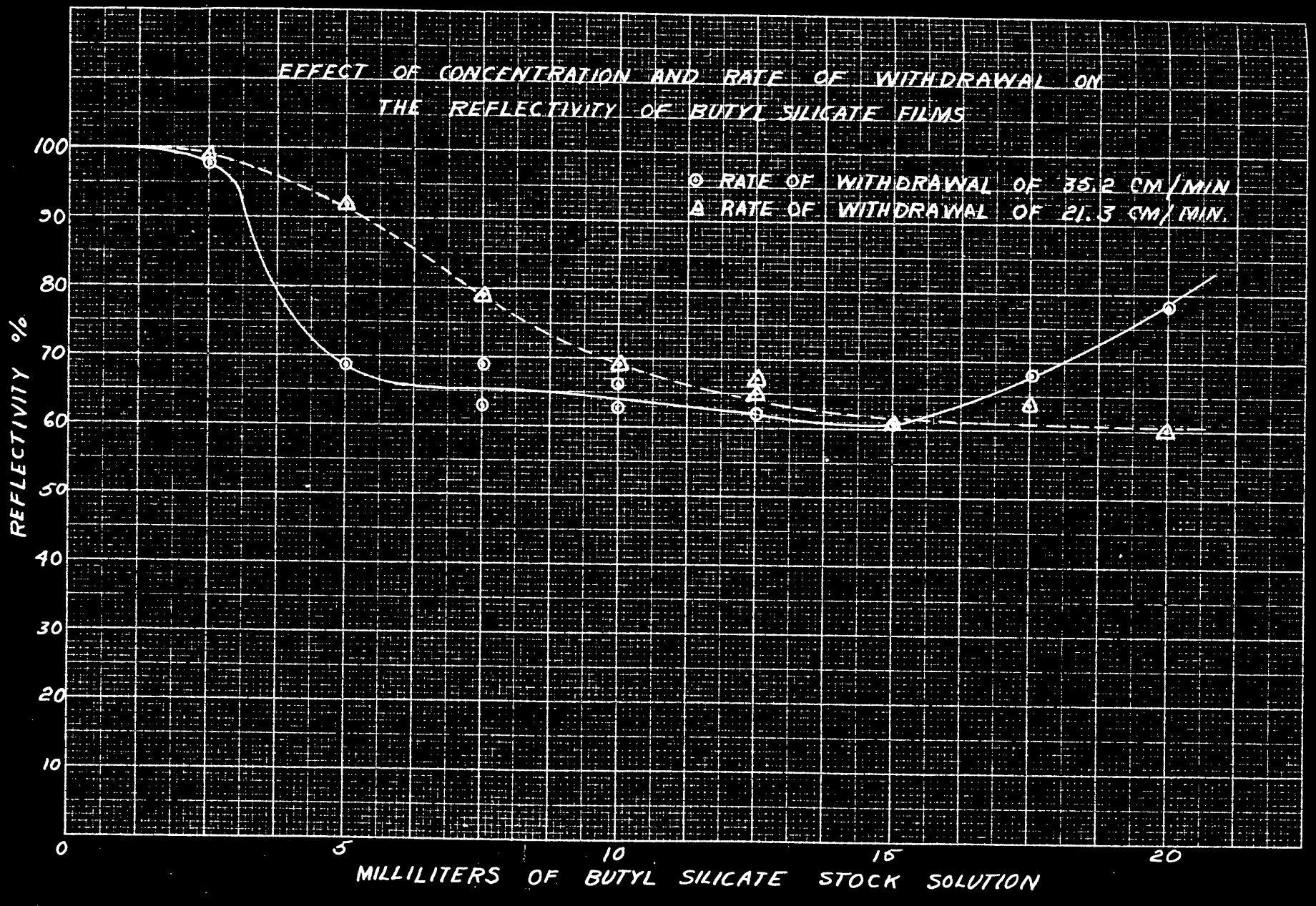
EFFECT OF CONCENTRATION AND RATE OF WITHDRAWAL ON
THE REFLECTIVITY OF BUTYL SILICATE FILMS
⊙ RATE OF WITHDRAWAL OF 35.2 CM/MIN
△ RATE OF WITHDRAWAL OF 21.3 CM/MIN.
REFLECTIVITY %
100
90
80
70
60
50
40
30
20
10
0
5
10
15
20
MILLILITERS OF BUTYL SILICATE STOCK SOLUTION

PLATE 5

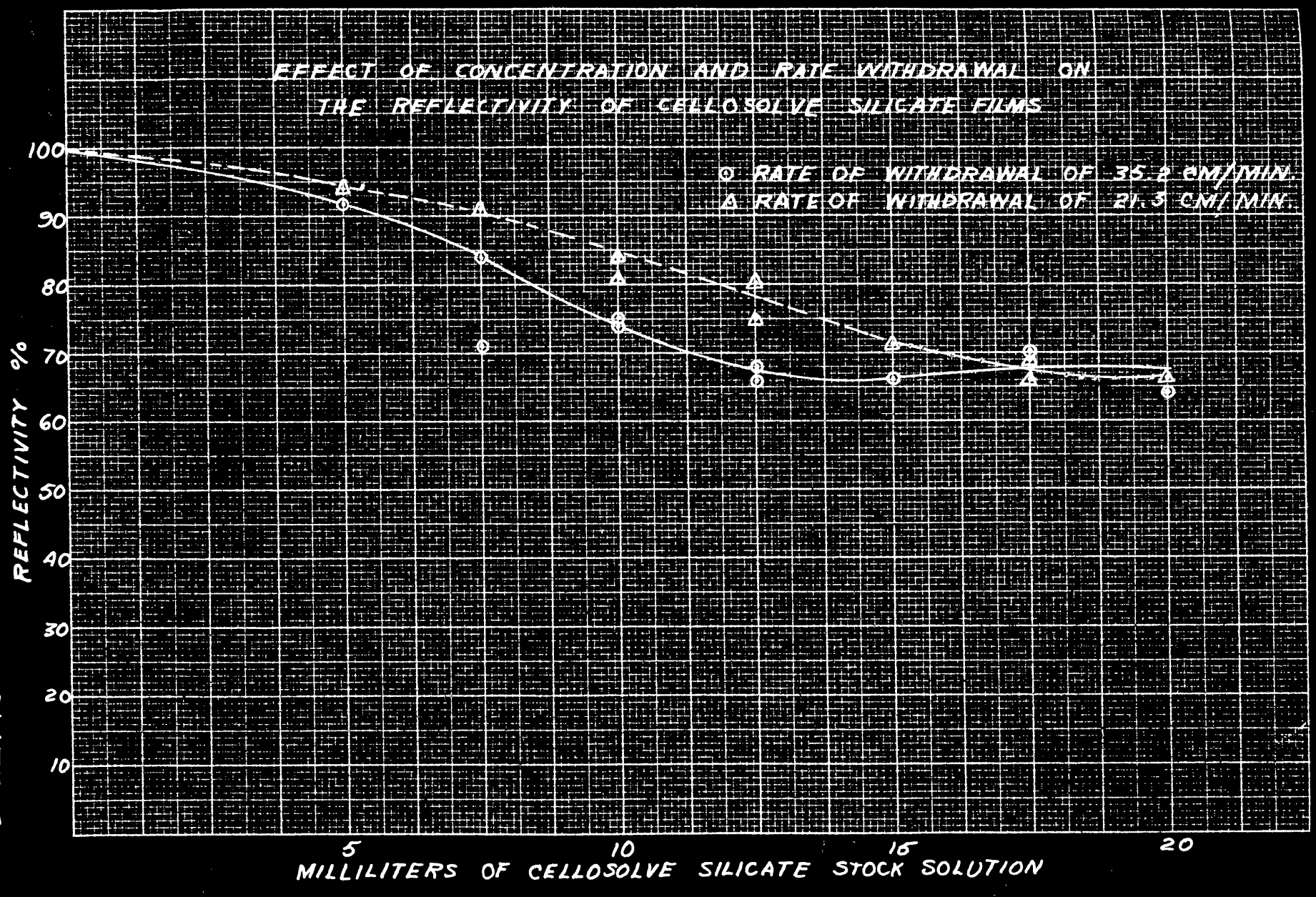
EFFECT OF CONCENTRATION AND RATE WITHDRAWAL ON
THE REFLECTIVITY OF CELLOSOLVE SILICATE FILMS
○ RATE OF WITHDRAWAL OF 35.2 CM/MIN.
△ RATE OF WITHDRAWAL OF 21.3 CM/MIN.
REFLECTIVITY %
100
90
80
70
60
50
40
30
20
10
5
10
15
20
MILLILITERS OF CELLOSOLVE SILICATE STOCK SOLUTION

PLATE 6

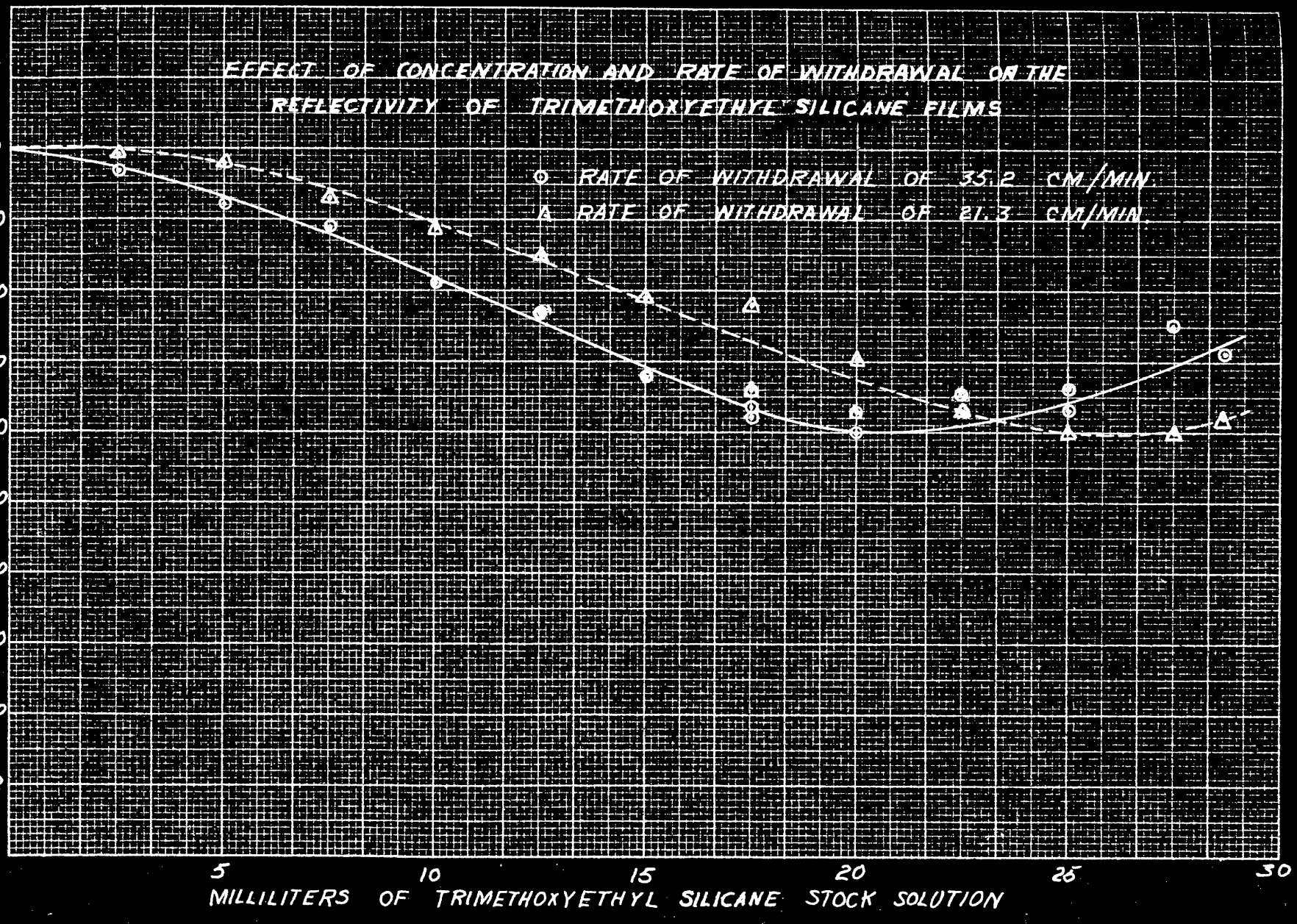
EFFECT OF CONCENTRATION AND RATE OF WITHDRAWAL ON THE
REFLECTIVITY OF TRIMETHOXYETHYE SILICANE FILMS
RATE OF WITHDRAWAL OF 35.2 CM/MIN.
RATE OF WITHDRAWAL OF 21.3 CM/MIN.
REFLECTIVITY %
100
90
80
70
60
50
40
30
20
10
5
10
15
20
25
30
MILLILITERS OF TRIMETHOXYETHYL SILICANE STOCK SOLUTION

PLATE 7

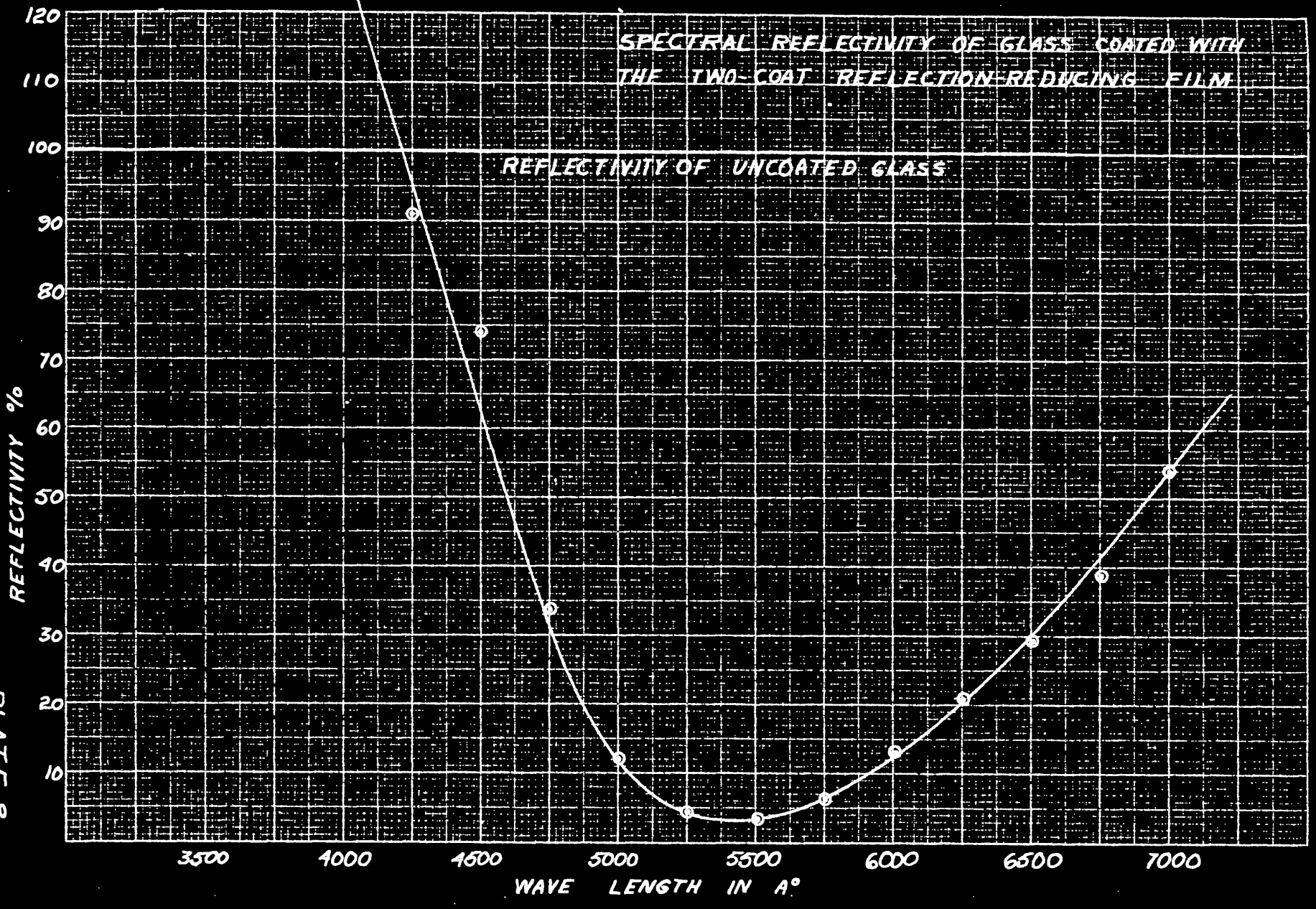
SPECTRAL REFLECTIVITY OF GLASS COATED WITH THE TWO-COAT REFLECTION-REDUCING FILM
REFLECTIVITY OF UNCOATED GLASS
REFLECTIVITY %
120
110
100
90
80
70
60
50
40
30
20
10
3500
4000
4500
5000
5500
6000
6500
7000
WAVE LENGTH IN A°

PLATE 8

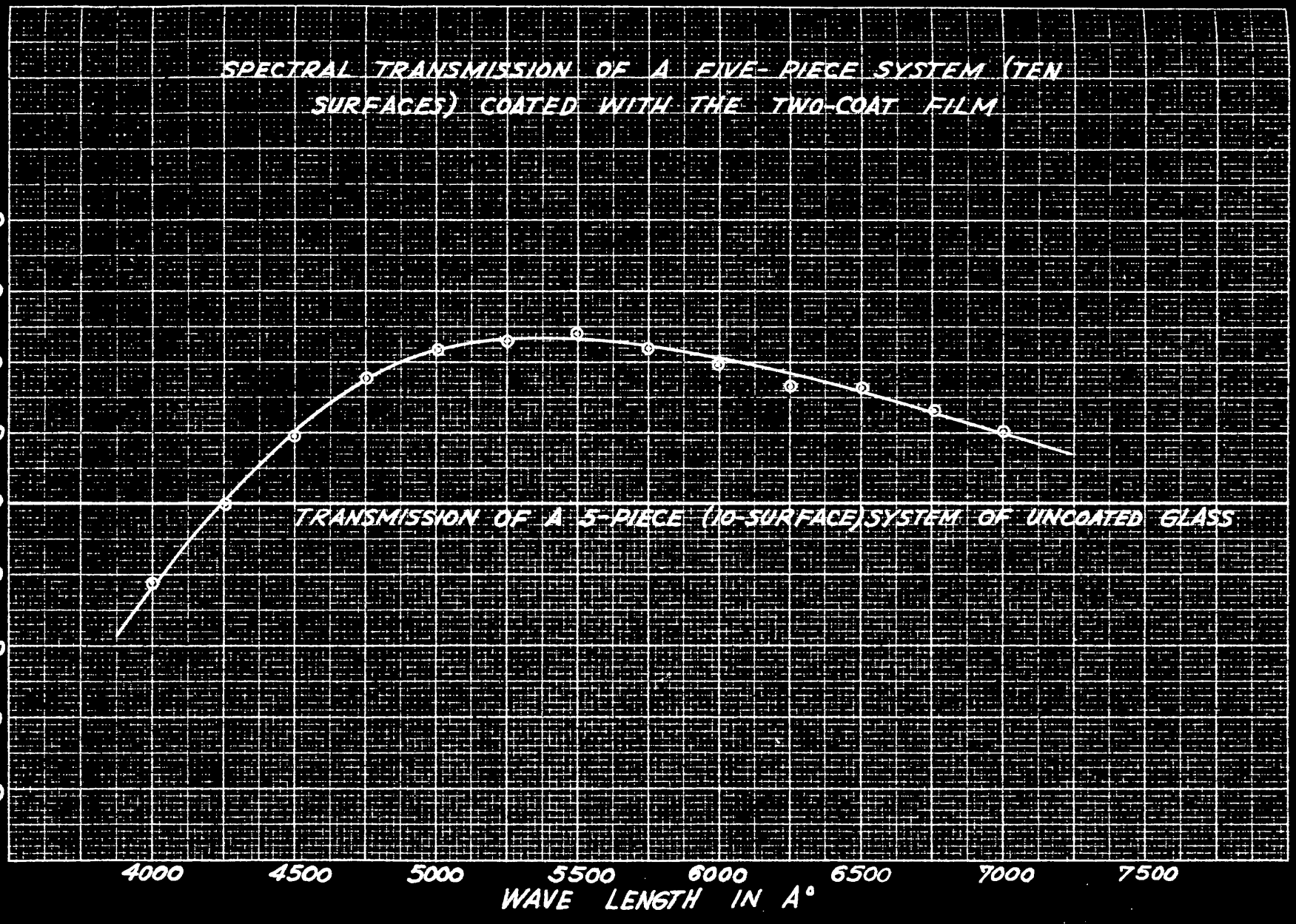
SPECTRAL TRANSMISSION OF A FIVE-PIECE SYSTEM (TEN SURFACES) COATED WITH THE TWO-COAT FILM
TRANSMISSION OF A 5-PIECE (10-SURFACE) SYSTEM OF UNCOATED GLASS
TRANSMISSION %
180
160
140
120
100
80
60
40
20
4000
4500
5000
5500
6000
6500
7000
7500
WAVE LENGTH IN A°

PLATE 9

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3) Second Report on Reflection Reducing Coatings for Glass and Transparent Plastic Surfaces. NRL Report P-2008 of 27 February 1943. (Confidential)

4) Moulton & Tillyer Patent Application. Serial #511, 364, Filed 22 November 1943.

5) Oleophobic Films Adsorbed from Oil Solutions. NRL Report P-2206 of November 1943. (Restricted)