tance since high viscosity media offer much greater sources of frictional resistance than the lower viscosity materials.

This comprises a very convenient determination since in the evaluation of the dielectric constant on balancing the A. C. bridge, the resistance as well as the capacitance must be balanced and, therefore, two determinations are made at one time. Figure 4 presents the per cent power factor change for the polymerized and the oxidized oils.

Examination of the arithmetic plot of viscosity-power factor shows that for any given viscosity the power factor of the oxidized oil samples is considerably greater than that of the polymerized oil. Consequently, power factor is mainly dependent on two variables: viscosity and oxygenated structure, the latter of which apparently contributes more. This could very well be another tool to be used to great advantage in the study of oxidation and oxidative polymerization during the processing of drying oils.

Summary

Three physical test methods for drying oils have been described. They are:

1. Molecular weight determinations in cyclohexane for thermally polymerized and mechanically oxidized oils. Results have been compared with those obtained in benzene. Theoretical and factual reasons have been given for the apparent validity of average molecular weight determinations in cyclohexane.
2. Dielectric constant determinations for treated oils. It has been shown that the dielectric increases as an oil is oxidized but that no increase in dielectric occurs on thermal polymerization. The dielectric constant is mainly a function of the oxygenated structure of the oil.
3. Power factor determinations. The power factor increases on polymerization and on oxidation. The values obtained for any given viscosity are considerably greater for the oxidized samples.

Molecular weight and dielectric constant determinations have been used to advantage in our plants and laboratories. Our knowledge on power factor determinations, however, is as yet not extensive but it is felt that with more background data on this constant a potentially meaningful tool for oil evaluation can be developed.

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Calcium Oxide-Soybean Oil Paints Having Reduced Tack and Increased Durability[1]

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Introduction

THE utilization of soybean oil as a drying oil has continued to grow since domestic oil was first offered to the industry. This growth has been slow because certain inherent properties limited its use. However, during and since World War II, the shortage of other drying oils has forced chemists and technologists to learn how to use it. At present, the paint and varnish industry is using soybean oil at the rate of nearly 150 million pounds per year.

The utilization of large percentages of soybean oil in outside white paints and many other protective coatings has resulted usually in a tacky film. This film collects dust and dirt which adheres to the surface until removal by chalking or self-cleaning. Another objectionable feature of soybean oil in paints is that the coatings tend to soften and re-liquefy when exposed to the high temperatures and humidities of the summer months. These softened coatings retain dust and dirt which becomes imbedded in the film so deeply that it cannot be washed out. This results in coatings that are unsightly in appearance and difficult to repaint. Also, the durability of the coatings is impaired by the stresses and strains set up by internal movements caused by repeated softening and hardening.

There are a number of ways in which the effect of these disadvantages can be reduced, in part or entirely; by proper pigmentation (7, 9); the use of soybean oil reacted with maleic anhydride (4); the use of soybean oil converted to pentaerythritol esters (2); the use of long-oil resins containing phthalic anhydride and glycerol (3); the use of oil modified with other dibasic acids and polyalcohols in small amounts to form molecules of higher functionality (13); the use of oils modified by copolymerization with styrene (8, 10), by copolymerization with tung oil (11), by blending with linseed and other unsaturated oils (7, 9); and other methods.

In connection with studies at the Northern Regional Research Laboratory on the catalytic isomerization of soybean oil, a conjugated oil was produced that dried rapidly to a tack-free coating when used as the oil vehicle in a basic lead carbonate paint (12). But these coatings, after an outdoor exposure of a week at 45° south in hot, humid weather, softened badly and collected a large amount of dirt which could not be removed by washing, and the coatings remained soft for a long time. This condition was particularly true of coatings prepared from oil isomerized at 200° to 220°C. and was probably

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TABLE I

Comparative Qualities of Coatings Containing Calcium Oxide and Non-Break Soybean Oil[1]
(Weathering tests made at 45° south)

Pigment components	Drying		Yellowing	Dirt retention	Reflectance		Gloss	
% by weight, balance oil	Dry to touch, hours	Free from tack, days	After storage in dark for 1 month	After exposure for 1 month	Before exposure	After exposure 8 months	Before exposure	After exposure for 1 week
75 basic carbonate lead	70+	120+	Light yellow	Considerable	87	29	38	19
71 basic carbonate lead + 4 CaO	10½	77	White	None	88	72	16	13
71 basic carbonate lead + 4 ZnO	12	120+	Light cream	Moderate	84	64	38	20
55 basic carbonate lead + 20 ZnO	11	96	Light cream	Light	85	68	40	22
71 basic carbonate lead + 4 MgO	13	50	White	Light	88	70	....	....
71 basic carbonate lead + 4 SrO	8	....	Light cream	Light	....	....	....	....
71 basic carbonate lead + 4 $CaCO_3$	40+	....	Medium yellow	Considerable	....	....	....	....
71 basic carbonate lead + 4 CaC_2O_4	40+	....	Medium yellow	Moderate	....	....	....	....
71 basic carbonate lead + 4 $CaSO_4$	40+	....	Medium yellow	Heavy	....	....	....	....
71 basic carbonate lead + 4 $MgSiO_3$	40+	....	Deep cream	Considerable	85	61	42	19
71 basic carbonate lead + 4 $Ca_3(PO_4)_2$	11	....	Medium yellow	Moderate	....	....	....	....
70—35% leaded zinc	14½	....	Light cream	Considerable	80	48	48	22
66—35% leaded zinc + 4 CaO	12½	....	White	None	79	72	17	16
66—35% leaded zinc + 4 $MgSiO_3$	18	....	Light cream	Considerable	75	51	34	14
70 zinc sulphide pigment	30	....	Light cream	Considerable	63	45	29	15
66 zinc sulphide pigment + 4 CaO	25	....	White	None	72	70	14	14
66 zinc sulphide pigment + 4 $MgSiO_3$	28	....	Medium cream	Considerable	70	50	26	14
70 titanium-barium pigment	60	....	Medium cream	Considerable	84	67	43	14
66 titanium-barium pigment + 4 CaO	12½	....	White	None	91	82	20	15
66 titanium-barium pigment + 4 $MgSiO_3$	62	....	Medium cream	Considerable	82	65	38	16
75 basic carbonate lead and raw linseed oil	4½	77	Deep yellow	Light	85	44	35	29

[1] All of the paints in Tables I-IV contained oil drier (Pb and Mn) conforming to Federal Specifications TT-D-651, in ratio of 5% to the oil volume.

caused by slow oxidation of isomerized oleic acid (15).

While experimenting with many pigments and chemicals that might be used with basic lead carbonate to eliminate this after-tack, it was found that such basic materials as the oxides of lead, zinc, strontium, magnesium, and calcium, among others, were more or less efficient in eliminating both residual and after-tack. Calcium oxide, because of its low price and abundance, was favored for the bulk of this laboratory's experimental work even though its use in paints gave coatings that dried flat. This was not considered a too serious objection because paint coatings generally lost most of their gloss during exposure to the sun (see Table I). In fact, it might be considered as an advantage because the coating containing calcium oxide would remain flat throughout its service instead of being glossy where shaded and flat where exposed to the sun.

To test the relative merits of calcium oxide and other pigments and materials a large number of paints were made. Most of these were pigmented with basic lead carbonate because of its tendency to develop after-tack with soybean oil while a few were pigmented with 35% leaded zinc, titanium-barium, or zinc sulphide pigments. The trade names of the pigments used in the preparation of these paints and their sources were as follows: Basic-lead carbonate, "Dutch Boy-333," National Lead Company; 35% leaded zinc, "Lehigh-61," New Jersey Zinc Company; titanium-barium pigment, "Titanox B-30," Titanium Pigment Company; zinc sulphide pigment, "Cryptone MS-130," New Jersey Zinc Company. The oils used were either soybean or linseed oil as single oil vehicles. Most of the soybean oil used was a non-break oil with an iodine number of 135 and an acid value of 2.0, but alkali-refined oil also was used in a few paints. Both oils were obtained from commercial sources. These paints were then tested comparatively for their qualities of drying, non-yellowing, light reflectance, dirt retention, and durability as well as for their loss of gloss after a week of outdoor exposure. Studies were made also with the X-ray camera to determine the reactions of the oxides of calcium and magnesium and the hydroxides of bismuth and aluminum in the paints.

Although the studies reported here have not been completed, they are published now to acquaint others who wish to investigate the use of calcium oxide and other basic materials for their possibilities in both inside and outside paints.

Improved Drying Times of Coatings

Twenty paints were made in accordance with the formulas listed in Table I. Their drying times were determined in a room in which constant temperature (78°F.) and relative humidity (50±3%) conditions were maintained throughout the testing period. The drying time determinations were made on a Sanderson drying meter after single coats were brushed out to full uniform coats on glass discs. The drying tests were of 70 to 72 hours' duration. This drying meter is designed to drop the sand uniformly at constant speed in a spiral track upon the coating. Two drying times were determined, one being approximately "dry to touch" time or the point at which the deposited sand was removed from the coating by a vigorous brushing with a bristle brush. The other corresponded to "free from tack" time, or the time when the deposited sand fell off the coating completely when the disc was inverted and tapped sharply with a brush handle.

From these drying time determinations listed in Table I it may be noted that:

1. Calcium oxide definitely shortened the drying time of basic lead carbonate paints made with non-break soybean oil while calcium phosphate and the oxides of magnesium, strontium, and zinc likewise shortened the drying time of coatings from similar paints.
2. Calcium oxide improved greatly the drying time of the coatings of the titanium-barium pigmented paints and slightly improved the drying time of zinc sulphide and 35% leaded zinc paint coatings.
3. Calcium oxide definitely improved the "free from tack" drying time of basic lead carbonate when used alone, and it was superior to zinc oxide in similar paint coatings.

Improved Non-Yellowing Qualities of Coatings

To test the comparative yellowing of paints containing calcium oxide the same paints were brushed out to three full coats on 6-inch panels of cedar siding with 3 days allowed for drying between coats. This coating procedure was used in preparation of cedar panels used in all other studies herein described. The panels were then stored at 78°F. and 50±3% relative humidity in a room in which there was no natural light. After a month they were examined visually for yellowing, with the results shown in Table I. It may be seen from this table that:

1. Calcium oxide eliminated the yellowing of the coatings containing basic lead carbonate, 35% leaded zinc, titanium-barium, and zinc sulphide pigments while magnesium oxide did likewise with basic lead carbonate.
2. Zinc oxide and strontium oxide decreased noticeably the yellowing of the coatings of the basic lead carbonate paints.

Improved Light Reflectance Qualities of Coatings

The light reflectance qualities of the coatings of 15 of the paints containing non-break soybean oil were determined by means of the Hunter portable photoelectric glossmeter (45°, 0°). These readings were the averages of five that were taken before and after exposure of the coatings outdoors at 45° south for 8 months, beginning in December.

The readings listed in Table I show that:

1. Calcium oxide definitely tends to retain the reflectance quality of basic lead carbonate paints after an outdoor exposure of 8 months.
2. Zinc oxide also tends to retain the reflectance qualities of similar paints, but to a slightly less degree than calcium oxide.

Reduced Dirt Retention Qualities of Coatings

Cedar panels coated with these same paints were exposed for outdoor weathering at 45° south beginning in July. After an exposure of a month the panels were examined for dirt retention after washing with running water.

The results of these observations are listed in Table I. They show that:

1. Calcium oxide was the only pigment or chemical that eliminated dirt retention on coatings from basic lead carbonate paints.
2. Calcium oxide eliminated dirt retention from the coatings of paints containing 35% leaded zinc, titanium-barium, and zinc sulphide pigments.
3. Magnesium and strontium oxides with basic lead carbonate showed considerable reduction of the dirt retention on the coatings for their respective paints.

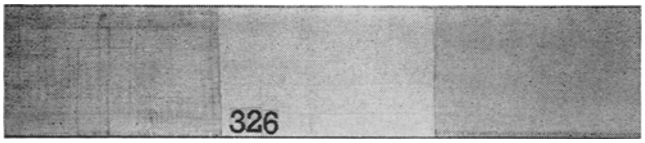

FIG. 1. Relative dirt retention of non-break soybean oil paints after outdoor weathering at 45° south for one month at Peoria, Illinois. Panel was washed with water and sponge.

Left—Basic carbonate lead paint.

Center—Basic carbonate lead paint containing 4% CaO.

Right—Basic carbonate lead paint containing 4% ZnO.

Comparative Specular Gloss Determinations

It has been noted previously that the coatings of outside white paints in which calcium oxide was used, dried to flat or non-glossy coatings. To determine the relative gloss of paint coatings containing calcium oxide before and after exposure in comparison with other paint coatings, 14 paints were made in accordance with the formulas listed in Table I. The gloss of the coatings on cedar panels was determined on the seventh day and before exposure, and again after the panels had been exposed outdoors at 45° south for a week. The specular gloss determinations were made by means of a Hunter portable photoelectric glossmeter (45°, —45°). These readings (average of five) are given in Table I where it may be noted that:

1. The gloss values of paint coatings containing calcium oxide, while low (13 to 20) before exposure, remain practically the same after a week of outdoor exposure while similar paint coatings that contain no calcium oxide or those in which zinc oxide, aluminum oxide, or magnesium silicate are substituted for calcium oxide, showed marked decrease in gloss readings —sometimes dropping as low as those of the calcium oxide coatings.
2. Calcium oxide paint coatings had relatively the same gloss readings in all four pigmentations.

Improved Durability Qualities of Coatings

The durability of the coatings of paints containing calcium oxide or other pigments or chemicals has been determined by both accelerated and outdoor weathering tests. The latter tests were made on test fences at Peoria, Ill., at 45° south and at 90° north and south. The 90° south exposure test results were selected for presentation in Tables II, III, and IV because the results of this type of exposure are considered more reliable. However, there were no serious differences noted among the tests and all were in good agreement.

The cedar test panels of each Table were selected for having the same quality of wood before painting, and they were exposed comparatively. The results of the 90° south exposure tests were as follows:

1. Table II shows that after an exposure of 32 months, the coatings from a paint containing 4% cal-

TABLE II

Comparative Durability of Raw Linseed and Non-break Soybean Oil Paint Coatings Containing Basic Carbonate Lead and Various percentages of calcium oxide

Weights of paint components		Exposure tests at 90° south (Peoria, Ill.)
% pigment	Oil (25%)	After 32 months
75 basic carbonate lead	Soybean oil	Very heavy, deep checks and cracks
75 basic carbonate lead	Linseed oil	Very heavy, deep checks and cracks
71 basic carbonate lead + 4 CaO	Soybean oil	Light surface checks
71 basic carbonate lead + 4 CaO	Linseed oil	Considerable fine surface checks and cracks
67 basic carbonate lead + 8 CaO	Soybean oil	Very heavy, deep checks and cracks, moderate scaling
67 basic carbonate lead + 8 CaO	Linseed oil	Very heavy surface checks and cracks
63 basic carbonate lead + 12 CaO	Soybean oil	Very heavy, deep checks and cracks, moderate scaling
63 basic carbonate lead + 12 CaO	Linseed oil	Very heavy surface checks and cracks

cium oxide, 71% basic carbonate lead, and 25% non-break soybean oil was more durable than a similar paint coating containing raw linseed as the oil vehicle. On the surface the former checked only lightly while the latter checked and cracked considerably. The Table shows also that both linseed and soybean oil paints containing 4% of calcium oxide were definitely superior to similar paints containing higher percentages of calcium oxide or none at all. The 4% calcium oxide paints were better in appearance and, since breakdowns were on the surface rather than deep or through the surface, they were also better for repainting.

TABLE III

Comparative Durability of Paint Coatings Containing Non-break Soybean Oil, Basic Carbonate Lead With Alkaline Earth Oxides, etc.

Pigment components	Exposure tests at 90° south (Peoria, Ill.)
% by weight, balance oil	Results noted after 3 years
75 basic carbonate lead	Very heavy, deep checks and cracks
71 basic carbonate lead + 4 CaO	Light to moderate surface checks
71 basic carbonate lead + 4 $MgSiO_3$	Very heavy erosion to exposed wood
71 basic carbonate lead + 4 MgO	Considerable deep cracks
71 basic carbonate lead + 4 SrO	Considerable deep checks and cracks
71 basic carbonate lead + 4 BaO	Heavy, deep alligatoring
71 basic carbonate lead + 4 ZnO	Heavy, deep checks and cracks
71 basic carbonate lead + 4 calcium stearate	Very heavy, deep checks and cracks
71 basic carbonate lead + 4 aluminum stearate	Heavy, deep checks and cracks
71 basic carbonate lead + 4 $CaCO_3$	Heavy, deep checks and cracks
71 basic carbonate lead + 4 CaC_2O_4	Heavy, deep checks and cracks
71 basic carbonate lead + 4 $Ca_3(PO_4)_2$	Very heavy, deep checks and cracks
71 basic carbonate lead + 4 mineralite	Considerable deep, heavy checks and cracks

2. Table III shows that the paints containing 4% of calcium oxide were superior to similar paints containing the same percentage of magnesium oxide, magnesium silicate, strontium oxide, zinc oxide, etc., after an exposure of 3 years because, again, the coating breakdown occurred on the surface rather than through the surface, and therefore gave improved appearance. It may be noted that each of the paints mentioned in this table contained the same weights of basic lead carbonate (71%) and non-break soybean oil (25%) together with the 4% of calcium oxide, etc.

3. Table IV shows that paints containing 4% calcium oxide with 66% of 35% leaded zinc and oil vehicles of non-break, or alkali-refined soybean or raw linseed oils, gave coatings that, after 2 years' exposure, were checked and cracked on the surface only, in comparison with the deep checks and/or cracks on the paint coatings of leaded zinc (35%) alone or with 4% magnesium silicate. It also shows that the same amount of calcium oxide or magnesium silicate with the zinc sulphide pigment increased chalking and erosion but eliminated the cracking of the coatings from the paints containing only the zinc sulphide pigment when the oil vehicles were non-break or alkali-refined soybean oils. It should be noted that the paint coatings containing the zinc sulphide pigment alone with raw linseed oil had better durability qualities than similar paint coatings containing calcium oxide or magnesium silicate.

X-Ray Studies

Paints containing conjugated soybean oil (12) were used throughout the X-ray studies since the after-tack phenomena was more pronounced with this oil. X-ray examination of films prepared from paints containing 25% calcium oxide as the only pigment and 5% oil drier showed that no crystalline organic compounds were formed in drying and that the crystalline components appeared from the inorganic pigments added to the oil. Examination of these films at 3, 6, 9, 13, 47, and 122 days showed that the oxide gradually changed to calcium hydroxide and that no crystalline calcium carbonate was formed.

Since calcium oxide might act as a dehydrating agent, other dehydrating agents were tested. When films containing 25% anhydrous sodium sulfate and calcium sulfate were examined by X-rays at 3, 7, 18, and 40 days of drying, no hydration of the sulfates was observed. Both sulfate films developed excessive after-tack and this condition persisted for more than 5 months. These coatings showed a definite acidic reaction when tested with wet litmus paper.

Studies were made of the coatings of paints from conjugated soybean oil plus drier containing calcium oxide, magnesium oxide, aluminum hydroxide, and bismuth hydroxide in varying percentages of 2, 5, 8⅓, and 16⅔. The X-ray pictures of these coatings made after 3, 7, and 14 days' drying showed:

1. Calcium oxide was changed to the hydroxide in almost 3 days. Calcium hydroxide subsequently disappeared from the X-ray pictures as the drying progressed; first from the 2% and finally, after several weeks, from the 16⅔% pigment paints. The only "calcium oxide" coating which showed even a slight amount of tack after 5 weeks' drying was the 2% calcium oxide paint, and this probably was caused by the lack of a greater percentage of basic material in the paint.

TABLE IV

Comparative Durability of Raw Linseed, Alkali Refined (A.R.), and Non-break (N.B.) Soybean Oil Paint Coatings Containing Leaded Zinc (35%) or Zinc Sulphide Pigment With Calcium Oxide or Magnesium Silicate

Weights of paint components		Exposure tests at 90° south (Peoria, Ill.)
% pigment	Oil (30%)	Results noted after 2 years
70 leaded zinc	N.B. soybean	Heavy macro checks, moderately deep
70 leaded zinc	A.R. soybean	Very heavy, deep macro checks and cracks
70 leaded zinc	Linseed	Considerable deep cracks
66 leaded zinc + 4 CaO	N.B. soybean	Considerable fine surface checks and cracks
66 leaded zinc + 4 CaO	A.R. soybean	Heavy surface checks and cracks
66 leaded zinc + 4 CaO	Linseed	Moderate fine surface checks and cracks
66 leaded zinc + 4 $MgSiO_3$	N.B. soybean	Heavy,deep checks and cracks
66 leaded zinc + 4 $MgSiO_3$	A.R. soybean	Considerable deep checks and cracks
66 leaded zinc + 4 $MgSiO_3$	Linseed	Considerable deep cracks
70 zinc sulphide pigment	N.B. soybean	Light, deep cracks. Considerable chalk
70 zinc sulphide pigment	A.R. soybean	Trace of cracks. Moderate chalk
70 zinc sulphide pigment	Linseed	No checks or cracks. Light chalk
66 zinc sulphide pigment + 4 CaO	N.B. soybean	No checks or cracks. Moderate erosion to wood
66 zinc sulphide pigment + 4 CaO	A.R. soybean	No checks or cracks. Heavy erosion to wood
66 zinc sulphide pigment + 4 CaO	Linseed	No checks or cracks. Light erosion to wood
66 zinc sulphide pigment + 4 $MgSiO_3$	N.B. soybean	No checks or cracks. Moderate erosion to wood
66 zinc sulphide pigment + 4 $MgSiO_3$	A.R. soybean	No checks or cracks. Considerable erosion to wood
66 zinc sulphide pigment + 4 $MgSiO_3$	Linseed	No checks or cracks. Very heavy erosion to wood

2. Magnesium oxide, unlike calcium oxide, did not appear to change to hydroxide during the drying process, but its disappearance was very pronounced, especially for the 2-, 5-, and 8⅓-% paints. These coatings, like those of the calcium oxide paint, showed no tack except a light tack on the 2% magnesium oxide coating.

3. Aluminum hydroxide paint coatings did not give any X-ray information because the original pigment was amorphous and remained so throughout the drying process. All coatings developed considerable after-tack.

4. Bismuth hydroxide did not appear to react or disappear from the X-ray pictures during the drying process and all coatings developed considerable after-tack.

Discussion and Conclusions

The utilization of approximately 5% of calcium oxide in outside white paints on cedar panels on test fences improves the coatings of these paints, especially when the oil vehicles are either non-break or alkali-refined soybean oil, in the following respects:

1. They dry more rapidly to a hard coating that is free from residual and/or after-tack.
2. They do not yellow in dark, shaded exposures.
3. They have greatly improved reflectance qualities.
4. They have greatly reduced dirt retention.
5. They dry flat or without appreciable gloss, and stay uniform in appearance in service, whether in shaded or sunlit areas.
6. They have increased resistance to deep checking, cracking, and alligatoring—the defects that make satisfactory repainting difficult.

It may be concluded from the X-ray and paint studies that the property of calcium oxide to reduce the after-tack of soybean oil paint coatings is shared by many strongly basic compounds.

The important chemical reaction involved appears to be the neutralization of organic acids to form calcium or other alkaline earth metallic salts. Initial hydration of the oxide to hydroxide may occur but hydration might be a side reaction only because water could be formed by the inter-action of two organic acid molecules with the oxide. This water might then react with the oxide to give the hydroxide.

Qualitative tests indicate clearly that materials which react acidic to litmus are formed when soybean oil films are exposed to the air. The slight tack found on 2% calcium oxide and magnesium oxide films may indicate that insufficient alkali is present to neutralize all acidic products. Elm (6), and others (1, 14) have shown that acidic cleavage products are formed when linseed oil films are exposed and Slansky (14) has shown that these acidic materials are not hydrolytic products from the glyceride. The known plasticizing and dissolving effects of the lower fatty acids on organic molecules and on oil films, in particular, is well-known. It appears reasonable to assume that the correct explanation for the behavior of calcium oxide and the other alkaline earth oxides in these paint and oil films is that the acidic cleavage products are neutralized to form salts which do not soften the film or give it undesirable properties.

Previous workers have reported the formation of salts in the film when red lead was used (5), and a recent publication indicates that the use of certain metallic oxides, including calcium and aluminum, can be used as light stabilizers for lead silicate pigments (16). However, to our knowledge no one has previously recognized the beneficial effects which are obtained when calcium oxide is used in soybean oil paints.

The use of names of firms or commercial products in this article is for identification; it is not an endorsement of either the product or the manufacturer.

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