This invention relates to wax emulsions and to the preparation thereof. More particularly, it relates to wax emulsions which are usable as coatings in the paper industry.

One of the fastest growing segments of the paper industry is the production of paper and paperboard laminates containing plastic films. These laminates are normally referred to as functional coatings and find their widest use in the decorative field and the packaging (largely food) industry, but also have other applications where the combined effect of the properties of paper and a plastic film are needed.

The packaging industry, for instance, has for many years used a two component package wherein the item to be wrapped is first covered with a film of some type, such as polyethylene, glassine, or some other film with good grease proof and/or water resistant properties, and then inserted into a paper box which provides the bulky and decorative wrap. The trend in recent years has been to the single unit package with a coating or similar barrier on the inside of the package to provide the necessary grease and/or water resistance and optionally they may have a coating on the outside of the package which will accept print to give “eye appeal” thus eliminating separate outer and inner wraps and yet accomplishing the same goal with a lower cost single wrap package.

The functional coatings are normally applied by the paper converter, who purchases his paper and paperboard from the papermill and applies the plastic films using a hot melt, solvent or extrusion technique and then prints and cuts the paper and paperboard into the finished package. The functional coatings available in the prior art for the packaging industry require specialized machines and can be applied to paper and paperboard by conventional papermaking equipment. It is readily apparent that great savings in time and money could be gained if these functional coatings could be applied utilizing conventional papermaking equipment.

The application of aqueous emulsions to paper and paperboard utilizing conventional papermaking machinery is known in the art. There are a multitude of water emulsions of waxes, polyethylene, polyvinylacetate, butadine, styrene, polyvinylidene chloride, acrylics, etc. on the market which claim various properties such as grease resistance, water resistance, abrasion and scuff resistance, wax and oil holdout, etc. which are applied to the surface of paper and paperboard and dried during the conventional paper making process. However, for a variety of reasons these emulsions previously developed in the prior art have not been altogether satisfactory for large scale commercial operations in the production of a single unit package. The paper and paperboard coated with the prior art emulsions have many drawbacks as shown by the following.

Many of these emulsions penetrated too far into the paper film when applied in the aqueous state and did not stay on the surface of the paper where they are needed if they are to function properly. Other aqueous emulsions were good for such purposes as paper glue without giving a good film but were too tacky and caused severe blocking at room temperature. Other emulsions did not give good films unless they were dried at temperatures well exceeding the temperatures available on conventional papermaking equipment. Still others gave films which were excellent in every respect except that they were too brittle, and had poor bend and fold properties and therefore were inadequate for use in the packaging industry.

It is an object of this invention to provide new and improved water dispersible coating compositions suitable for coating paper and paperboard.

It is a further object of this invention to provide aqueous emulsions containing waxes and polyethylene resins which can be applied by conventional papermaking machines to produce functional films on paper and paperboard.

It is a further object of this invention to provide improved coating compositions for paperboard and paper comprising a mixture of polyethylene, waxes and other resins which when applied to paper will render the paper grease proof, water repellent, and non-blocking. Other objects of this invention will in part be obvious and will in part appear hereinafter.

It has been discovered that the foregoing objects are readily accomplished by preparing wax-polyethylene resin blends which contain refined paraffin waxes from natural mineral sources, synthetic hard paraffin waxes, polyethylene resins or a mixture of polyethylene resins and thereafter emulsifying the wax-polyethylene resin mixtures with particular emulsification bases disclosed hereinafter thus forming the coating compositions of this invention. In the preferred embodiment of my invention an aqueous emulsion of a polymer latex such as polyvinyl acetate, poly styrene, etc., or a blend of such latices having a solids content ranging from about 40% to 60% by weight of said latex emulsification is incorporated in the emulsified wax-polyethylene coating composition. When a polymer latex or mixture of polymer latices is combined with the emulsified wax-polyethylene coating composition, the weight ratio of the polymer latex to the wax-polyethylene blend may vary from about 4:1 to about 1:4 based upon the solids content of the polymer latex and the wax-polyethylene blend. The emulsified coating compositions of my invention are easily applied to paper or paperboard utilizing existing conventional papermaking machinery.

The wax-polyethylene blends used in my invention contain from about 20% to about 52% by weight of refined paraffin wax based on the total weight of the wax-polyethylene blends; from about 35% of about 75% by weight of hard paraffin wax based on the total weight of the waxpolyethylene blend and from about 6% to about 20% by weight of polyethylene resin or mixtures of various polyethylene resins based on the total weight of the wax-polyethylene blend.

Refined paraffin waxes which can be used in my wax-polyethylene blends are those aliphatic hydrocarbon waxes obtained from natural sources and which have a melting point ranging from about 122° F. to about 165° F. Such waxes are of the type normally used for the coating or waxing of paper. As employed herein, the term refined paraffin wax refers to hard crystalline waxes derived from distillates obtained from the breakdown of petroleum oils of a mixed base of paraffin waxes. Preparation of this wax from the distillate may conveniently be effected by chilling the distillate to a temperature below 32° F. and then filtering the solid wax from the oil in a filter press. The wax collected in the filter press is identified in the art as “slack wax” and it is characterized by the fact that it contains a large quantity of oil. In most refineries this oil is removed from the “slack wax” by “sweetening” followed by a redistillation of the “slack wax.” Generally the wax is further treated with sulfuric acid or percolation through clay. In the preferred embodiment of my invention the wax-polyethylene resin coating composition contains from about 44% to about 48% by weight, based upon the total weight of the wax-polyethylene blend, of the re-
fined paraffin wax having a melting point preferably of about 148° F. to about 153° F.

The synthetic hard paraffin waxes used in this invention are synthetic polymers of carbon monoxide and hydrogen having a molecular weight of 8,192,172 to about 12,000. They can be best described as mineral waxes. They are prepared by the polymerization of carbon monoxide and hydrogen by means of the Fischer-Tropsch process. They are hard, brittle, and white in color. They are long-chain aliphatic hydrocarbon with relatively short side-chains. They must be essentially free of residual oil and sulfur. When melted, these hard paraffin waxes are fluid and water clear. They have a relatively sharp melting point ranging from about 212° F. to about 225° F. and a solidification point ranging from about 190° F. to about 212° F. They should have a saponification value less than 0.5 and an acid value of less than 0.1, a dielectric constant of approximately 3, and an electrical resistance of approximately 10¹⁵ ohm x cm. and a needlepoint penetrometer hardness of no greater than 5 (100 grams/5 secs./77° F.). In the preferred embodiment of my invention, the wax-polyethylene blend should contain from about 40% to about 55% by weight of synthetic hard paraffin wax based upon the total weight of the wax-polyethylene blend. Illustrative of synthetic hard paraffin waxes that have been found useful in the practice of my invention are "Paraffin wax" and the like.

Polyethylene resins found useful in my wax-polyethylene blends are those polyethylene resins which have a molecular weight ranging between an average of 5,000 and 12,000. The polyethylene resins when used in my method of composition must not be saponifiable.

The polyethylene resins used in this invention were produced by a variety of methods known to those skilled in the art. For example, a high temperature method of preparing high molecular weight ethylene polymers is described in detail in United States Patent No. 2,153,569. We have found that a thermoplastic resin and softens at temperatures above 130° C. It is very tough although relatively inelastic. The polyethylene resins which I use in my novel emulsified coating compositions are translucent, white in color, and resemble hard paraffin waxes in appearance and texture. In the preferred embodiment of my invention, the wax-polyethylene blend which contains from about 12% to about 16% by weight of a polyethylene resin based upon the total weight of the wax-polyethylene blend and have an average molecular weight of 7,000.

In preparing the coating compositions, the wax-polyethylene materials, i.e., the refined paraffin wax, the hard paraffin wax and the polyethylene resin, are first blended together in a liquid state. The method chosen to achieve this blend is not critical and is normally accomplished by melting all of the aforementioned ingredients and adding the wax-polyethylene resin to the melt. However, since polyethylene does not melt at normal temperatures, in the preferred embodiment the refined paraffin wax and the synthetic hard paraffin wax are melted together with constant agitation until a homogeneous blend of these waxes is attained and thereafter the polyethylene resin is added. Two waxes with agitation until a uniform consistency is obtained throughout. This blend is then mixed with the emulsification base described below and emulsified.

The emulsification base used herein contains from about 0.1% to about 7% by weight of a primary emulsifier based on the total weight of the wax-polyethylene blend; from about 1.5% to about 15% by weight of a secondary emulsifier having a melting point ranging from about 80° C. to about 95° C.; and from about 80% to about 98% by weight of water based upon the total weight of the wax-polyethylene blend. Optionally the emulsification base may contain from about 0.1% to about 0.5% by weight of a water softening agent based on the weight of the wax-polyethylene blend.

I have found that saponified oxygenated hard paraffin waxes are excellent for use as the primary emulsifier in the emulsification bases of my invention. These waxes are produced by oxidizing either a synthetic hard paraffin wax such as those hard paraffin waxes previously described which are used in preparing the wax-polyethylene blend, or by oxidizing a regular hard paraffin wax of natural mineral origin. These waxes may be oxidized by a variety of procedures such as heating the wax to at least 10° C. above the melting point of the wax and bubbling a stream of air throughout the molten wax. Catalysts such as metallic soaps and organo metallic peroxides may also be employed in effecting the oxidation of the waxes. However it is to be understood that this invention is not directed to the oxidation of hard paraffin waxes and that the various preparations of these waxes are well known to those skilled in the art. The saponified hard paraffin waxes which I use in the preparation of the primary emulsifier should have a solidification point range of from about 185° F. to about 200° F., an acid value range of from about 20 to about 60; and a saponification value range of from about 100 to 110.

The amount of saponified oxygenated hard paraffin waxes employed in the emulsified wax-polyethylene coating compositions can vary from about 0.1 percent to about 7 percent by weight, based upon the total weight of the wax-polyethylene blend. The secondary emulsifiers of my invention may suitably be the following Polysorbates, also solubilized proteins such as casein, soya bean protein, etc. and mixtures thereof.

Alkalas found suitable for solubilizing the proteins which are utilized in this invention are ammonium hydroxide, organic amines, water soluble non-oxidizing sodium and potassium salts such as sodium hydroxide, sodium carbonate, sodium ortho-phosphate, sodium metasilicate, sodium orthosilicate, potassium hydroxide, potassium carbonate, tri-potassium ortho-phosphate, potassium metasilicate, potassium ortho-silicate, borax, etc. The amount of alkali employed varies according to the acidity of the protein and the strength of the alkali employed. Care should be taken to see that the pH of the aqueous solution of the water solubilized protein is between about 7.0 and about 10. When the casein is used as the secondary emulsifier, it should be acid precipitated, that is, in an unhydrolyzed condition. When soybean protein is utilized, the casein is acid precipitated to give a molecular weight range between 220,000 and 380,000.

When it is desired to add water softeners to the emulsification bases of this invention, any water soluble phosphate complex can be added such as sodium pyrophosphate, soda ash of the blend of sodium carbonate and potassium phosphate to the emulsification base. It is to be understood that my invention is not directed to the softening of water per se and that water softening
agents are to be employed in my invention only in those cases where difficulty is encountered in adequately dispersing wax-polyethylene blend as used in my invention well known in the art and any water softening agent can be used which will not prevent the emulsification of the wax-polyethylene blend with the emulsification bases.

The ingredients of the emulsification bases listed above are admixed with the sodium hydroxide at elevated temperatures, or by boiling the boiling point of the final emulsification base solution. The ingredients readily go into solution at elevated temperatures and no special precautions need be taken in preparing the solutions of these emulsification bases. The wax-polyethylene blend as prepared above is then slowly added to the solution of the emulsification base which is maintained at or near its boiling point while the mixture is vigorously agitated. Upon completion of the addition of the wax-polyethylene blend to the emulsification base, a primary emulsion of an unstable nature is formed. The particle size of the emulsion in primary emulsion is then further reduced by applying shear to the particles, e.g., by passing the emulsion through a colloid mill or a homogenizer such as a Manton-Gaulin Homogenizer until the particle size of the final emulsion is then below 2 μ (0.002 millimeter).

In the preferred embodiment of my invention the primary emulsion is passed through a Manton-Gaulin Homogenizer at 4,500 p.s.i. and a temperature of approximately 98°C. to 100°C. is maintained. After the particle size of the primary emulsion has been reduced to below 2 μ, the emulsion is quickly cooled to below 40°C. Cooling may be accomplished by any means which will provide for rapid cooling of the emulsion such as a heat exchanger. If the emulsions are to be stored for protracted periods of time, small amounts of formaldehyde are added in amounts ranging from about 0.1% to about 3% by weight of formaldehyde, based upon the total weight of the emulsified coating composition. The addition of formaldehyde will prevent decomposition of the constituents of the final emulsion. It is to be understood of course that any preservative which is compatible with the emulsion may be added to the emulsion to prevent damage due to bacteria.

In the preferred embodiment of my invention, an aqueous emulsion of a polymer latex such as polyvinyl acetate, polybutadiene, polyisobutylene, polyvinylidene chloride and thereof can be incorporated into the emulsified wax-polyethylene coating compositions of this invention if so desired in accordance with conventional practice.

The application of my emulsified wax-polyethylene coatings to paper and paperboard can be done in various ways. Any equipment or procedure that has been utilized previously in the art to apply fluid coatings to paper or paperboard can be utilized in applying the coating compositions of this invention to paper or paperboard. Thus, for example, equipment normally found in paper-making operations such as spray devices, brush devices, roll coaters, trailer bladers, doctor blades, air blades, or similar devices can be employed in applying the coating compositions of this invention to paper or paperboard.

The temperatures required to dry the emulsions disclosed in this invention range from 212°F. to 600°F. These temperatures can be produced on conventional paper-making machines by infra-red lamps, steam dryers and hot air blowers. When paper is coated with the emulsified coating compositions of this invention, it is preferred that the paper have a coating of greater than about 1 mil in thickness on its surface. Coatings of much greater thickness may be applied to paper if it is so desired and multiple coatings may be applied to the paper as desired.

It is not fully known precisely why my novel emulsified coating compositions perform as they do. However, it is believed that a synergistic effect is obtained from the emulsification of the wax-polyethylene coating blends with the emulsification bases which enable the emulsion so formed to break immediately upon its application in the form of a thin film on the paper. Thus the coating is not fully absorbed by the paper. Therefore deleterious effects, such as reverse migration are avoided when the emulsified coating compositions are employed. However, it is known that my novel coating compositions do penetrate the paper sufficiently to become firmly affixed to the paper thus forming a smooth, even coating.

For a fuller understanding of the nature and objects of the invention, reference should be had to the following examples which are given merely as further illustrations of the invention and are not to be construed in a limiting sense.

Example 1

A. A wax-polyethylene blend was prepared as follows:
18.7 pounds of synthetic hard paraffin wax ("Paraffin") and 21 pounds of refined natural paraffin wax having a melting point ranging between 145°F. and 155°F. were placed in a steam jacketed kettle and melted at temperature of 110°C., with constant agitation until the mass had become a uniform liquid. Then 7 pounds of a polyethylene resin having a molecular weight of approximately 7,000 ("Epole 1100") was slowly added to the melted wax blend in the jacketed kettle until all ingredients were dissolved into a homogeneous hot melt.

B. An emulsification base for the wax-polyethylene coating composition was prepared as follows:
A primary emulsifier was first prepared as follows: 0.48 pounds of an oxygenated hard paraffin wax ("Hoechst Wax-5") and 0.1 pound of an aqueous 50% sodium hydroxide solution and 4.8 pounds of water were placed in a second steam jacketed kettle and the temperature of the ingredients was brought to the boiling point. The ingredients in this kettle were blended for about 30 minutes after which the weight of the ingredients was brought back to 5.38 pounds by adding water to replace the water evaporated during the boiling.

A secondary emulsifier was then prepared as follows: 1.8 pounds of casein, 45.2 pounds of water, 0.14 pound of concentrated ammonium hydroxide (28% NH₃), 0.2 pounds of sodium silicate solution (37.5% solids) and 0.11 pound of sodium metaphosphate were brought together in a third steam jacketed kettle, the temperature raised to approximately 212°F. and the mixture agitated until all ingredients were dissolved.
The ingredients in the second and third kettles were then mixed together to form the emulsification base with constant agitation at approximately 212°F. until a uniform solution of the emulsification base was obtained. The emulsified coating composition was prepared as follows: the wax-polyethylene blend in the first kettle was then added to the uniform solution of the emulsification base in a slow stream while the resulting mixture of the wax-polyethylene blend and the emulsification base was constantly agitated until a dispersion of the wax-polyethylene blend in the emulsification base was obtained. The dispersion of the wax-polyethylene blend in the emulsification base was then passed through a Manton-Gaulin Homogenizer at 4500 p.s.i. and a temperature of 100°C, was maintained. After homogenization, the resultant fine emulsion was passed through a heat exchanger which rapidly cooled the emulsion to below 40°C. To this emulsion, 0.32 pound of a 40% aqueous formaldehyde solution was added with constant stirring. The emulsion prepared in this example remained stable for a period of over 6 months.

Example II

A mixture containing 15 lbs. of the emulsified wax-polyethylene coating composition prepared in Example I, 15 lbs. of an aqueous emulsion of polyvinyl acetate homopolymer containing approximately 50% by weight of solids ("Flexac FA-5") and 15 lbs. of an aqueous emulsion of a polymer latex composed of polyvinyl acetate and acrylic acid containing approximately 30% by weight of solids ("Flexband F-306") was prepared by mixing the components at room temperature and stirring the resulting mixture until a homogeneous emulsion was obtained. The emulsion prepared in this example remained stable for a period of over 6 months.

Example III

The emulsified coating compositions of Examples I and II were applied as coatings to one side of samples of Brite-Pak Paper. The paper had a weight of 60 pounds per 1,000 square feet. Wire wrapped rods (Nos. 16 and 40) were used on separate sheets for this purpose. The coated paper samples were dried in an oven at 300°F. until all moisture was removed therefrom. After drying, the coated paper samples were removed from the oven and it was observed that the coated side of the paper had a very high gloss. These coated samples were extremely resistant to abrasion and scuffing. The adhesion of the coating compositions to the paper was excellent. The coated samples did not exhibit any reverse migration of the wax through the paper. The coating formed a film of at least about 1 mil in thickness on the surface of the paper and did not penetrate too deeply into the paper.

To demonstrate the improved characteristics of my novel coating compositions the paper coated with the compositions of Examples I and II were tested as follows: the aforementioned samples were placed face to face and face to back and heated in an oven for 3 hours at 65°C while having a pressure of approximately 20°C. No evidence of blocking was found in the paper samples so treated. Thereafter the coated samples were placed on a laboratory bench with the coating face upwards and blotters which were saturated individually with turpentine, water, castor oil and corn oil were placed upon the coated surfaces of the paper. The blotters which were saturated with these liquids were permitted to remain on the coated surface of the paper for a period of one week at which time the papers were then examined. It was found that none of the liquids contained in the blotters penetrated through the paper.

The coated samples of paper were then tested for flexibility by creasing the paper sharply and unbinding the creases several times. It was observed that no cracks or breaks appeared in the coatings applied to the paper.

Having described my invention what I claim as new and desire to secure by Letters Patent is:

1. An emulsified coating composition for application to paper and paperboard products, comprising (1) a wax-polyethylene blend comprising (a) a refined paraffin wax having a melting point ranging from about 122°F. to about 165°F. and present in amounts of from about 20% to about 52% by weight based upon the weight of said wax-polyethylene blend, (b) a synthetic hard paraffin wax having a melting point ranging from about 212°F. to about 225°F. and present in amounts of from about 35% to about 75% by weight, based upon the weight of said wax-polyethylene blend and (c) a polyethylene resin having an average molecular weight ranging from about 5,000 to about 12,000 and present in amounts of from about 6% to about 20% by weight based upon the weight of said wax-polyethylene blend and (2) an emulsification base comprising (a) a primary emulsifier selected from the group consisting of saponified oxygenated synthetic hard paraffin waxes having a solidification point ranging from about 185°F. to about 212°F. and saponified oxidized natural hard paraffin waxes having a solidification point ranging from about 185°F. to about 200°F. and present in amounts of from about 0.1% to about 7% by weight based upon the weight of said wax-polyethylene blend, (b) a secondary emulsifier which is a water solubilized protein present in amounts of from about 1.5% to about 15% by weight, based upon the weight of said wax-polyethylene blend and (c) water present in amounts of from about 80% to about 98% by weight based upon the weight of said wax-polyethylene blend.

2. The composition of claim 1, wherein said refined paraffin wax has a melting point ranging from about 148°F. to about 153°F. and is present in amounts of from about 44% to about 48% by weight, based upon the weight of said wax-polyethylene blend.

3. The composition of claim 1, wherein said synthetic hard paraffin wax is present in amounts of from about 40% to about 55% by weight, based upon the weight of said wax-polyethylene blend.

4. The composition of claim 1, wherein said polyethylene resin has an average molecular weight of about 7,000 and is present in amounts of from 12% to about 16% by weight, based upon the weight of said wax-polyethylene blend.

5. The composition of claim 1, wherein said secondary emulsifier is water solubilized casein.

6. The composition of claim 1, wherein said secondary emulsifier is water solubilized soya bean protein.

7. The composition of claim 1, wherein a water softening agent is present in said emulsification blend in amounts of from about 0.1% to about 0.5% by weight, based upon the weight of said wax-polyethylene base.

8. The composition of claim 1, including the additional component of an aqueous emulsion of a polymer latex selected from the group consisting of polyvinyl acetate, polybutadiene, polyisobutylene, polyvinylidene chloride, polyisoprene and mixtures thereof having a solidification point ranging from about 40% to about 60% by weight of the total, said aqueous emulsion of the polymer latex being present in a weight ratio with regard to said wax-polyethylene blend of from about 4:1 to 1:4 based on its solids content.

9. An emulsified coating composition for application to paper and paperboard products, comprising (1) a wax-polyethylene blend comprising (a) a refined paraffin wax having a melting point ranging from about 148°F. to about 153°F. and present in amounts of from about 44% to about 48% by weight based upon the weight of said wax-polyethylene blend, (b) a synthetic hard paraffin wax having a melting point ranging from about 212°F. to about 225°F. and present in amounts of from about 35% to about 75% by weight, based upon the weight of said wax-polyethylene blend and (c) a polyethylene resin having an average molecular weight ranging from about 5,000 to about 12,000 and present in amounts of from about 6% to about 20% by weight based upon the weight of said wax-polyethylene blend.

10. The emulsified coating composition of claim 9, wherein the ratio of the refined paraffin wax to the synthetic hard paraffin wax is from about 2:1 to about 3:1.
said wax-polyethylene blend and (c) polyethylene resin having an average molecular weight of about 7,000 and present in amounts of from about 12% to about 16% by weight based upon the weight of said wax-polyethylene blend and (2) an emulsification base comprising (a) a primary emulsifier selected from the group consisting of saponified oxygenated synthetic hard paraffin waxes having a solidification point ranging from about 185° F. to about 212° F. and saponified oxygenated natural hard paraffin waxes having a solidification point ranging from about 185° F. to about 200° F. and present in amounts of from about 0.1% to about 7% by weight, based upon the weight of said wax-polyethylene blend, (b) a secondary emulsifier which is a water solubilized cationic surfactant present in amounts of from about 1.5% to about 15% by weight, based upon the weight of said wax-polyethylene blend and (c) water present in amounts of from about 80% to about 98% by weight, based upon the weight of said wax-polyethylene blend and (d) a water softening agent present in amounts of from about 0.1% to about 0.5% by weight, based upon the weight of said wax-polyethylene blend.

10. The composition of claim 9, wherein there is present an aqueous emulsion of a polymer lattice selected from the group consisting of polyvinyl acetate, polybutadiene, polystyrene, polybutylacrylate, polyacrylic acid, polyvinylidene chloride, polystyrene and mixtures thereof having a solids content ranging from about 40% to about 60% by weight of the total, said aqueous emulsion of the polymer lattice being present in a weight ratio with regard to said wax-polyethylene blend of from about 4:1 to 1:4 based upon its solids content.

11. A process of preparing emulsified coating compositions for application to paper and paperboard products which comprises slowly adding the homogeneous mixture of the wax-polyethylene blend of claim 1 at a temperature ranging from 212° F. to about 225° F. to the emulsification base of claim 1 which is maintained at a temperature ranging from about 200° F. to about 212° F., agitating the resulting mixture of said blend and emulsification base to obtain an emulsion and thereafter stabilizing said emulsion by reducing the particle size of the particles contained therein to below 0.002 millimeter.

12. The process of claim 11 wherein said particles contained in said emulsion are reduced in size by passing said emulsion through a colloid mill.

13. The process of claim 11 wherein said particles contained in said emulsion are reduced in size by passing said emulsion through a homogenizer.

14. Paper and paperboard products coated with the emulsified coating composition of claim 8.

15. Paper and paperboard products coated with the emulsified coating composition of claim 10.

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