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## UNITED STATES PATENT OFFICE

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## ASPHALT EMULSION

No Drawing. Original application filed March 3, 1928, Serial No. 258,992. Divided and this application filed December 29, 1928. Serial No. 329,309.

and has for its objects the provision of certain improvements in the manufacture of asphalt emulsions and the provision as a new 5 article of manufacture of an improved as-

phalt emulsion. This application is a division of our Patent

No. 1,714,982.

- The asphalt emulsions of commerce are 10 for the most part mixtures of asphalt and water with various agents designed to effect and maintain an emulsification of the as-phalt and water. Colloidal clays have been largely used commercially as so-called emulsi-
- 15 fying agents, while numerous other substances have been suggested in the patent and other literature, such for example as colloidal vegetable and animal substances, soaps, alkalies, etc., which are true emulsifying 20 agents. Relatively large amounts of these
- agents have been prescribed as necessary in order to produce a reasonably stable asphalt and water emulsion, and/or suspensions in the case of clays and the like.
- We have discovered that tri-sodium phos-25 phate crystalline (Na<sub>3</sub>PO<sub>4</sub>+12H<sub>2</sub>O), or dehydrated tri-sodium phosphate (Na<sub>3</sub>PO<sub>4</sub>) in relatively small amounts effects very satis-
- factory and true emulsification of asphalt 30 and water. We have moreover found that this emulsifying action is only effective when limited quantities of tri-sodium phosphate are used. In other words, there appears to be a critical relationship between the amount
- of tri-sodium phosphate used and the effec-tiveness of the emulsification, whereby satisfactory emulsification can only be obtained when the amount of tri-sodium phosphate employed is between critical predetermined
- 40 limits, which vary with different kinds of asphalts. Thus, when the amount of tri-sodium phosphate employed as the emulsify-ing agent is increased beyond about 0.4%Na<sub>3</sub>PO<sub>4</sub> calculated by weight with respect to
- 45 the emulsion in the case of a certain grade of Bermudez asphalt, the resulting mixture of asphalt and water while possessing cer-tain aspects of an emulsion is a markedly inferior product to that obtained when less 50 than 0.4% or about 0.24% of Na<sub>3</sub>PO<sub>4</sub> is em-

This invention relates to asphalt emulsions ployed as the emulsifying agent. While with a California steam distilled asphaltic base oil residual asphalt the best emulsion is obtained when using more than 0.4% but less than 0.6% Na<sub>3</sub>PO<sub>4</sub>.

Our present invention, based on the foregoing discoveries, involves the emulsification of asphalt and water by the emulsifying action of relatively small amounts of tri-sodium phosphate, preferably not exceeding 0.24% 60 Na<sub>3</sub>PO<sub>4</sub>, by weight on the weight of the finished emulsion, in some cases. In the practice of the invention we have secured excellent results with from 0.1 to 0.3% of Na<sub>3</sub>PO<sub>4</sub>, calculated by weight with respect 65 to the weight of the emulsion. In no case should the tri-sodium phosphate exceed about 0.75% Na<sub>3</sub>PO<sub>4</sub> by weight on the emulsion, and the optimum results will usually be obtained when the amount of tri-sodium 70

phosphate is considerably less than 0.6% The following examples illustrate the practical application of the principles of the invention

100 kilograms of water are heated approxi- 75 mately to boiling, as for example by the injection of live steam. One kilogram of crystalline tri-sodium phosphate equal to about 0.4 kg. Na<sub>3</sub>PO<sub>4</sub> is added to this hot water. 100 kilograms of freely flowing molten as- 80 phalt are then gradually run into the hot water containing the dissolved tri-sodium phosphate. The mixture is vigorously stirred while the molten asphalt is being added to the water. The stirrer used for this pur- 85 pose should be of a fairly active type, such for example as a propeller stirrer, and should be arranged to disseminate the asphalt promptly after it contacts with the water. When the asphalt has been thoroughly and 90 uniformly dispersed throughout the water, the mixture is run through an emulsifying machine, such, for example, as a colloid mill. The resulting emulsion discharged from the emulsifying machine is relatively hot and 95 while cooling to room temperature should be gently stirred, with or without artificial cool-

As another example of the application of the invention, we will describe the manufac- 100 1,757,088

ture of an emulsion containing three parts of the invention we will describe the manufacasphalt and one part of water. The water (250 kilograms) containing four (4) kilograms of crystalline tri-sodium phosphate (1.6 kg.  $Na_3PO_4$ ) is heated to a temperature of about 80° C. The molten asphalt (750 kilograms) at about 135° C. is introduced into the water in a regulated stream and with continuous agitation from a fast moving proto peller agitator. If the asphalt is added too rapidly, the emulsion may boil over, also the asphalt may "float" because it is not taken up by the water as fast as it is fed. These conditions should be guarded against since 15 it is preferable to get the asphalt completely emulsified at the time it enters the water. The preliminary coarse emulsion formed in this way is now fed, continuously and with appropriate stirring, into a mechanical emul-20 sifier, such as a colloid mill, from which the finished fine emulsion is continuously discharged. The emulsion discharged from the is particularly applicable in preparing emulemulsifier is cooled, artificially if desired, with gentle stirring and canned, or other-wise prepared for storage or use. The oper-25 wise prepared for storage or use. ations of cooling and canning are preferably conducted in a continuous manner to prevent asphalt skin formation caused by undue exposure of the warm and rich emulsion to air. The cold emulsion resulting from the fore-going operation is of a dark brown color and 30 of a heavy consistency but will yet flow which is remarkable considering the high asphalt content (3 asphalt to 1 water) and the small 35 amount of emulsifying agent (1.6 parts  $Na_3PO_4$  to 750 parts asphalt or 1000 parts emulsion). The emulsion, while very stable in the cans, reforms true asphalt in a very few minutes when coated on a concrete wall, <sup>40</sup> cardboard, metal, etc.; the coating being just as water-proof, black and shining as when

- formed from non-emulsified asphalt. The concentrated emulsion may, if desired, be thinned with water to form a more dilute 45 emulsion. For example, two parts of the emulsion may be mixed with one part of
- water to make a thin brown emulsion of milk-like consistency containing about 50% pure asphalt.
- As a third example of the application of 50 the invention, we will describe the manufacture of an emulsion containing 60% asphalt and made from a California steam distilled asphaltic base oil residual asphalt.
- 55 The water (67 kilograms) containing 1 kilogram  $Na_3PO_4$  is heated to a temperature of about 90° C. The molten asphalt (100 kilograms) at about 150° C. is introduced into the water in a regulated stream and with con-
- 60 tinuous agitation from a fast moving propeller agitator. The emulsion discharged from the emulsifier is cooled very quickly by artificial means and canned or otherwise prepared for storgae or use.
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ture of an emulsion containing 60% asphalt and made from a straight Mexican steam distilled asphaltic base oil residual asphalt. The water (67 kilograms) containing  $0.75_{70}$ kilograms crystalline tri-sodium phosphate (0.3 kilograms  $Na_3PO_4$ ) is heated to a temperature of about 90° C. The molten asphalt (100 kilograms) at about 150° C. is introduced into the water in a regulated stream 75 and with continuous agitation from a fast moving propeller agitator. The emulsion discharged from the emulsifier is cooled very. quickly by artificial means and canned or otherwise prepared for storage or use. It 80 has also been found that even smaller amounts of tri-sodium phosphate may be advantageously employed.

While the invention is of general application and can be advantageously practiced s5 with substantially all asphaltic materials, it sions of asphalts containing relatively small percentages of mineral matter. Thus, for example, natural or residual Bermudez 90 asphalts, California or similar asphaltic base oil residue asphalts, or cut-back asphalt cements thereof, containing around 1 to 7% of mineral matter are admirably suited for the practice of the invention. Similarly, the 95 residual asphalts from the distillation of other asphaltic base oil, or cut-back asphalt cements thereof, are well suited for the practice of the invention. Emulsions made of such asphalts contain relatively small 10 amounts of mineral or similar inert matter, and consequently contain maximum percentages of the desirable hydrocarbon compounds of asphaltic materials. Moreover, in consequence of the small amount of tri- 105 sodium phosphate used, the emulsions are not contaminated with any substantial quantity of emulsifying agents.

The melting point of the asphalt used in practicing the invention should not be sub- 110 stantially higher than the boiling temperature of water, since otherwise objectionable steaming of the water will take place upon mixing the water and molten asphalt. Since 115the melting point of asphalt is to some extent dependent upon its hardness, as determined by its penetration factor, it follows that in general it is desirable to use asphalts  $\mathbf{of}$ relatively high penetration factors. 120 Where the penetration factor is too low, the asphalt may be cut-back with hydrocarbon oils, thereby producing what is known as asphalt cement, to appropriately increase its penetration factor. We have secured excel- 125 lent results in the practice of the invention using Bermudez asphalt products having a penetration factor of 30 and upwards at pared for storgae or use. 25° C., as determined by the Dow penetrom-As a further example of the application of eter and with the aforementioned California 134

penetration factor of 70.

The asphalt emulsions of the invention are characterized by their relatively low viscosity, considering their high asphaltic contents. Thus, the emulsions of the invention are freely flowing liquids, as contrasted with the paste-like products of equivalent asphalt content that have heretofore appeared on the market. Moreover, the products of the invention are true emulsions in which the asphalt is uniformly dispersed in a very fine state of subdivision. Under a high powered microscope, the emulsions of the invention 15 are seen to consist of minute globules of

asphalt very uniformly distributed throughout the water, which constitutes the continuous or external phase of the emulsion.

The emulsions of the invention are more-20 over characterized by their brown color,

thereby further evidencing the very fine state of subdivision of the black dispersed phase (asphalt). The color of an asphalt-water emulsion becomes lighter (i. e., varying from 25 black to brown) as the asphalt becomes more

- finely subdivided and dispersed in the water. This is believed to be due to the increased reflection and scattering of light in consequence of the increased subdivision and dis-
- 30 persion of the asphalt particles. This color phenomenon is utilized in the preparation of the emulsions, and the operator judges the completeness of the dissemination of the asphalt throughout the water and hence the
- 35 desired end-point in the mixing and/or emulsifying operations by the brown color of the resulting product.

The emulsions of the invention are furthermore characterized by their relatively rapid 40 drying qualities, that is, their ability, upon application in the form of a thin coating, to "set" or reform within a few minutes into an impermeable coalescent coating of true pure asphalt, typified by its shining black appear-45 ance. When applied to a surface with a spray

- gun, the asphalt dries and sets almost immediately, and when applied by a brush or the like, dries and sets in a very few minutes. The drying and setting of the asphalt is more
- **50** rapid, the more porous the material to which the emulsion is applied. This rapid drying quality of the emulsion of the invention is due, we believe, to the high degree of subdivision and dissemination of the asphalt where-55 by greatly increased surface areas are ex-

posed for evaporation of the water.

The emulsions of the invention, when applied and dried, produce an asphalt coating of characteristic black and glossy appearance. 60 This is largely due to the fact that the coat-

ing consists of pure asphalt uncontaminated with any substantial amounts of emulsifying agents or other inert substances. This is in striking contrast with the so-called asphalt emulsions containing clay and the like, which

asphaltic base oil residue asphalts having a when dry have a dull, flat appearance. This absence of any substantial amount of emulsifying agents or other inert substances in the asphalt coating produced by the emulsions of the invention, results in securing the 70 maximum effectiveness of the asphalt as a waterproofing or protective medium. It is to be understood, however, that non-emulsion breaking fillers may be added where very thick coatings are desired and suitable fillers 75 giving an initially water permeable char-acteristic may be used so that the water phase may evaporate or flow off, for if a heavy coating of the pure emulsion be attempted, the outer surface or skin thereof under certain so conditions as applied to certain materials would not allow the water to evaporate after such a skin had been formed.

The emulsions of the invention are very stable and have no tendency to break down 85 on standing. When stored in cans or other receptacles, some slight settling may take place, but the relatively thin layer of supernatant liquor is readily mixed in and the uniformity of the emulsion re-established by 20 simply stirring. The emulsions, more par-ticularly those of relatively high asphalt content, can be readily diluted or thinned with water accompanied by appropriate stirring to produce more fluid or less con- 95 centrated emulsions, as desired.

We do not desire to be restricted to any theory of the effectiveness of small amounts of tri-sodium phosphate in emulsifying water and asphalts, and the fact remains that 100 small amounts of tri-sodium phosphate produce remarkably effective emulsification of asphalt and water. We believe that the effective emulsifying action of small amounts of tri-sodium phosphate, characteristic of the 105 present invention, is due in part to the chemical, and perhaps physical, composition and behavior of tri-sodium phosphate, as well as to its presence in amounts preferably not exceeding around 0.6% (Na<sub>3</sub>PO<sub>4</sub>) by weight 110 on the emulsion, and in no case exceeding about 0.75%.

It should be noted that in accordance with our invention, the complete emulsification may be effected with a fraction of one per cent 115 (1%) of tri-sodium phosphate. Other agents or substances may, however, be added to the emulsion for imparting thereto qualities or properties quite apart from that of emulsification. In this connection, however, care 120 must be taken not to add substances that exercise a deleterious effect upon or break down the emulsion. In all cases, however, the additive emulsifying agent will not substantially exceed 1.5% and the emulsion will 125 consist principally of water and asphalt. In their preferred form, employing asphalts of low mineral content, the emulsions of the invention contain by weight over 90%, and preferably not less than 95%, of combined 130

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water and asphaltic constituents soluble in carbon bisulfide.

While we have particularly mentioned trisodium phosphate, other water-soluble alkaline phosphates of monovalent metals, such

as potassium phosphates of monovalent metals, such as potassium phosphate and/or ammonium phosphate or mixtures of the same may be employed as the emulsifying agent, and are to be understood as included in the scope of
the appended claims. The relative cheapness and commercial availability of tri-sodium

and commercial availability of tri-sodium phosphate, however, make it a convenient agent in practice. Also the separate addition of the necessary component metal salts and 16 a phosphoric reagent may under certain con-

ditions be employed.

We claim: <sup>–</sup>

1. An asphalt-water emulsion containing tri-sodium phosphate as an emulsifying 20 agent in amount not exceeding 0.75% Na<sub>3</sub>PO<sub>4</sub>

by weight on the emulsion. 2. An asphalt-water emulsion containing tri-sodium phosphate as an emulsifying agent in amount not exceeding 0.3% Na<sub>3</sub>PO<sub>4</sub>

- 25 by weight on the emulsion.
  3. An asphalt-water emulsion containin
  - 3. An asphalt-water emulsion containing about 0.1 to 0.3% by weight of  $Na_3PO_4$  as an emulsifying agent.
- 4. An asphalt-water emulsion containing 30 tri-sodium phosphate as an emulsifying agent in amount not exceeding 0.3% Na<sub>3</sub>PO<sub>4</sub> by weight on the emulsion and characterized by substantially uniform dispersion of the asphalt in the form of minute globules and
- 35 further containing not less than 90% by weight of combined water and asphalt constituents soluble in carbon bisulfide and not less than one part by weight of asphalt for each part by weight of water.
- 5. An asphalt-water emulsion containing tri-sodium phosphate as an emulsifying agent in amount not exceeding 0.3% Na<sub>3</sub>PO<sub>4</sub> by weight on the emulsion and characterized by substantially uniform dispersion of the
- 45 asphalt in the form of minute globules and further containing not less than 90% by weight of combined water and asphalt constituents soluble in carbon bisulfide and at least two parts by weight of asphalt for each part by weight of water.
- 6. An asphalt-water emulsion containing as an emulsifying agent a tri-basic phosphate of an alkali metal in amount not exceeding the equivalent of 0.75% Na<sub>3</sub>PO<sub>4</sub> by weight on the emulsion.

7. An asphalt-water emulsion in the production of which a tri-basic phosphate of an alkali metal has been added in amount not exceeding the equivalent of 0.75% Na<sub>3</sub>PO<sub>4</sub>
60 by weight on the emulsion.

8. An asphalt-water emulsion in the production of which tri-sodium phosphate has been added as an emulsifying agent in amount not exceeding 0.75% Na<sub>3</sub>PO<sub>4</sub> by 65 weight on the emulsion.

9. An asphalt-water emulsion in the production of which tri-sodium phosphate has been added as an emulsifying agent in amount not exceeding 0.75% Na<sub>3</sub>PO<sub>4</sub> by weight on the emulsion and characterized by substantially uniform dispersion of the asphalt in the form of minute globules.

In testimony whereof we affix our signatures.

ARTHUR L. HALVORSEN. 75 PIERCE M. TRAVIS. 75

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