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3,442,808

LUBRICATING OIL ADDITIVES

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12 Claims

ABSTRACT OF THE DISCLOSURE

Lubricating oil additives prepared by reacting alkenyl succinic anhydride with the Mannich condensation product prepared by condensing alkyl substituted phenol, formaldehyde and polyalkylene polyamine. The additives can optionally be further reacted with a boron compound such as boric acid, boric anhydride or a boric acid ester.

This invention concerns new chemical compositions and lubricating oil compositions containing the same. The subject matter of the instant invention also concerns a novel class of chemical compositions which act as dispersants when incorporated as additives into engine lubricating oils. The invention particularly concerns new Mannich condensation products.

The prior art portrays a need for improved engine lubricating oil additives which function as dispersants to retain in suspension insoluble particles such as the products of fuel combustion, those of incipient oxidation, and those of lubricant deterioration. The control of the deterioration of lubricating oils has long been a problem particularly in the formulation of the compositions of lubricating oil additives.

It has been known for many years that deterioration of hydrocarbon lubricating oils causes the formation of substances which deleteriously affect the surfaces which the lubricants are developed to protect through the maintenance of reduced frictional forces between said surfaces. Lubricating oil deterioration not only causes the formation of surface-deleterious substances, but it also results in the agglomeration of sludge-like materials in the lubricants and the deposition of varnish on the surfaces.

For many years it has been a practice to include as additives in lubricating oils, chemical compositions which tend to reduce oil deterioration and the consequent formation of the sludge and varnish-like materials. Broadly included in the classification of additives are corrosion and oxidation inhibitors, and dispersing agents. These additives generally function to reduce the corrosion of the surfaces to be protected, to stabilize the lubricating oil, to inhibit deterioration by oxidation, and to function as dispersants which tend to prevent the agglomeration of sludge and the deposition of varnish and sludge caused by the formation of the products of deterioration.

Frequently, the inclusion into lubricating oils of corrosion, oxidation, and dispersant agents is highly desirable; however, the problems of compatibility of these three or more functional agents creates serious problems in the formulation of useful additive-containing lubricating oils. Much research and development effort has been expended in the search for improved lubricating oil additives which function in the above-enumerated manner. The research efforts have been directed toward the development of three or more separate agents, as well as a single agent, or a plurality of compatible agents that will perform all of the desired functions when incorporated into hydrocarbon lubricating oils.

Various Mannich condensation products are known in the prior art as lubricating oil additives; however, the

teachings of the disclosures indicate that these Mannich products have previously been considered for use as oxidation inhibitors and as anti-wear additives. The former tend to reduce the rate of formation of oxidation products and the latter tend to coat surfaces with films and reduce friction resulting in less wear; consequently, there is some reduction of particulate and product contamination of the lubricating oil medium. It is also well known that various Mannich products produced for use as oxidation inhibitors and anti-wear additives demonstrate limited solubility in lubricating oil, which factor is also an undesirable characteristic of an additive. In view of the above, it has long been attempted to develop Mannich products and derivatives thereof which have novel functional properties as well as improved solubility in lubricating oils when incorporated therein.

Accordingly, it is an object of this invention to provide new compositions of matter.

It is an additional object of this invention to provide new compositions of matter which are useful as dispersants in lubricating oil compositions.

Another object of this invention is to provide lubricating oil dispersants which have improved solubility in said oil and which exhibit superior compatibility with other oil included additives.

An additional object of this invention is to provide a novel process for the preparation of compositions useful as dispersants.

Another object of this invention is to provide improved lubricating compositions.

Other objects and advantages of the invention disclosed and claimed herein will become apparent from the specification, including various embodiments, examples, and the claims.

It has been discovered that the solubility of Mannich condensation products is appreciably improved by reacting the Mannich products with a polyalkenyl succinic anhydride at a temperature sufficient to remove water of condensation. The modified Mannich reaction products impart excellent dispersant properties to the lubricating oil compositions while maintaining an unusually high degree of solubility within said lubricating oil compositions. The invention includes the reaction of an alkylphenol, formaldehyde, and a polyalkylene amine to form a Mannich product. The ratio of phenol, formaldehyde, and amine is from about 1:1:0.5 to about 1:2.5:2. The conventional Mannich product so formed is known to have anti-wear and oxidation-inhibiting properties but limited oil solubility. This conventional Mannich product is then reacted with a polyalkenyl succinic anhydride to yield the highly effective low-temperature dispersants for lubricating oils. The Mannich polyalkenyl succinic anhydride reaction product can be treated with a small amount of a boron-containing compound to provide another modified Mannich reaction product. Boration further improves compatibility with other additives. Each of these products exhibits surprising and superior dispersant properties when incorporated into lubricating oils. Each of these products also maintains a high degree of solubility with the lubricating oil additive-containing matrix.

By polyalkenyl succinic anhydride is meant the reaction product of maleic anhydride with a polymer of a monoolefin having three or four carbon atoms, such as propylene or butylene. The polymers should be a viscous liquid polyolefin having a viscosity measured in Saybolt Universal Seconds (SUS) at 210° F. of from about 50 sec. to about 10,000 sec. Such viscous liquid polypropylenes and polybutylenes (including polyisobutylenes) are well known to those skilled in the art and they can be conveniently prepared, for example, by polymerizing propylene-and-butylene containing hydrocarbon fractions

in the presence of aluminum chloride or other Friedel-Crafts catalysts. These viscous liquid polypropylenes and polybutylenes contain a limited amount of residual unsaturation so that they can be reacted with maleic anhydride to form an alkenyl succinic anhydride. These polypropylenes and polybutylenes can be reacted with substituted maleic anhydrides with other anhydrides of similar unsaturated aliphatic dicarboxylic acids to form corresponding alkenyl derivatives.

Typically a polybutenyl succinic anhydride can be prepared by reacting about 0.85 mole of maleic anhydride per mole of polybutene, the polybutene having a molecular weight of about 860. Although not all of the maleic anhydride enters into the reaction at about 450° F., only about 5 weight percent is thermally decomposed and a small amount is stripped out. The yield of polybutenyl succinic anhydride is about 50–80% based on the polybutene used.

Suitable Mannich products for modification according to the process of this invention are prepared by condensing an alkylphenol, formaldehyde, and a polyalkylene amine. The alkylphenol may be a C₁–C₆₀ alkyl substituted phenol, preferably a C₉ alkyl substituted phenol (nonylphenol). the polyalkylene polyamine is derived from ethylenediamine or condensation products of ethylenediamine, such as diethylene triamine, triethylene tetramine, tetraethylenepentamine, or pentaethylene hexamine. Tetraethylenepentamine (TEPA) is the preferred polyalkylene polyamine.

Example I.—Preparation of conventional Mannich product

The preparation of a conventional Mannich product is a condensation of TEPA with nonylphenol and formaldehyde. Three thousands three hundred twenty nine lbs. of TEPA, 17.6 lb. moles, is added to a 2,000 gallon kettle blanketed with an inert gas such as nitrogen. One thousand nine hundred thirty six lbs. of nonylphenol is added over a 30-minute period with thorough mixing and the temperature is adjusted to within the range of about 100–110° F. One thousand four hundred twenty five lbs. of 37% aqueous formaldehyde containing 17.6 lb. moles of CH₂O, is charged to the above mixture over a period of about three hours. The temperature will rise to about 130–150° F. and the reacting material is held for about two hours with stirring. Subsequently, the reactants are heated to about 300° F. in the presence of an inert gas such as nitrogen and about 145 gallons of water is distilled therefrom. The reacting material is held at 300° F. for about one hour prior to cooling to ambient temperature.

The properties of the above reaction product are as follows:

Activity, percent	100
Nitrogen, percent	20–21
Gravity, lb./gal.	8.53
Vis. 210° F., SSU	333
Flash, ° F.	420
Molecular weight	622

Example II.—Modification of conventional Mannich product

The Mannich product described above is modified by reaction with a polybutenyl succinic anhydride to form the dispersant additive of this invention in the following manner.

Seven thousand one hundred twenty pounds of about 54% polybutenyl succinic anhydride, 4.0 lb. moles, prepared in the manner described above in SAE 5W lubricating oil diluent, is charged to a 2000 gallon kettle blanketed with an inert gas such as nitrogen and 4,316 pounds of SAE 5W lubricating oil is added and the temperature is adjusted to within the range of about 260–280° F. One

thousand three hundred sixty eight lbs. of the Mannich condensation product prepared in accordance with the foregoing description is added over a period of about two hours during which the temperature increases about 20° F. The material is heated to about 300° F. within about one hour, and the reactants are held at about 300° F. for three hours in the presence of an inert gas such as nitrogen. Subsequently, the material is filtered through a sparkler filter.

The properties of the above reaction product as follows:

Activity, percent	40
Nitrogen, percent	2.1
Gravity, lbs./gal.	7.56
Vis. 210° F., SSU	600
Flash, ° F.	380

Example III.—Alkylenyl succinimide modified Mannich

About 648 grams (6 moles) of para cresol and 2270 grams (12 moles) tetraethylenepentamine were combined in a 5-liter flask equipped with a stirrer. About 972 grams of 37% formaldehyde (12 moles) were added over a period of four hours. The exothermic heat increased the reaction temperature to about 133° F. The product was then heated between about 311° F. and 327° F. with nitrogen blowing over a period of about ten hours. A total of about 570 ml. of water were distilled out. The product yield was 3000 grams.

About 382 grams (.75 moles) of the above Mannich product were dissolved in 300 grams of chloroform and combined with 2580 grams of polybutenyl succinic anhydride containing a polybutenyl substituent having a viscosity of about 1000 SUS at 210° F. (1.5 mols) and 750 grams of 5-grade oil in a 5-liter flask and heated between about 210–302° F. for about ten hours. The volatile material was allowed to escape as vapor. The yield of this product was 3625 grams and it contained 3.41% nitrogen and 0.15% chlorine.

Example IV.—Alkylenyl succinimide modified Mannich

648 grams (6 moles) of mixed cresol were placed in a 5-liter flask equipped with a stirrer. Through a dropping funnel, 2270 grams (12 moles) of tetraethylenepentamine was added. A small exothermic heat was observed. 972 grams (12 moles) of 37% formaldehyde was added dropwise. The mixture was heated between about 122–302° F. for 16 hours with nitrogen blowing. 345 ml. of water were condensed in a trap. The yield of product was 3121 grams. The nitrogen content was 25%.

The polyalkylenyl succinic anhydride modification of the above Mannich was prepared as follows. 382.5 grams of the above Mannich (.75 mole) was added to a mixture of 2580 grams of 54% active polybutenyl succinic anhydride containing a polybutenyl substituent having a viscosity of about 1000 SUS at 210° F. (1.5 moles) and 564 grams of 5-grade oil in a 5-liter flask. The mixture was heated with nitrogen blowing for about ten hours between about 77 and 302° F. The yield of product was 3452 grams and contained 2.70% nitrogen.

Example V.—Alkylenyl succinimide modified Mannich

Phenol was alkylated with polypropylene as follows. About 1600 grams (2 moles) of polypropylene, 1-liter of hexane and 210 grams of a phenol-BF₃ complex (about 2 moles) were reacted in a 5-liter flask at ambient temperatures for approximately two hours. The product was washed with water and steam stripped to about 302° F., then dried by blowing with an inert gas. The yield was 1657 grams, the molecular weight 885, and the hydroxyl number was 51.

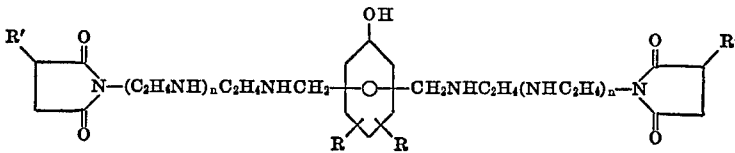
The Mannich product of the above phenol was prepared as follows. 1600 grams of the alkyl phenol (1.8 moles) and 681 grams of tetraethylenepentamine (3.6 moles)

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were combined in a 5-liter flask equipped with a stirrer. About 294 grams of 37% formaldehyde (3.6 moles) were added dropwise. The mixture was heated with stirring for about ten hours between about 122 and 144° F. About 170 mls. of water were collected in a trap. The yield was 2222 grams.

The succinimide of the above Mannich was prepared as follows. About 1000 grams of the Mannich (.772 mole) was dissolved in 100 mls. of toluene and added dropwise to 2660 grams of a polybutenyl succinic anhydride containing a polybutenyl substituent having a viscosity of about 1000 SUS at 210° F. (1.54 moles) heated to about 257° F. in a 5-liter stirred flask. The mixture was heated with nitrogen gas blowing for about ten hours at about 257-302° F. The yield was 3623 grams. The product contained 2.78% nitrogen.

By way of mere illustration and not limitation, the composition of the modified Mannich reaction products disclosed herein may be shown structurally in accordance with the following:



wherein R' is a polyolefin constituent derived from a mono-olefin having three to four carbon atoms, preferably a polybutylene having a viscosity at 210° F. of from about 50 to 10,000 SUS; *n* equals 0-10, preferably about 3; and R and R₁ are hydrogen or a C₁-C₆₀ alkyl-substituent, preferably, R being hydrogen and R₁ being C₉.

The aforementioned boron modification of the modified Mannich product may be carried out with boric acid, a boric acid ester, a boric anhydride, etc., at a temperature of from about 120-400° F., using from 0.2 to 2.0 or more moles of boron per mole of modified Mannich reaction product. Although up to as high as 1.5 atoms of boron per gram atom of nitrogen can be reacted, it is desirable to have a boron to nitrogen (B/N) ratio within the range of about 0.05 to 1.0, preferably the (B/N) weight ratio is in the range of 0.10 to 0.5, inclusive.

Lubricating oils containing Mannich condensation products in accordance with this invention were tested in the Lincoln M.S. V Test Sequence designed by Ford Motor Company. The Lincoln Sequence test procedure evaluates low temperature dispersancy characteristics of a lubricating oil. Briefly, the test consists of using the oil to be tested as a lubricating oil in a V-8 Lincoln engine under prescribed test conditions. Accordingly, 5 quarts of oil are placed in the crankcase and the engine is started and run in accordance with the 4-hour cycle:

	Phase 1	Phase 2	Phase 3
Duration (min.)	45	12	7
Speed, r.p.m.	500	2,500	2,500
Load, brake horse power	No Load	105	105
Temperature, ° F.:			
Water jacket	115-120	125-130	170-170
Oil sump	120-125	175-180	205-215
Air/fuel ratio	9.5±0.5	15.5±0.5	15.5±0.5

The 4-hour cycle is repeated a total of 48 times (192 hours running time). After each 16 hours of operation the engine is shut down for 8 hours. The oil level is adjusted with fresh oil to a level of 5 quarts. At the termination of the test, the hot oil is drained. The engine is then disassembled and examined for deposits of varnish and sludge among other observable results.

In the Lincoln M.S. Test Sequence V, after the formulated lubricant oil has been tested and the engine disassembled for inspection of the parts thereof for sludge and varnish deposits among other inspections, the total

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varnish and sludge ratings are made from the individual ratings of the following engine parts.

Varnish:

Piston
Rocker arm cover
Push rod cover
Cylinder walls
Oil pan

Sludge:

Push rod chamber
Rocker arm assembly
Rocker arm cover
Oil screen
Oil pan
Push rod cover
Timing gear cover
Valve deck

The varnish and sludge rating for each and every one of the foregoing parts is made. Then an average rating is made for each type of part, for example, the varnish

ratings for each of the eight pistons are added and divided by 8 for the average piston varnish. The sum of the average ratings for each class of parts is found for both sludge and varnish. The rating of total varnish is the sum of the average ratings of the five classes of parts. A varnish rating of 7.0 or higher for each class of parts is considered to be passing.

The rating of total sludge is the sum of the average ratings for the eight classes of parts multiplied by 3/4 since the perfect total sludge rating is 50. The perfect total varnish rating is also 50.

Lincoln Engine Tests 1, 2, and 3

In the first and second formulations subjected to the Lincoln Sequence Test the blend of additives included 3.75% of the Mannich condensation product made in accordance with the teachings of EXAMPLE II and was combined with 1.0% of an overbased magnesium sulfonate that is readily available commercially and 1% of a zinc dialkyldithio-phosphate oxidation inhibitor also commercially available. The third formulation included 2.5% by weight of the novel dispersants of this invention and the other additives of formulations 1 and 2 were present in the same amounts. All three tests were effective in demonstrating the high performance of the novel dispersant additive as shown below in Table I.

TABLE I.—LINCOLN MS V RESULTS

Oil formation	Sludge rating	Varnish rating	Oil ring filling, percent	Oil screen plugging, percent
1	42	43	0	2
2	42	43	0	2
3	32	45	2	2

Engine tests 4, 5, 6, and 7

From these tests, data was obtained on the novel additive of this invention. The tests were conducted in the Ford 289 cubic inch displacement engine in a manner similar to the above-described Lincoln tests.

In formulations 4 and 5, the following additive compositions were used in a SAE 20 base oil. The novel dispersant of EXAMPLE II was present in the amounts of 6.0 weight percent and 5.0 weight percent in formulations 4 and 5. Formulation No. 4 also contained 2 weight percent overbased calcium sulfonate, 0.4% basic calcium

phenate and 1% by weight zinc dialkyldithio-phosphate. Formulation No. 5 included 1% overbased magnesium sulfonate and 1% zinc dialkyldithio-phosphate.

In formulations Nos. 6 and 7 ingredients were added to a SAE 30 lubricating oil. In formulation 6 the additives included 5% of the novel dispersant, 1% of an overbased calcium sulfonate and 1% of a zinc dialkyldithio-phosphate. The combination of additives in formulation 7 included 6.0% of the novel dispersant of this invention, 4% basic calcium phenate, 2% of an overbased calcium sulfonate and 1% of a zinc dialkyldithio-phosphate. All the constituents except the novel dispersant of this invention are readily available on the open market. The test results of these formulations were effective in demonstrating the superiority of the novel dispersants as shown in Table II below.

TABLE II.—FORD 289 ENGINE TEST RESULTS

Oil formation	Sludge rating	Varnish rating	Oil ring filling, percent	Oil screen plugging percent
4.....	47.1	47.6	0	0
5.....	48.1	42.5	0	0
6.....	48.1	42.5	0	0
7.....	47.1	44.0	0	0

Field tests were conducted on several motor oils containing well-known commercially available ashless dispersants, and the novel dispersant of this invention in order to gather comparative data on effectiveness. The automobiles employed were 1965 model Ford Galaxies with 289 cu. in. displacement engines. The driving was about one-half city and one-half suburban. A readily available premium gasoline was used. The lubricating oils were SAE-10 base containing by vol. percent the following:

	Percent
Ashless dispersant.....	5
Polymethacrylate viscosity index improver.....	5
300 TBN calcium sulfonate.....	1
Alkenyl succinic acid.....	0.2
Zinc dithiophosphate.....	0.7

The instant novel dispersant disclosed and claimed herein is compared below with another ashless dispersant commercially available. The ratings for varnish and sludge are given on a scale where 10 is perfectly clean.

Dispersant	Instant	Other
Test miles (thousands).....	11	12
Rocker arm cover rating:		
Varnish.....	9.7	9.7
Sludge.....	9.8	9.7

The dispersants prepared as disclosed herein can be used as addition agents in lubricating oils and especially in lubricating oils for use in internal combustion engines. The addition agents impart excellent low temperature dispersancy characteristics to the lubricating oil. Although amounts of the dispersancy addition agent may be varied as desired, it is believed advantageous to use at least about 0.2 weight percent and up to 20 weight percent of the addition agent of this invention and preferably 0.5 to 10 weight percent in a suitable lubricating oil. Although the preferred lubricating oils are the mineral lubricating oils, the use of the additive compositions is not restricted thereto. Other lubricating oil bases can be used, such as hydrocarbon oils, both natural and synthetic, for example, those obtained by the polymerization of olefins, as well as synthetic lubricating oils of the alkylene oxide type and the mono- and polycarboxylic acid ester type, such as the esters of adipic acid, sebacic acid, azelaic acid; it is also contemplated that various other well-known additives, such as anti-oxidants, anti-foam agents, pour point depressors, extreme pressure agents, corrosion inhibitors, anti-wear agents, etc., may be incorporated in lubricating oils containing the additives of our invention.

Concentrates of a suitable oil base containing more than 10 percent, for example, up to 75 percent or more, of the

additive of this invention alone or in combination with other additives may be prepared and can be used for blending with hydrocarbon oils or other oils in the proportions desired for the particular conditions of use to give a finished lubricating product containing the additives of this invention. In order to exemplify the use of the reaction products prepared in accordance herewith as low dispersancy lubricating oil addition agents, examples of formulated lubricating oils containing such reaction products were prepared.

While in the foregoing specification this invention has been described in relation to preferred embodiment thereof, and many details have been set forth for the purpose of illustration, it will be apparent to those skilled in the art that the invention is susceptible to other embodiments and that many of the details set forth herein can be varied considerably without departing from the basic principles of the invention.

What is claimed is:

1. A composition comprising the reaction product of heating to a temperature sufficient to remove water of condensation, a Mannich compound obtained by condensing an alkyl substituted phenol, formaldehyde and a polyalkylene polyamine with 1-2 moles of alkenyl succinic anhydride, said alkenyl substituent being derived from a polyolefin having a viscosity at 210° F. of from about 50 to about 10,000 Saybolt Universal Seconds.

2. The composition of claim 1 reacted at a temperature of from about 120-400° F. with a small amount of boron containing compound selected from the group consisting of boric acid, boric acid esters and boric anhydride, in a mole ratio of from 0.2 to at least 2.0 moles of boron per mole of said composition of claim 1.

3. The composition of claim 1 wherein the phenol is a mono-substituted phenol.

4. The composition of claim 1 wherein the phenol is a di-alkyl substituted phenol.

5. A lubricant composition comprising a major proportion of a lubricating oil and from about 0.2-20% by wt. of the composition of claim 1.

6. The composition of claim 1 wherein the alkenyl substituent is a polybutene having a molecular weight within the range of about 300-10,000.

7. A composition comprising the reaction product of heating to a temperature of from about 77° F. to about 302° F. a Mannich compound obtained by condensing a nonylphenol, formaldehyde and tetraethylenepentamine with 1-2 moles of alkenyl succinic anhydride, said alkenyl substituent being derived from a polyolefin having a viscosity at 210° F. of from about 50 to about 10,000 Saybolt Universal Seconds.

8. The composition of claim 7 reacted at a temperature of from about 120-400° F. with a small amount of boron containing compound selected from the group consisting of boric acid, boric acid esters and boric anhydride, in a mole ratio of from 0.2 to at least 2.0 moles of boron per mole of said composition of claim 7.

9. A lubricant composition comprising a major proportion of a lubricating oil and from about 0.2-20% by wt. of the composition of claim 8.

10. A composition comprising the reaction product of heating to a temperature of from about 77° F. to about 302° F., a Mannich compound obtained by condensing a cresol, formaldehyde, and tetraethylenepentamine with 1-2 moles of alkenyl succinic anhydride, said alkenyl substituent being derived from a polyolefin having a viscosity at 210° F. of from about 50 to about 10,000 Saybolt Universal Seconds.

11. A lubricant composition comprising a major proportion of a lubricating oil and from about 0.2-20% by wt. of the composition of claim 10.

12. A process for producing a lubricant additive composition comprising the steps of reacting about 2 moles of alkenyl succinic anhydride, said alkenyl substituent being derived from a polyolefin having a viscosity of 210°

F. of from about 50 to about 10,000 Saybolt Universal Seconds, with about 2 moles of the Mannich product produced by condensing an alkyl substituted phenol, formaldehyde and a polyalkylene polyamine, at a temperature of from about 77° F. to about 302° F.

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PATRICK P. GARVIN, *Primary Examiner.*

U.S. Cl. X.R.

10 252—51.5; 260—326.3, 326.5, 519, 559

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,442,808 Dated May 6, 1969

Inventor(s) Thornton P. Traise, Randel Q. Little and Roger W. Watson

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 3, line 24, the word "the" should be capitalized; line 35, the word "thousands" should be --thousand--. Column 4, line 10, the word "as" (first occurrence) should be --are--. Column 5, line 54, the word "quartes" should be --quarts--; in the table, under "Phase 2", the first number should be --2-- instead of "12"; under "Phase 3" the first number should be --75-- instead of "7"; under "Phase 3", opposite "Water jacket" the numbers "170-170" should be --170-175--; opposite "Oil sump" the numbers "205-215" should be --205-210--. Column 6, Table I, first column should be headed --Oil Formulation-- instead of "Oil formation"; in the heading of the last column, "percen" should be --percent--. Column 7, Table II, first column, the heading "Oil formation" should be --Oil Formulation--; line 25, the word "mottor" should be --motor--. Column 8, line 12, the word "embodiment" should be --embodiments--; line 70, the word "lubriacting" should be --lubricating--; line 75, the word "of" should be --at--.

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