United States Patent

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[54] MULTIFUNCTIONAL LUBRICANT ADDITIVE COMPOSITIONS AND LUBRICATING OILS CONTAINING

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[51] **Int. Cl.** **C10m 1/48**

[58] **Field of Search** ..... 252/32.7 E, 33, 42.7

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[57] **ABSTRACT**

Balanced oil additive compositions can be used in the engine, gears and elsewhere of mobile equipment. Appropriate concentrations of basic detergents, dispersants, antioxidants, extreme pressure agents and friction modifiers are balanced.

12 Claims, No Drawings

MULTIFUNCTIONAL LUBRICANT ADDITIVE COMPOSITIONS AND LUBRICATING OILS CONTAINING

BACKGROUND OF THE INVENTION

The present invention relates to multifunctional lubricant additive compositions and to lubricating oils containing the same. More particularly, the invention relates to oil compositions suitable for use not only in the engine but also in the gears and elsewhere, of mobile equipment such as a vehicle powered by an internal combustion engine, especially a diesel engine.

The invention is particularly useful in the lubrication of tractors, especially large tractors, dumpers, graders and other heavy duty applicances, and in vehicles having common sump lubrication of engine and axle wherein a limited slip differential is used.

The art has developed an increasing variety of oil-based fluids designed or adapted to perform many specific functions. Among such functions are engine lubrication, power transmission, hydraulic control and lubrication of gears such as those in the final drive axle of a vehicle. Other such functions of particular interest in connection with the present invention, are the oil immersion of disc brakes, which we refer to as wet brakes, and use in power take-off facilities for instance on a tractor.

Whereas these various functions may be served individually by several separate oils each closely adjusted to a specific function, it is a disadvantage to have to deal with so many different oils in the operation and maintenance of one vehicle, being not only inconvenient but liable to lead to serious and costly error on the part of the less skilled operator. There is, therefore, a preference for single oil compositions which can perform satisfactorily more than one such function. This preference calls for additives or additive combinations for lubricants which are capable of fulfilling diverging demands.

Requirements of oils for various functions which give rise to imcompatibility with other functions, include those tabulated below.

ENGINE LUBRICATION

Detergents and dispersants for prevention of sludge, prevention of ring sticking and maintenance of general cleanliness.

Antioxidants.

Mild antiwear agents to protect the valve train.

TRANSMISSION:

Mild antiwear agents.

Critical viscosity requirements (for hydrostatic transmission)

HYDRAULIC SYSTEM

Mild antiwear agents in some cases.

Temperature/viscosity control additives.

POWER TAKE-OFF CLUTCHES

High enough static friction (to permit torque transmission).

FINAL DRIVE AXLE GEARS

Moderate to considerable antiwear and extreme pressure agents.

WET BRAKES AND LIMITED SLIP DIFFERENTIALS

Specific friction modifiers to prevent objectionable chatter.

The problems encountered in attempts to reconcile these requirements, are many. The detergents and dispersants commonly used in engine oils may interfere with the operation of antiwear and extreme pressure agents, and reduce axle gear protection. Long-chain friction modifiers necessary to reduce static friction and achieve acceptable wet brake anti-chatter

may break down in the engine and cause deposit problems. Excess friction modifier may prevent proper power take-off operation by reducing torque capacity; insufficient modifier will permit brake chatter. (Chatter is a 'stick-slip ' effect which appears when static friction in disc brakes is too high and dynamic friction is too low). Some of the most efficient friction modifiers, such as soaps, may interfere with axle gear protection.

The use of oil thickeners of a polymeric nature, for example polymethacrylates, will tend to set up top groove carbon filling problems in many diesel engines, and necessitate increased detergent.

The selection of detergents, extreme pressure agents and friction modifiers is thus a critical matter in respect of the nature, the relative balance, and the total dosage, of these ingredients.

Attempts have hitherto been made to satisfy the requirements of more than one of the aforesaid functions in a single oil Multifunctional tractor oils are known but they are deficient in axle gear protection and in any case such oils are not envisaged for use in wet brakes. Other multipurpose oils for application to wet brakes, transmission, hydraulic systems or axle gears, have not been envisaged for engine lubrication. A truly universal oil satisfying the requirements of all the aforesaid functions has not hitherto been considered.

It is an object of the present invention to provide an additive composition and an oil containing said composition suitable for use at least in the engine, and elsewhere, such as in powered axle gears and wet brakes, of a mobile powered appliance, for example a tractor.

In accordance with the present invention, there is provided an additive formulation for a multipurpose lubricating oil comprising a balanced combination of:

A. from about 2 to about 15 parts by weight of a composition comprising
   i. from about 50 to about 95 percent by weight of a mineral oil soluble or dispersible, basic detergent having a Total Base Number (TBN) of at least about 75,
   ii. from about 5 to about 50 percent by weight of a mineral oil soluble antioxidant selected from a metal alkyl dithiophosphate and a phenol, and

B. from 1 to about 8 parts by weight of a composition comprising
   i. from about 30 to about 95 percent by weight of a sulfurized C_{12}-C_{24} fat, or
   ii. from about 30 to about 95 percent by weight of a combination of from about 50 to about 80 percent by weight of a sulfur-containing compound selected from the group consisting of alkyl sulfides and alkyl polysulfides wherein the alkyl groups have from 1 to 8 carbon atoms, and sulfurized polyolefins wherein the olefins have from 2 to 8 carbon atoms per molecule, and from about 20 to about 50 percent by weight of a friction modifying agent selected from the group consisting of sperm oil, a C_{12}-C_{24} fatty acid and a C_{12-24} alkenyl sarcosine, and
   iii. from about 5 to about 70 percent by weight of (3) an organic phosphite or an organic acid phosphate containing at least one alkyl or alkenyl group having from about 12 to about 24 carbon atoms, or (4) from about 5 to about 70 parts by weight of a combination of an organic phosphite, the alkyl groups of which have from 1 to about 8 carbon atoms and said friction modifying agent.

Preferred additive formulations comprise from about 5 to about 10 parts by weight of (A) and from about 2 to about 5 parts by weight of (B).

COMPONENT (A)

Component (A) can be considered to constitute an engine oil additive composition.

In component (A), the basic detergent is preferably present to the extent of from about 60 to about 90 percent by weight. The antioxidant is present preferably in an amount of from about 5 to about 25 percent by weight.

It is also contemplated that a portion of the detergent can be replaced by a dispersant of the character defined below. Thus, up to about 60 and preferably up to about 40 percent by weight of said detergent can be replaced by one or more of the dispersants.

As a further modification, a portion of the antioxidant can be replaced by an antiwear agent of the character defined below.

BASIC DETERGENT

The basic detergents are basic alkaline earth metal sulfonates and phenates which include those in which excess metal has been combined with the normal metal salts thereof. These compounds are referred to as "overbased" metal sulfonates and phenates. The higher the degree of overbasing the less is necessary to form the oil soluble complex. The metal used in these overbased salts are taken from Group II of the Periodic Table: barium, calcium, and magnesium are especially effective. Those overbased sulfonate salts described in U.S. Pat. Nos. 3,133,019 and 3,158,572 are representative of suitable sulfonate reactants. They include the petroleum and aromatic sulfonates. The alkaline earth metal phenates described in U.S. Pat. No. 2,916,454 are examples of suitable phenate reactants.

Still other typical basic detergents are described in U.S. Pat. Nos.: 2,739,124; 3,036,971; 3,046,224; 3,133,019; 3,213,019; 3,259,576; and 3,350,310.

The amount of metal in the overbased salts may range from about 1 to about 20 percent of the total compound. These basic compounds are also rated for alkalinity by a total base number (TBN) in terms of milligrams of potassium hydroxide per gram of sample, using the ASTM D-664 method. Suitable basic salts having TBN's ranging from about 150 to 400 may be used.

It is also contemplated that a portion of the basic detergent can be replaced by a neutral or low base number (TBN of 10–100), metal-containing detergent such as a calcium, barium or magnesium sulphonate, phosphonate or phenate, or of a reaction complex containing such a compound, such as a phosphosulfurized polybutene barium phenate sulphonate. Thus, the neutral or low TBN detergent can be included with one or more basic detergents, with the proviso that the TBN of the detergents is at least about 75.

ANTIOXIDANTS

Antioxidants used herein include phenols and metal alkyl dithiophosphates, the metals of which are either zinc, cadmium or nickel, and the alkyl groups of which contain from about 4 to about 12 carbon atoms.

Typical phenols include:

4-tertiary butyl catechol,

2,4-ditertiary butyl p-cresol,

2,6-ditertiary butyl-4-methyl phenol, and

2,2'-ethylene bis-2-6-ditertiary butyl p-cresol.

The metal alkyl dithiophosphates are illustrated by zinc dihexyl dithiophosphate, zinc dioctyl dithiophosphate, cadmium dibutyl dithiophosphate and nickel diamyl dithiophosphate.

DISPERSANT

The dispersant is substantially ash-free and preferably comprises an alkenyl succinic anhydride polyalkylene polyamine. Such dispersants are formed by reacting alkenyl succinic acid anhydrides having from about 8 to about 18 carbon atoms in the alkenyl group thereof, with a polyamine having the formula $H_2N(R\text{-}NH)_nH$, wherein R is ethylene or propylene and n is an integer of from 1 to 6.

Methods for preparing the alkenyl succinic acid anhydrides are well known, the most feasible method comprising the reaction of an olefin with maleic acid anhydride (U.S. Pat. No. 2,638,450).

Examples of the alkenyl succinic acid anhydrides include: butenyl succinic acid anhydride; and polybutene (molecular weight, 900) succinic acid anhydride. Representative polyamines include: ethylenediamine, propylenediamine, diethylenetriamine, triethylenetetramine, tetraethylenepentamine, dipropylenetriamine and amino substituted alkylamines, the latter having the formula $RNHCH_2CH_2Ch_2NH_2$ wherein R represents a mixture of alkyl groups derived from a fatty acid.

Other suitable dispersants include glycol esters of said alkenyl succinic acid anhydrides, obtained by esterifying the anhydrides with glycols. Typical glycols for such reaction include ethylene glycol, propylene glycol and trimethylene glycol.

Still other suitable dispersants include hydroxylamine esters of said alkenyl succinic acid anhydrides. These are obtained by esterifying the anhydrides with hydroxyl alkyl amines typified by ethanolamines and propanolamines.

A preferred dispersant is a polybutene (molecular weight, 900) succinic anhydride tetraethylene pentamine.

ANTI-WEAR AGENTS

There can also be present in component (A) from about 0.1 to about 2 percent by weight of an anti-wear agent comprising a metal alkyl dithiophosphate, wherein the metal is zinc, cadmium or nickel and the alkyl groups have from about 4 to about 12 carbon atoms. A preferred agent is zinc dihexyl dithiophosphate.

COMPONENT (B)

Component (B) can be considered to have friction modifying and extreme pressure characteristics.

In component (B), the sulfurized fat (a) (1) is preferably present in an amount of from about 60 to about 90 percent by weight. The sulfur-containing compound or polymer based thereon (a) (2) is preferably present in an amount of from about 50 to about 80 percent by weight of (B), with the balance a friction modifier.

The phosphites or phosphates (b) (3) and (b) (4) are preferably present in an amount of from about **10** to about **30** percent by weight of (B). Friction modifying agents can be present in amounts of from about 20 to about 50 percent by weight of a combination of (b) (4) of (B). When either or both of the respective compounds providing sulfur (a) and phosphorus (b) is a short chain compound (C_1-C_8 alkyl), it is preferred to use 5–50 percent by weight of the friction modifier.

In component (B), the weight ratio (a)/(b) can range from about 10:1 to about 1:1, more preferably 5:1 to 2:1. The weight ratio of sulfur to phosphorus is preferably from 5:1 to 20:1. Fatty acid compounds can be replaced by their corresponding salts.

SULFURIZED FATS

Conventional sulfur-containing compositions can be employed in the additive compositions. These are compositions in which sulfur is loosely or firmly bound. Typical of such compositions are sulfurized animal, marine or vegetable oils and terpenes containing up to about 20 percent by weight of sulfur. Sulfurized lard and sulfurized sperm oils are representative compositions. Procedures for preparing such compositions are provided in U.S. Pat. No. 2,993,858.

Other suitable sulfur-containing products include sulfurized cardanyl alkyl ethers (2,361,353) and sulfur-perchloromethylmercaptan products (2,329,324).

The sulfur-containing compounds, (B) (2), are alkyl sulfides, alkyl polysulfides or sulfurized polyolefins. Representative of such compounds are: diamyl sulfide; di tertiary butyl disulfide and sulfurized polybutenes containing up to about 50, and preferably about 40, percent by weight of sulfur.

Friction modifying agents include sperm oil, C_{12-24} fatty acids such as lauric, myristic, palmitic, stearic and oleic acids, and substituted fatty acids typified by oleyl sarcosine.

ORGANIC PHOSPHITES AND ORGANIC ACID PHOSPHATES

As indicated, the phosphites can be those containing at least one alkyl group having from about 12 to about 24 carbon atoms, illustrated by dioleyl phosphite and dilauryl phosphite. Other phosphites are those in which the alkyl groups have from 1 to about 8 carbon atoms, and are illustrated by dibutyl phosphite and amyl phenyl phosphite.

The organic acid phosphates are represented by: mono- and di-stearyl acid phosphates; mixed C_{14}-C_{18} alkyl acid phosphate; lauryl acid phosphates; and oleyl acid phosphates.

LUBRICANT COMPOSITIONS

The components of the additive thus selected are blended to obtain a combination which, when incorporated in a suitable base oil, provides a balance of properties. That is to say, the requirements of the various functions to be served by the oil are all met by the balancing of the contending factors; the actual numerical proportions of each ingredient to satisfy such a balance vary according to their specific nature and that of the oil and according to other factors such as the type of appliance and performance called for, but are readily determined in practice within the area of choice provided by the invention. It will be appreciated for instance that the extreme maximum or minimum specified amounts of the various ingredients are not necessarily applicable together in the same composition.

According to the present invention, therefore, a multipurpose lubricating oil composition comprises 3 to 23 parts by weight in 100 parts of the total oil composition, i.e. 3 to 23 percent by weight, of an additive composition comprising (A) and (B) as defined above, dispersed in an oil of lubricating viscosity of synthetic, mineral or mixed origin. Any oil can be used which is suitable as a lubricant base. Other additives can be incorporated for various known purposes such as viscosity index improvers, e.g. polymethacrylates.

It has been found surprisingly that oils of the present invention not only fulfill requirements for engine oil, transmission and hydraulic fluids but also those for wet brakes and power take-off clutches, so that the oil of the invention represents a major advance in providing a universal oil for a powered vehicle of the type referred to.

The lubricating vehicles employed can comprise any of the conventional oils of lubricating viscosity including mineral or synthetic lubricating oils, or mixtures thereof. Mineral lubricating oils can be of any suitable lubricating viscosity ranging from about 45 SSU at 100° F., to about 2,000 SSU at 100° F. and preferably from about 50 to about 250 SSU at 210° F. These oils may have viscosity indexes, varying from below 0 to about 100 or higher. Viscosity indexes from about 70 to about 95 are preferred.

The following Examples, given in Table 1 for the purpose of illustrating the invention, represent additive combinations according to the invention, with varying balances of requirement, for engine, rear axle and wet brake performances. The figures represent parts by weight.

Each of the foregoing Examples of additive combination is incorporated in a base mineral oil stock with suitable viscosity index improver, namely, to make up to 100 parts by weight, including optional amounts of for example 0.15 parts of a silicone defoamant such as dimethyl silicone (DCF 200 of Dow Corning).

The base mineral oil stock and viscosity index improver will, of course, be selected according to known requirements, within the limits of requirements set by the various functions to be served. A typical balanced oil contains by weight

65 percent 150-second neutral paraffinic stock

22.5 percent 500-second neutral paraffinic stock

10 percent 100-second solvent naphthenic stock together with as viscosity index improver

2.5 percent high molecular weight polymethacrylate (Acryloid 940 of Rohm & Haas).

This oil has a final viscosity in the SAE 20/30 region, and a low temperature viscosity of 4,000 c.p.s. at 0° F. If in order to increase low temperature fluidity a too volatile naphthenic component is used, engine oil consumption problems are encountered.

The foregoing additive combinations of Examples 1 to 8, made up with the aforesaid base oil and viscosity index improver, were tested and compared with two prior art multifunctional oils, I and II, with the results tabulated below in Table 2. Oil I was a conventional "universal" tractor oil containing a detergent/dispersant combination with an anti-oxidant, employing an overbased calcium sulphonate and a low base number barium phosphonate/sulphonate, with zinc dithiophosphate. Oil II was a multi-purpose oil containing a proprietary additive combination which is recommended for use in wet brake oils, and includes a low base number barium detergent, a friction modifier and zinc dialkyl dithiophosphate.

The test methods used to obtain the results in Table 2 are as follows:

Viscosity at 210° F: IP * 71.

Viscosity at 0° F: Brookfield viscosity determination according to CRC Designation L-45-1262.

IAE Gear Test IP 166.

Petter AV-1 IP 175 (DEF ** 2101D, modified)

Petter W-1 IP 176 (DEF 2101D, modified)

(* IP: Institute of Petroleum

** DEF: British Ministry of Defense Specification)

WET BRAKE TRACTOR CHATTER TEST

Chatter is a loud rattling, clanking sound which is entirely different from any noise usually heard on a tractor, and which occurs most noticeably in field service when the brake on one side is applied while the tractor is moving slowly. This test procedure simulates such service, and most resembles turning the tractor around with one wheel stationary when reversing to run back down an adjacent crop row.

A John Deere 4010 tractor, with right brake and planetary housing especially compartmented to permit testing of 1-gallon quantities of lubricant, is operated on a stationary pad with the left axle locked. Each test consists of a series of stepwise brake applications covering a range of speeds and brake pres-

TABLE I

	Example							
	1	2	3	4	5	6	7	8
Additive Material:								
Basic calcium sulphonate total base No. 300	2.25	2.35	3.0	3.0	2.6	2.6	2.6	2.25
P_2S_5-polybutene barium phenate/sulphonate	1.25	----	----	----	1.3	1.3	1.3	1.0
Ash-free dispersant [1]	1.25	2.3	3.0	3.0	1.9	1.9	1.9	1.25
Zinc dihexyl dithiophosphate	1.75	1.75	1.0	1.0	0.8	0.8	0.8	0.6
Sulphurised sperm oil (13 wt. percent S)	3.0	3.0	3.0	5.0	3.0	----	----	3.0
Dioleyl phosphite	1.0	1.0	1.0	1.0	1.0	1.0	1.0	----
Sperm oil	----	----	----	----	0.50	0.50	0.50	----
Sulphurised polybutene (40 wt. percent S)	----	----	----	----	----	1.80	1.80	----
Oleyl sarcosine	----	----	----	----	----	----	0.50	----
C_{14}-C_{18} alkyl acid phosphate	----	----	----	----	----	----	----	----

[1] Polybutenyl succinic anhydride tetraethylene pentamine reaction product in which the polybutenyl group has a molecular weight of about 900.

sures. Runs are started with the brake friction surface approximately 10° F. above ambient temperature. Observations of brake chatter are obtained from recordings of locked axle torque, taken with strain gauge instrumentation, on a CED oscillograph. Axle revolutions per minute, and friction pad surface temperature, are also recorded. A new brake disc and new friction pads are fitted before each test.

Wet brake chatter is assessed by comparing friction and torque gauge readings obtained on the test oil with those obtained on a reference oil. The good antichatter performance of the reference oil has been confirmed by field experience.

TABLE 2

Example	I	2	3	4	5	6	7	8	I	II
Viscosity, cs. 210° F.	8.6	9.1	9.5	9.9	9.7	8.7	8.7	8.6	11.1	9.0
Viscosity, cp.s., 0° F.	3750	3375	4140	.....	3900	3400	3410	3500	.....	4000 max.
DEF 2101D engine performance:										
Petter AVI average rating (10 max., 7 pass)...	7	7	7	.....	7	9	9	7	7	.4
Petter W1	Pass	Pass	Pass	.....	Pass	.....			Pass	.....
Load carrying 4 ball seizure, kg.	280	280	270	.....	370	340	230	190		210
4 ball wear, scar diameter mm. at 40 k.gm., 200 °F. 600 r.p.m. for 2 hrs.	0.33	0.46	0.45	0.41	.....	0.45	.....		0.44	0.35
IAE gear test, lbs. 2000 r.p.m. 110° C.	130	115	110	.....					105	90
Wet brake chatter	Good	Good	Good	Good	Excellent	Good	Excellent	Fair	Unacceptable	Good

The data in the foregoing Table 2 show the essential advantages of the composition of the invention, namely the ability to lubricate engines (which multipurpose wet brake oils cannot) and wet brake anti-chatter and load-carrying performance superior to those of a conventional "universal" tractor oil.

The invention thus provides for the first time an oil which can be used effectively in all lubricating functions of vehicles of the type referred to.

What is claimed is:

1. An additive composition for a multipurpose lubricating oil, comprising

A. from about 2 to about 15 parts by weight of a composition comprising

i. from about 50 to about 95 percent by weight of a mineral oil soluble or dispersible, overbased metal salt selected from the group consisting of alkaline earth metal sulfonates and phenates and having a Total Base Number of at least 75, and

ii. From about 5 to about 50 percent by weight of a mineral oil soluble antioxidant selected from the group consisting of tertiary ditertiary alkylated phenols and a metal alkyl dithiophosphate, the metal of which is selected from the group consisting of zinc, cadmium and nickel, and the alkyl groups of which each have from about 4 to about 12 carbon atoms, and

B. from about 1 to about 8 parts by weight of a composition comprising

i. from about 30 to about 95 percent by weight of

1. a sulfurized C_{12-24} fat, or

2. from about 30 to about 95 percent by weight of a combination of from about 50 to about 80 percent by weight of a sulfur-containing compound selected from the group consisting of an alkyl sulfide and an alkyl polysulfide the alkyl groups of which each have from 1 to about 8 carbon atoms, and sulfurized polyolefins wherein the olefins have from 2 to about 8 carbon atoms per molecule, and from about 20 to about 50 percent by weight of a friction modifying agent selected from the group consisting of sperm oil, a C_{12-24} fatty acid and a C_{12-24} alkenyl sarcosine, and

ii. from about 5 to about 70 percent by weight of

3. an organic phosphite containing at least two alkyl or alkenyl group having from about 12 to about 24 carbon atoms or an organic acid phosphate each alkyl or alkenyl group of which has from about 12 to about 24 carbon atoms, or

4. a combination of 50 to about 80 percent by weight of an organic di-alkyl phosphite, the alkyl groups of which have from 1 to about 8 carbon atoms, and from about 20 to about 50 percent by weight of said friction modifying agent.

2. An additive formulation of claim 1 containing from 5 to 10 parts by weight of (A) and from 2 to 5 parts by weight of (B).

3. An additive formulation according to claim 1, wherein the detergent (A) (i) is a calcium sulfonate having a Total Base Number of about 300.

4. An additive formulation according to claim 1, wherein the antioxidant (A) (ii) is zinc dihexyl dithiophosphate.

5. An additive formulation according to claim 1 containing a dispersing amount of an alkenyl succinic anhydride polyalkylene polyamine.

6. An additive formulation according to claim 5 wherein the alkenyl anhydride polyalkylene polyamine is a polybutene succinic anhydride tetraethylene pentamine.

7. An additive formulation according to claim 1, wherein the sulfurized fat (B) (a) (1) is sulfurized sperm oil containing about 13 percent by weight of sulfur.

8. An additive formulation according to claim 1, wherein the sulfurized polyolefin (B) (a) (2) is a sulfurized polybutene containing about 40 percent by weight of sulfur.

9. An additive formulation according to claim 1, wherein the C_{12-24} alkenyl sarcosine is oleyl sarcosine.

10. An additive formulation according to claim 1 wherein the organic phosphite (B) (b) (3) is dioleyl phosphite.

11. An additive formulation according to claim 1, wherein the organic acid phosphate (B) (b) (3) is a C_{14-18} mixed alkyl acid phosphate.

12. A multipurpose lubricating oil composition comprising an oil of lubricating viscosity and from about 3 to about 23 percent by weight, based upon the total oil composition of an additive formulation of claim 1.

* * * * *

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,652,410 Dated March 28, 1972

Inventor(s) RALPH HOLLINGHURST and ANDREW G. PAPAY

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

In the Title after "CONTAINING" read --SAME--.

Column 1, line 3 after "CONTAINING" read --SAME--.

Under Columns 5 and 6, in Table I, under 3.0 in Ex. 8 insert --0.5--.

Under Columns 7 and 8, in Table II, under Ex. II, line 5 for ".4" read --4--.

Column 7, line 49 after "tertiary" insert --and--.

Signed and sealed this 8th day of August 1972.

(SEAL)
Attest:

EDWARD M.FLETCHER,JR.
Attesting Officer

ROBERT GOTTSCHALK
Commissioner of Patents