United States Patent Office 3,046,102 Patented July 24, 1962

3,046,102

STABILIZED DISTILLATE FUEL OIL

Harry J. Andress, Jr., Pitman, and Paul Y. C. Gee, Woodbury, N.J., assignors to Socony Mobil Oil Company, Inc., a corporation of New York

No Drawing. Filed Oct. 6, 1958, Ser. No. 765,296
6 Claims. (Cl. 44—71)

This invention relates to the improvement of non-lubricating petroleum fractions. It is more particularly concerned with distillate fuel oils containing additives adapted to inhibit the appearance of sediment during prolonged storage periods, to prevent screen-clogging, and to prevent rusting of ferrous metal surfaces.

It is well known that fuel oils are prone to form sludge or sediment during periods of prolonged storage. This sediment, of course, has an adverse effect on burner operation, because it has a tendency to clog screens and nozzles. In addition to sediment formed during storage, most fuel oils contain other impurities, such as rust, dirt, and entrained water. The sediment and impurities tend to settle out on equipment parts, such as nozzles, screens, filters, etc., thereby clogging them and causing the equipment to fail.

A further factor, incident to the storage and handling of fuel oils, is the "breathing" of storage vessels. This results in the accumulation of considerable amounts of water in the tanks, which presents a problem of rusting in the tanks. Then, when the oil is removed for transportation, sufficient water may be carried along to cause rusting of ferrous metal surfaces in pipelines, tankers, and the like.

Generally, it has been the practice to overcome the aforedescribed difficulties with a separate additive for each purpose, i.e., with a sediment inhibitor, an anti-screen clogging agent, and an antirust agent. The use of several additives, however, give rise to problems of additive compatibility, thus restricting the choice of additive combinations. In addition, of course, the use of a plurality of additives unduly increases the cost of the fuel. It has been proposed to overcome two difficulties, e.g., sedimentation and screen clogging, with one additive. Insofar as is known, however, few single addition agents have been found effective against sedimentation, screen and nozzle clogging, and rusting of ferrous metal surfaces.

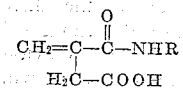
It has now been found that all three problems, i.e., sedimentation, screen clogging, and rusting, can be solved by the use of a single fuel oil addition agent. It has been discovered that a distillate fuel oil containing minor amounts of certain amic acids and amine salts thereof is effectively inhibited, simultaneously, against all three aforementioned difficulties.

Accordingly, it is a broad object of this invention to provide a fuel oil having properties improved with a minimum number of addition agents. Another object is to provide a fuel oil having a single additive adapted to inhibit sedimentation, to prevent screen clogging, and to prevent rusting of ferrous metal surfaces with which it comes in contact. A specific object is to provide certain novel amic acids or amine salts thereof and fuel oils containing them. Other objects and advantages of this invention will become apparent to those skilled in the art from the following detailed description.

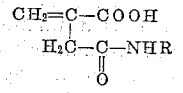
The present invention provides amic acids of itaconic acid and aliphatic primary and secondary amines having between about 4 carbon atoms and about 30 carbon atoms per molecule and the amine salts thereof with an aliphatic primary amine having between about 4 carbon atoms and about 30 carbon atoms per molecule; and distillate fuel oil containing a minor amount of them, sufficient to inhibit sedimentation and screen clogging and to prevent rusting of ferrous metal surfaces in contact therewith.

The addition agents contemplated herein are the amic acids of itaconic acid with aliphatic primary and secondary amines having between about 4 and about 30 carbon atoms per molecule; and their amine salts with aliphatic primary amines having between about 4 and about 30 carbon atoms per molecule. The amic acids are produced by reacting itaconic acid with the amine reactant in a molar ratio of 1:1, at a temperature varying between about 100° C. and about 175° C. from 2 to 5 hours. The reaction is accompanied by the elimination of water to form amide groups. Thus, it is preferable to carry out the condensation reaction using an azeotrope to remove the water, such as benzene, toluene, xylene, etc. The salt of the amic acid can be made readily by warming equimolar quantities of the amic acid and an aliphatic primary amine having between about 4 and about 30 carbon atoms per molecule. The salt-forming amine can be the same amine used in making the amic acid, or it can be a different amine. In the case where the salt-forming amine is the same used in the amic acid, the salt can be made by heating two moles of amine with one mole of acid under temperatures whereby one mole of water is evolved.

The itaconic amic acids can have the structural formulae:

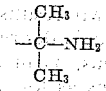


or



wherein R is an aliphatic hydrocarbon radical having between about 4 and about 30 carbon atoms. Indeed, in the aforedescribed condensation reaction, mixtures of both formulae can be produced. For this reason, and because the amines are often mixtures of amines, the amic acids of this invention are more accurately defined by their method of manufacture.

The amines utilizable in forming the amic acids and the salts thereof are the primary and secondary aliphatic amines having between about 4 and about 30 carbon atoms per molecule. These are the monoamines having a single open chain hydrocarbon group attached to a nitrogen atom. The aliphatic radical can be saturated or unsaturated, and branched-chain or normal chain. Likewise mixtures of these amines, as well as pure amines, can be employed. A very useful and readily available class of primary amines are the tertiary-alkyl, primary, monoamines in which a primary amino ($-NH_2$) group is attached to a tertiary carbon atom; and mixtures thereof. These amines all contain the terminal group,



Non-limiting examples of the amine reactants are t-butyl amine, n-butyl amine, dibutyl amine, t-hexyl primary amine, n-hexylamine, n-octylamine, n-octenylamine, t-octyl primary amine, dioctylamine, 2-ethylhexylamine, t-decyl primary amine, n-decylamine, t-dodecyl primary amine, n-undecylamine, dodecenylamine, dodecadienylamine, tetraydecylamine, t-tetradecyl primary amine, t-octadecyl primary amine, dioctadecylamine, hexadecylamine, octadecenylamine, octadecadienyl amine, t-eicosyl primary amine, t-docosyl primary amine, t-tetracosyl primary amine, and t-triacontyl primary amine. The amine reactants can be prepared in several ways well known to those skilled in the art. Specific methods of preparing

the t-alkyl primary amines are disclosed in the Journal of Organic Chemistry, vol. 20, page 295 et. seq. (1955). Mixtures of such amines can be made from a polyolefin fraction (e.g., polypropylene and polybutylene cuts) by first hydrating with sulfuric acid and water to the corresponding alcohol, converting the alcohol to alkyl chloride with dry hydrogen chloride, and finally condensing the chloride with ammonia, under pressure, to produce a t-alkyl primary amine mixture.

The fuel oils that are improved in accordance with this invention are hydrocarbon fractions having an initial boiling point of at least about 100° F. and an end-boiling point no higher than about 750° F., and boiling substantially continuously throughout their distillation range. Such fuel oils are generally known as distillate fuel oils. It is to be understood, however, that this term is not restricted to straight-run distillate fractions. The distillate fuel oils can be straight-run distillate fuel oils, catalytically or thermally cracked (including hydrocracked) distillate fuel oils, or mixtures of straight-run distillate fuel oils, naphthas and the like, with cracked distillate stocks. Moreover, such fuel oils can be treated in accordance with well known commercial methods, such as, acid or caustic treatment, hydrogenation, solvent refining, clay treatment, etc.

The distillate fuel oils are characterized by their relatively low viscosities, pour points, and the like. The principal property which characterizes the contemplated hydrocarbons, however, is the distillation range. As mentioned hereinbefore, this range will lie between about 100° F. and about 750° F. Obviously, the distillation range of each individual fuel oil will cover a narrower boiling range falling, nevertheless, within the above-specified limits. Likewise, each fuel oil will boil substantially continuously throughout its distillation range.

Particularly contemplated among the fuel oils are Nos. 1, 2, and 3 fuel oils used in heating and as diesel fuel oils, and the jet combustion fuels. The domestic fuel oils generally conform to the specifications set forth in A.S.T.M. Specifications D396–48T. Specifications for diesel fuels are defined in A.S.T.M. Specifications D975–48T. Typical jet fuels are defined in Military Specification MIL–F–5624B.

The amount of itaconic amic acid or amine salt of amic acid additives that is added to the distillate fuel oil in accordance with this invention will depend, of course, upon the intended purpose and the particular amic acid or salt selected, as they are not all equivalent in their activity. Some may have to be used in greater concentrations than others to be effective. In most cases, in which it is desired to obtain all three beneficial results, namely, to inhibit sedimentation, to reduce screen clogging, and to prevent rusting of ferrous metal surfaces, additive concentrations varying between 10 pounds per thousand barrels of oil and about 200 pounds per thousand barrels of oil will be employed. It may not always be desired, however, to accomplish all three aforementioned results. In such cases, where it is desired to effect only one or two results, lower concentrations can be used. Thus, if it is desired only to prevent rust under dynamic conditions, as in a pipeline, it has been found that concentrations as low as about 5 p.p.m., i.e., about one pound of additive per thousand barrels of oil, are effective. In general, therefore, the amount of amic acid or of amine salt of amic acid that can be added to the distillate fuel oil, in order to achieve a beneficial result, will vary generally between about one pound per thousand barrels of oil and about 200 pounds per thousand barrels of oil. Preferably, it will vary between about 10 pounds and about 200 pounds per thousand barrels of oil.

If it is desired, the fuel oil compositions can contain other additives for the purpose of achieving other results. Thus, for example, there can be present foam inhibitors and ignition and burning quality improvers. Examples of such additives are silicones, dinitropropane, amyl nitrate, metal sulfonates, and the like.

The following specific examples are for the purpose of illustrating the amic acids and salts and the fuel oil compositions of this invention, and of exemplifying the specific nature thereof. It is to be strictly understood, however, that this invention is not to be limited by the particular additives and fuel oils, or to the operations and manipulations described therein. Other amic acids or amine salts thereof and fuel oils, as discussed hereinbefore, can be used, as those skilled in the are will readily appreciate.

AMIC ACIDS AND SALTS

The amine reactants used in the specific working examples are mixtures of pure amines. "Amine A" is a mixture of primary amines having a carbon atom of a tertiary butyl group attached to the amino (—NH_2) group and containing 12 to 15 carbon atoms per amine molecule and averaging 12 carbon atoms per amine molecule. This mixture contains, by weight, about 85 percent tertiary dodecyl amine, about 10 percent tertiary pentadecyl amine, and relatively small amounts, i.e., less than about 5 percent of amines having less than 12 or more than 15 carbon atoms.

"Amine B" is a mixture of tertiary-alkyl primary amines averaging 18 to 24 carbon atoms per molecule. It has a tertiary carbon atom attached to the —NH_2 group. It contains, by weight, about 40 percent t-octadecyl primary amine, about 30 percent t-eicosyl primary amine, about 15 percent t-docosyl primary amine, about 10 percent t-tetracosyl primary amine, and a small amount, less than 5 percent, other amines as high as t-triacontyl primary amine.

"Amine C," "Amine D," "Amine E," "Amine F," and "Amine G" are mixtures of normal aliphatic primary amines having the weight percent compositions set forth in Table I.

Table I

Normal Amine	Amine C	Amine D	Amine E	Amine F	Amine G
Hexyl			3		
Octyl			90		
Decyl			7	2	
Dodecyl				95	
Tetradecyl		2		3	
Hexadecyl	10	24			6
Octadecyl	10	28			90
Octadecenyl	35	46			4
Octadecadienyl	45				

"Amine H" is a mixture of dialkylamines in which the N-alkyl radicals are 8 percent octyl, 9 percent decyl, 47 percent dodecyl, 18 percent tetradecyl, 8 percent hexadecyl, and 10 percent octadecyl.

In the run set forth in the following examples, the reactions were carried out using azeotropic distillation to remove water formed in the condensation. The reaction vessel, in each case, was provided with a reflux condenser equipped with a water take-off trap.

Example 1.—A mixture of 65 grams (0.5 mole) of itaconic acid, 100 grams (0.5 mole) of Amine A, 100 grams of kerosine having a boiling point range from 343 to 519° F., and 50 cc. of benzene was refluxed at 125–130° C. until the evolution of water ceased. The amount of water collected during the reaction was 10 cc. (theory 9 cc.). All the benzene was distilled off. The final product, the Amine A itaconic amic acid, which contained 40% kerosine was clear and fluid at room temperature.

Example 2.—A mixture of 130 grams (1 mole) of itaconic acid, 300 grams (1 mole) of Amine B, 200 grams of diluent oil (paraffinic oil of 100 S.U.S. at 100° F.) and 100 cc. of benzene was refluxed at 125° C. until water evolution stopped. The amount of water collected during the reaction was 18 cc. (theory 18 cc.). All the benzene was distilled off. The final product, the Amine

B itaconic amic acid, containing 33% diluent oil, was clear and fluid at room temperature.

Example 3.—A mixture of 54.2 grams (0.4165 mole) of itaconic acid, 50 grams (0.1665 mole) of Amine C, 50 grams (0.25 mole) of Amine A, 100 grams of kerosine (boiling range 343–519° F.) and 70 cc. of benzene was refluxed at 117–130° C. until water stopped coming over. The amount of water collected during the reaction was 8 cc. (theory 7.5 cc.). All the benzene was distilled off. The final product, the mixed Amine C-Amine A itaconic amic acid, containing 41% kerosine, was clear and fluid at room temperature.

Example 4.—A mixture of 54.2 grams (0.4165 mole) of itaconic acid, 50 grams (0.1665 mole) of Amine D, 50 grams (0.25 mole) of Amine A, 100 grams of xylene and 75 cc. of benzene was refluxed at 120° C. until water stopped coming over. The amount of water collected during the reaction was 8.5 cc. (theory 7.5 cc.). All the benzene was distilled over. The final product, the mixed Amine D-Amine A itaconic amic acid, containing 41% xylene was clear and fluid at room temperature.

Example 5.—A mixture of 32.5 grams (0.25 mole) of itaconic acid, 75 grams (0.25 mole) of Amine C, 50 grams of xylene and 50 cc. of benzene was refluxed at 125° C. until water stopped coming over. The amount of water collected during the reaction was 4.5 cc. (theory 4.5 cc.). All the benzene was distilled off. The final product, the Amine C itaconic amic acid, which contained 33% xylene was clear and fluid at room temperature.

Example 6.—A mixture of 32.5 grams (0.25 mole) of itaconic acid, 32.25 grams (0.25 mole) of Amine E, 50 grams xylene and 60 cc. benzene was refluxed at 100–105° C. until the evolution of water ceased. The amount of water collected during the reaction was 4.5 cc. (theory 4.5 cc.). All the benzene was distilled off. The final product, the Amine E itaconic amic acid, which contained 46% xylene was clear and fluid at room temperature.

Example 7.—A mixture of 65 grams (0.5 mole) of itaconic acid, 97.5 grams (0.5 mole) of Amine F, 100 grams of xylene and 75 cc. of benzene was refluxed at 110° C. until water stopped coming over. The amount of water collected during the reaction was 10 cc. (theory 9 cc.). All the benzene was distilled off. The final product, the Amine F itaconic amic acid, which contained 40% xylene was clear and fluid at room temperature.

Example 8.—A mixture of 65 grams (0.5 mole) of itaconic acid, 140 grams (0.5 mole) of Amine G, 140 grams of xylene and 75 cc. of benzene was refluxed at 115° C. until water stopped coming over. The amount of water collected during the reaction was 10 cc. (theory 9 cc.). All the benzene was distilled off. The final product, the Amine G itaconic amic acid, which contained 41% xylene was clear and fluid at 50° C. and solidifying at room temperature.

Example 9.—A mixture of 43.5 grams (⅓ mole) of itaconic acid, 150 grams (⅓ mole) of Amine H, 100 grams of xylene and 75 cc. of benzene was refluxed at 125° C. until water stopped coming over. The amount of water collected during the reaction was 6 cc. (theory 6 cc.). All the benzene was distilled off. The final product, the Amine H itaconic amic acid, which contained 35% xylene was clear and fluid at room temperature.

Example 10.——A mixture of 32.5 grams (0.25 mole) of itaconic acid, 50 grams (0.25 mole) of Amine A, 75 grams of xylene and 50 cc. of benzene was refluxed at 125° C. to form an Amine A itaconic amic acid. The temperature was held at 125° C. until water stopped coming over. The amount of water collected during the reaction was 4.5 cc. (theory 4.5 cc.). To the Amine A itaconic amic acid was added at room temperature with stirring 75 grams (0.25 mole) of Amine C to form an amine salt. The mixture was stirred at 80–85° C. for 3 hours. The product, the Amine C salt of Amine A itaconic amic acid, was distilled at 95° C., under a pressure of 5 mm. of mercury, until no more benzene and xylene came over.

Example 11.—A mixture of 32.5 grams (0.25 mole) of itaconic acid, 75 grams (0.25 mole) of Amine C, 75 grams kerosine (boiling range 343–519° F.) and 75 cc. of benzene was refluxed at 135° C. to form an Amine C itaconic amic acid. The temperature was held at 135° C. until water stopped coming over. The amount of water collected during the reaction was 6 cc. (theory 4.5 cc.). All the benzene was distilled off. To the Amine C itaconic amic acid was added at room temperature with stirring 50 grams (0.25 mole) of Amine A to form an amine salt. The mixture was stirred at 85–90° C. for 3 hours. The final product, the Amine A salt of Amine C itaconic amic acid, which contained 33% kerosine was clear and fluid at room temperature.

Example 12.—A mixture of 65 grams (0.5 mole) of itaconic acid, 149 grams (0.5 mole) of Amine D, 100 grams of xylene and 75 cc. of benzene was refluxed at 125° C. to form an Amine D itaconic amic acid. The temperature was held at 135° C. until water stopped coming over. The amount of water collected during the reaction was 9 cc. (theory 9 cc.). All the benzene was distilled off. To the Amine D itaconic amic acid was added at room temperature with stirring 100 grams (0.5 mole) of Amine A to form an amine salt. The mixture was stirred at 75° C. for 2 hours. The final product, the Amine A salt of Amine D itaconic amic acid, which contained 25% xylene was clear and fluid at room temperature.

Example 13.—A mixture of 65 grams (0.5 mole) of itaconic acid, 68 grams (0.5 mole) of Amine E, 100 grams of xylene and 75 cc. of benzene was refluxed at 115° C. to form an Amine E itaconic amic acid. The temperature was held at 115° C. until water stopped coming over. The amount of water collected during the reaction was 9 cc. (theory 9 cc.). All the benzene was distilled off. To the Amine E itaconic amic acid was added at room temperature with stirring 100 grams (0.5 mole) of Amine A to form an amine salt. The mixture was stirred at 75° C. for 2 hours. The final product, the Amine A salt of Amine E itaconic amic acid, which contained 31% xylene was clear and fluid at room temperature.

Example 14.—A mixture of 65 grams (0.5 mole) of itaconic acid, 97.5 grams (0.5 mole) of Amine F, 100 grams of xylene and 50 cc. of benzene was refluxed at 115° C. to form an Amine F itaconic amic acid. The temperature was held at 115° C. until water stopped coming over. The amount of water collected during the reaction was 9 cc. (theory 9 cc.). All the benzene was distilled off. To the Amine F itaconic amic acid was added at room temperature with stirring 100 grams (0.5 mole) of Amine A to form an amine salt. The mixture was stirred at 75° C. for 2 hours. The final product, the Amine A salt of Amine G itaconic amic acid, which contained 29% xylene was clear and fluid at room temperature.

Example 15.—A mixture of 65 grams (0.5 mole) of itaconic acid, 140 grams (0.5 mole) of Amine G, 100 grams of xylene and 50 cc. of benzene was refluxed at 125° C. to form an Amine G itaconic amic acid. The temperature was held at 125° C. until water stopped coming over. The amount of water collected during the reaction was 9 cc. (theory 9 cc.). All the benzene was distilled off. To the Amine G itaconic amic acid was added at room temperature with stirring 100 grams (0.5 mole) of Amine A to form an amine salt. The mixture was stirred at 75° C. for 2 hours. The final product, the Amine A salt of Amine G itaconic amic acid, which contained 26% xylene was clear and fluid at room temperature.

Example 16.—A mixture of 43.3 grams (⅓ mole) of itaconic acid, 66.7 grams (⅓ mole) of Amine A, 204 grams of diluent oil (paraffinic oil of 100 S.U.S. at 100°

F.) and 75 cc. of benzene was refluxed at 125° C. to form an Amine A itaconic amic acid. The temperature was held at 125° C. until water stopped coming over. The amount of water collected during the reaction was 6 cc. (theory 6 cc.). All the benzene was distilled off. To the Amine A itaconic amic acid was added at room temperature with stirring 100 grams (⅓ mole) of Amine B to form an amine salt. The mixture was stirred at 85–95° C. for 3 hours. The final product, the Amine B salt of Amine A itaconic amic acid, which contained 50% diluent oil was clear and fluid at room temperature.

SEDIMENTATION

The test used to determine the sedimentation characteristics of the fuel oils is the 110° F. Storage Test. In this test, a 500-milliliter sample of the fuel oil under test is placed in a convected oven maintained at 110° F. for a period of 12 weeks. Then, the sample is removed from the oven and cooled. The cooled sample is filtered through a tared asbestos filter (Gooch crucible) to remove insoluble matter. The weight of such matter in milligrams is reported as the amount of sediment. A sample of the blank, uninhibited oil is run along with a fuel oil blend under test. The effectiveness of a fuel oil containing an inhibitor is determined by comparing the weight of sediment formed in the inhibited oil with that formed in the uninhibited oil.

The additives described in the examples were blended in test fuel oil and the blends were subjected to the 110° F. Storage Test. The test results comparing the blended fuels and uninhibited fuels are set forth in Table II. The test fuel oil was a blend of 60 percent distillate stock obtained from continuous catalytic cracking and 40 percent straight-run distillate stock. It has a boiling range of between about 320° F. and about 640° F. and is a typical No. 2 fuel oil.

Table II

110° F. STORAGE TEST—12 WEEKS

Additive of Example	Conc'n, lbs./1,000 bbls.	Sediment, mg./liter
Blank		10
1	25	6
Blank		5
2	25	2
3	25	1
4		1
Blank		49
5	50	37
Blank		5
7	25	2
8	25	2
Blank		34
9	100	24
Blank		49
10	100	10
Blank		141
11	100	49
Blank		6
12	50	2
Blank		34
13	50	8
Blank		6
14	50	1
15	50	1
16	25	3

SCREEN CLOGGING

The anti-screen clogging characteristics of a fuel oil were determined as follows: The test is conducted using a Sundstrand V3 or S1 home fuel oil burner pump with a self-contained 100-mesh Monel metal screen. About 0.05 percent, by weight, of naturally-formed fuel oil sediment, composed of fuel oil, water, dirt, rust, and organic sludge is mixed with 10 liters of the fuel oil. This mixture is circulated by the pump through the screen for 6 hours. Then, the sludge deposit on the screen is washed off with normal pentane and filtered through a tarred Gooch crucible. After drying, the material in Gooch crucible is washed with a 50—50 (volume) acetone-methanol mixture. The total organic sediment is obtained by evaporating the pentane and the acetone-methanol filtrates. Drying and weighing the Gooch crucible yields the amount of inorganic sediment. The sum of the organic and inorganic deposits on the screen can be reported in milligrams recovered or converted into percent screen clogging.

Blends of the additives of the examples were prepared in the aforedescribed test fuel oil and subjected to the Screen Clogging Test. Results are set forth in Table III.

Table III

SCREEN CLOGGING

Additive of Example	Conc'n, lbs./1,000 bbls.	Screen Clogging percent
Blank		100
1	100	32
2	25	2
3	50	14
4	50	3
5	100	6
6	50	9
7	50	13
8	25	2
9	100	8
10	25	5
11	25	10
12	100	2
13	25	3
14	25	4
15	50	2
16		25

RUSTING

The method used for testing anti-rust properties of the fuel oils was the A.S.T.M. Rust Test D–665 operated for 48 hours at 80° F. using distilled water. This is a dynamic test that indicates the ability to prevent rusting of ferrous metal surfaces in pipelines, tubes, etc.

Blends of the additives described in the examples in the aforedescribed test fuel oil were subjected to the A.S.T.M. Rust Test D–665. Pertinent data are set forth in Table IV.

Table IV

A.S.T.M. RUST TEST D-665

Additive of Example	Conc'n, parts per million	Rust Test Result
Blank		Fail.
1	2.5	Pass.
2	50	Pass.
3	10	Pass.
4	10	Pass.
5	2.5	Pass.
6	50	Pass.
7	5	Pass.
8	5	Pass.
9	50	Pass.
10	50	Pass.
11	2.5	Pass.
12	10	Pass.
13	10	Pass.
14	5	Pass.
15	10	Pass.
16	25	Pass.

The Static Rust Test simulates conditions encountered in storage tanks, such as, the home fuel oil storage tank. In this test, a strip of 16–20 gauge sand blasted steel plate is placed in a clear quart bottle. The length of the strip is sufficient to reach from the bottom of the bottle into the neck of the bottle without interfering with the cap. One hundred cc. of synthetic sea water with pH adjusted to 5 (A.S.T.M. D–665) and 750 cc. of test oil are placed in the bottle. The bottle is capped tightly, shaken vigorously for one minute, and permitted to stand quietly at 80° F. for 21 days. At the end of that time, the amount of rust that occurs on the surface of the plate immersed in the water is used as a measure of effectiveness of the fuel to inhibiting rusting in storage vessels. It is generally preferred that no more than 5 percent of the surface should be rusted. This test is much more severe than the

A.S.T.M. Rust Test. Many additive compositions that pass the A.S.T.M. test fail in the Static Test. On the other hand materials that pass the Static Test always pass the A.S.T.M. test.

Blends of the additives of the examples in the aforedescribed test fuel oil were subjected to the Static Rust Test. Pertinent results are set forth in Table V.

Table V

STATIC RUST TEST

Additive of Example	Conc'n, lbs/1,000 bbls.	Percent Rusting
Blank		100
1	35	0
2	50	30
3	50	0
4	25	0
5	25	0
7	25	0
8	50	0
9	50	10
10	50	25
11	10	0
12	50	1
14	50	50
16	75	0

It will be apparent, from the data set forth in Tables II through V, that the amic acids of this invention and amine salts thereof are highly effective to reduce sedimentation and screen clogging and to inhibit rusting of ferrous metal surfaces. As is to be expected results will vary among specific materials used. In order to accomplish any given improvement, many of the additives can be used in relatively small amounts, as for dynamic rust prevention. If, on the other hand, it is desired to accomplish all the aforementioned beneficial results, this can be accomplished at the practical additive concentration of 50–100 pounds per thousand barrels of fuel oil.

Although the present invention has been described with with preferred embodiments, it is to be understood that modifications and variations may be resorted to, without departing from the spirit and scope of this invention, as those skilled in the art will readily understand. Such variations and modifications are considered to be within the purview and scope of the appended claims.

What is claimed is:

1. A distillate fuel oil containing between about 1 pound and about 200 pounds, per thousand barrels of fuel, of a material selected from the group consisting of (A) amic acids of itaconic acid and alphatic hydrocarbyl primary and secondary amines having between about 4 carbon atoms and about 30 carbon atoms per molecule and (B) salts of A with aliphatic hydrocarbyl primary amines having between about 4 carbon atoms and about 30 carbon atoms per molecule.

2. A distillate fuel oil containing between about 10 pounds and about 200 pounds, per thousand barrels of fuel, of an amic acid of itaconic acid and a mixture of primary tertiary-alkyl amines having a tertiary carbon atom attached to the amino group and containing 12 to 15 carbon atoms per molecule and averaging 12 carbon atoms per molecule.

3. A distillate fuel oil containing between about 10 pounds and about 200 pounds per thousand barrels of fuel, of an amic acid of itaconic acid and a mixture of primary amines containing, by weight, 6 percent hexadecyl amine, 90 percent octadecyl amine, and 4 percent octadecenyl amine.

4. A distillate fuel oil containing between about 10 pounds and about 200 pounds, per thousand barrels of fuel, of an amine salt of an amic acid of itaconic acid and a mixture of primary amines containing, by weight, 3 percent hexyl amine, 90 percent octyl amine, and 7 per cent decyl amine; with a mixture of primary tertiary-alkyl amines having a tertiary carbon atom attached to the amino group and containing 12 to 15 carbon atoms per molecule and averaging 12 carbon atoms per molecule.

5. A distillate fuel oil containing between about 10 pounds and about 200 pounds, per thousand barrels of fuel, of an amine salt of an amic acid of itaconic acid and a mixture of primary amines containing, by weight 2 percent decyl amine, 95 percent dodecyl amine, and 3 percent tetradecyl amine; with a mixture of primary tertiary-alkyl amines having a tertiary carbon atom attached to the amino group and containing 12 to 15 carbon atoms per molecule and averaging 12 carbon atoms per molecule.

6. A distillate fuel oil containing between about 10 pounds and about 200 pounds, per thousand barrels of fuel, of an amine salt of an amic acid of itaconic acid and a mixture of primary amines containing, by weight, 6 percent hexadecyl amine, 90 percent octadecyl amine, and 4 percent octadecenyl amine; with a mixture of primary tertiary-alkyl amines having a tertiary carbon atom attached to the amino group and containing 12 to 15 carbon atoms per molecule and averaging 12 carbon atoms per molecule.

References Cited in the file of this patent

UNITED STATES PATENTS

2,198,001	Dickey	Apr. 23, 1940
2,490,744	Trigg et al.	Dec. 6, 1949
2,604,451	Rocchini	July 22, 1952
2,699,427	Smith et al.	Jan. 11, 1955
2,718,503	Rocchini	Sept. 20, 1955
2,742,498	Smith et al.	Apr. 17, 1956
2,783,206	Messina	Feb. 26, 1957
2,827,483	Fishback et al.	Oct. 21, 1958
2,842,433	Newman et al.	July 8, 1958
2,851,344	Marsh et al.	Sept. 9, 1958
2,857,424	Cox	Oct. 21, 1958
2,908,711	Halter et al.	Oct. 13, 1959
2,944,969	Stromberg et al.	July 12, 1960