

UNITED STATES PATENT OFFICE

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METHOD OF INHIBITING FOAMING IN
STEAM BOILERSLewis O. Gunderson, Park Ridge, Ill., assignor to
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corporation of IllinoisNo Drawing. Application September 9, 1947,
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5 Claims. (Cl. 252—321)

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This invention relates to a method and a composition for conditioning water and, more particularly, for eliminating foaming conditions in steam boilers.

Foaming of boiler water is not, as commonly thought, equivalent to an accumulation of foam on top of the surface of the boiler water. When steam is rapidly withdrawn from a boiler with resultant foaming, there is no water surface within the boiler correlated with the water level indicated in the conventional water glass attached to the boiler. In other words, there is no sharp line of demarcation between solid water and foam in a boiler during rapid steam withdrawal.

The foaming of boiler water is actually a rapid expansion of the water in a steam generating area of the boiler brought about by the fact that rapidly forming small steam bubbles do not coalesce, if at all, until a definite short time after their formation. As a consequence, the entire volume of water in the generating area is expanded by myriads of small bubbles until the thus formed so-called "light water" may fill the steam space and become entrained with the steam leaving the boiler.

In other words, bubbles need not be particularly stable to cause boiler foaming. The stability of the bubbles need only be such that the bubbles last but a very few seconds after passing the plane of the water level indicated in the water glass.

I have now found that the slight degree of stabilization of bubbles which suffices to cause foaming of boiler water may be largely or completely inhibited by the addition to the water of tri-lauroyl diethylene triamine, tetralauroyl triethylene tetramine, tetralauroyl tetraethylene pentamine, or pentacapryl tetraethylene pentamine. These are novel compounds not heretofore prepared.

It is therefore an important object of the present invention to provide novel methods and compositions for preventing foaming in steam boilers, particularly by incorporation with said water of the above disclosed amides.

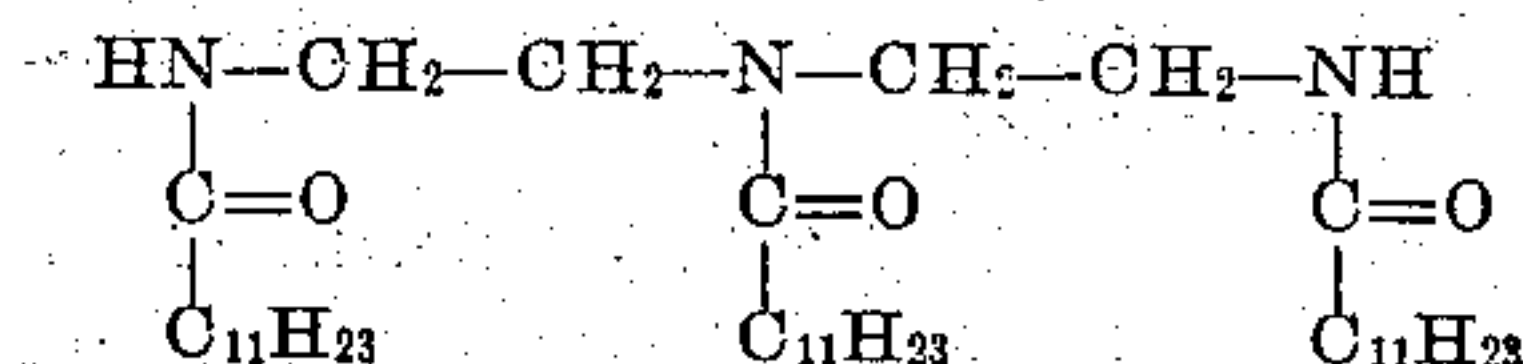
Other and further features and objects of the present invention will become apparent from the following detailed disclosure and appended claims.

The foam inhibiting compositions of the present invention may be introduced into steam boiler water in the form of colloidal dispersions that may be stabilized with tannin, gum arabic, pectin, or the like. If desired, the foam inhibiting compositions may be introduced into the boiler in the form of a solution in an appropriate sol-

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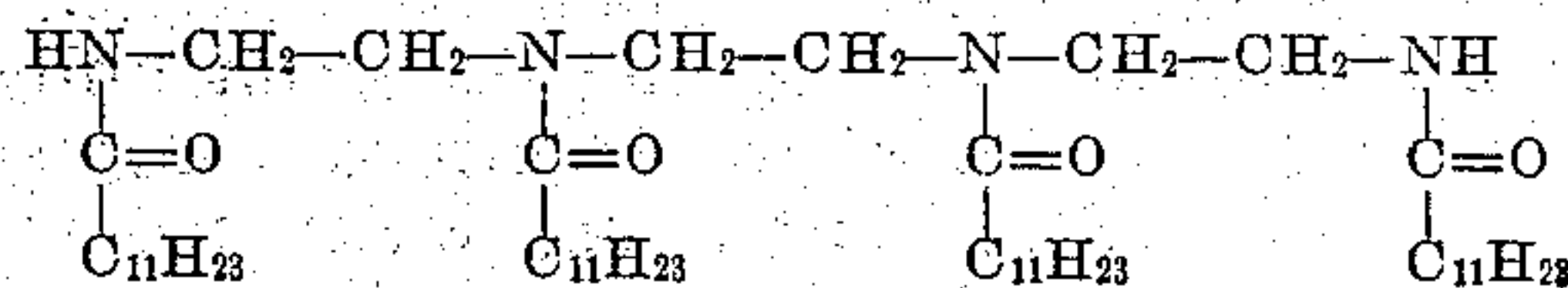
vent, such as isopropyl alcohol or the like. The dosages required are generally quite small, on the order of some few parts per million of boiler water. In general, from 0.1 to about 50 parts per million of foam inhibiting compounds may be added to boiler water, preferably in combination with tannin. Addition of foam inhibiting compounds may be repeated as required to prevent foaming.

Trilauroyl diethylene triamine having the formula:



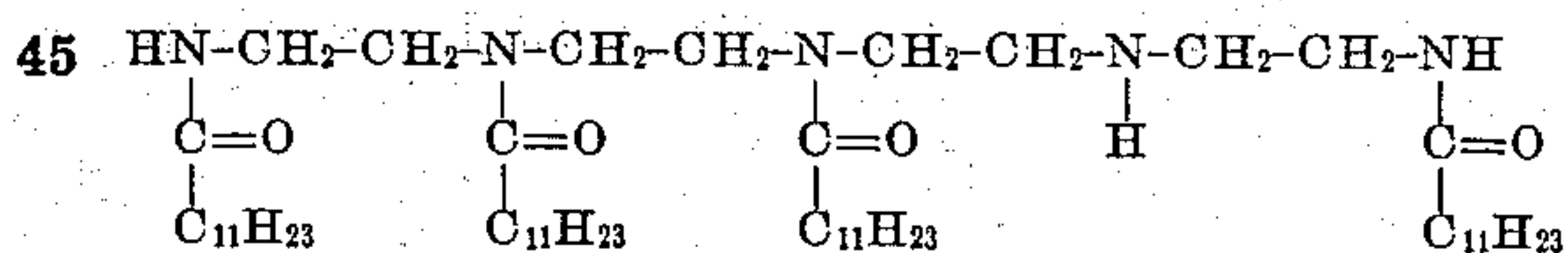
may be prepared as follows. 75 grams of lauric acid were heated with 12 grams diethylene triamine for 6 hours at 180° to 200° C. Titration of the reaction mass with $\frac{1}{10}$ normal alcoholic solution of sodium hydroxide, using Poirrier's Blue as indicator, showed that the reaction mass contained 1.4% of unreacted acid. The product is soluble in benzene and ethyl acetate and contains 6.65% nitrogen, as compared with the theoretical nitrogen content of 6.46%.

Tetralauroyl triethylene tetramine, having the formula:



may be prepared as follows. 75 grams of lauric acid were heated with 17 grams triethylene tetramine at 180° to 220° C. for 6 hours. Titration of the reaction mass with $\frac{1}{10}$ normal alcoholic sodium hydroxide solution, using Poirrier's Blue as indicator, showed that reaction mass contained 4.8% unreacted acids. The product is soluble in benzene and ethyl acetate and contains 7.22% nitrogen, as compared with the theoretical nitrogen content of 6.73%.

Tetralauroyl tetraethylene pentamine, having the formula:

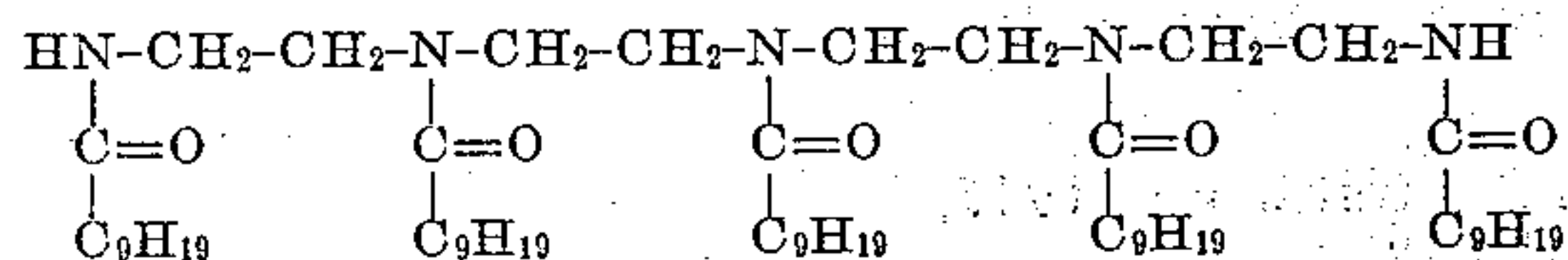


may be prepared as follows. 75 grams lauric acid were heated with 22 grams tetraethylene pentamine at 180° to 200° C. for 6 hours. Titration of the reaction mass with $\frac{1}{10}$ normal alcoholic sodium hydroxide solution, using Poirrier's Blue as indicator, showed that reaction mass contained 3.27% unreacted acid. The product is soluble in

ethyl acetate, and contains 7.60% nitrogen, as compared with the theoretical nitrogen content of 7.60%.

Tetralauroyl tetraethylene pentamine may also be prepared as follows. 80 grams of lauric acid were heated with 19 grams of tetraethylene pentamine at from 175 to 185° C. for 21 hours, yielding a product having an acid number of 28.1. The reaction mass obtained had a nitrogen content of 7.12%. Correction for the acid content indicates a nitrogen content of 7.90%.

Pentacapryl tetraethylene pentamine, having the formula:



may be prepared as follows. 345 grams capric acid, 54 grams tetraethylene pentamine and 20 grams of dry silica gel were heated together for 30 hours at from 160 to 210° C. Titration of the reaction mass with $\frac{1}{10}$ normal alcoholic sodium hydroxide solution, using Poirrier's Blue as indicator, showed that 35.4% of the acid had not reacted. The unreacted capric acid was removed by vacuum distillation, 124.2 grams being recovered. The residue from the distillation was recrystallized from 300 cc. hot acetone. The insoluble material which was filtered off weighed 72.9 grams and formed a light brown powder melting at 107° to 110° C. and having a nitrogen content of 7.29%, compared with a theoretical nitrogen content of 7.3%.

Pentacapryl tetraethylene pentamine may also be prepared as follows. 5 lbs. of capric acid and 1.1 lb. of tetraethylene pentamine were placed in a 4 liter beaker and heated with constant stirring over a free flame at 190° to 200° C. for 35 hours. At this time, the free acidity had dropped to 5.56%. The product obtained had a nitrogen content of 8.30% and, corrected for the unreacted acid, a nitrogen content of 7.29%.

The efficiency as foam inhibitor of the above identified compositions has been determined by testing in a laboratory boiler operating under 200 lbs. per sq. in. pressure and containing 500 cc. of a foaming water made up by adding to 1 liter distilled water $\frac{1}{2}$ gram $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, 2 grams anhydrous Na_2CO_3 and 0.3 gram anhydrous Na_2SO_4 . The above identified compositions were added to the foaming water in 1 mg. portions. Trilauroyl diethylene triamine was found to inhibit foaming for 164 minutes; tetralauroyl triethylene tetramine, for 170 minutes; tetralauroyl tetraethylene pentamine, for 124 minutes; and pentacapryl tetraethylene pentamine, for 198 minutes.

In this connection, it should be noted that tetracapryl tetraethylene pentamine as well as the caproyl amides of triethylene tetramine and diethylene triamine are not good foam inhibitors; nor are trilauroyl tetraethylene pentamine, trilauroyl triethylene tetramine and dilauroyl diethylene triamine.

Many details in composition and procedure

may be varied through a wide range without departing from the principles of this invention and it is, therefore, not my purpose to limit the patent granted on this invention otherwise than necessitated by the scope of the appended claims.

I claim as my invention:

1. The method of generating steam from a boiler water having a tendency to foam on boiling which comprises dispersing into said water a material selected from the group consisting of trilauroyl diethylene triamine, tetralauroyl triethylene tetramine, tetralauroyl tetraethylene pentamine and pentacaproyl tetraethylene pentamine, and heating the resulting aqueous dispersion to the boiling point, said material being added in an amount sufficient substantially to inhibit the tendency of said water to foam on boiling.

2. The method of generating steam from a boiler water having a tendency to foam on boiling which comprises dispersing into said water from 0.1 to 50 parts per million of a material selected from the group consisting of trilauroyl diethylene triamine, tetralauroyl triethylene tetramine, tetralauroyl tetraethylene pentamine and pentacaproyl tetraethylene pentamine, said material functioning to inhibit the tendency of said water to foam on boiling.

3. The method of generating steam from a boiler water having a tendency to form on boiling which comprises dispersing from 0.1 to 50 parts per million of pentacaproyl tetraethylene pentamine into said water, and heating the resulting aqueous dispersion to the boiling point, said pentacaproyl tetraethylene pentamine functioning to inhibit the tendency of said water to foam on boiling.

4. The method of generating steam from a boiler water having a tendency to foam on boiling which comprises dispersing from 0.1 to 50 parts per million of tetralauroyl tetraethylene pentamine into said water, and heating the resulting aqueous dispersion to the boiling point, said tetralauroyl tetraethylene pentamine functioning to inhibit the tendency of said water to foam on boiling.

5. The method of generating steam from a boiler water having a tendency to foam on boiling which comprises dispersing from 0.1 to 50 parts per million of tetralauroyl triethylene tetramine into said water, and heating the resulting aqueous dispersion to the boiling point, said tetralauroyl triethylene tetramine functioning to inhibit the tendency of said water to foam on boiling.

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The following references are of record in the file of this patent:

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