622359

COMMONWEALTH of AUSTRALIA
Patents Act 1952

APPLICATION FOR A STANDARD PATENT

I/We

W. R. Grace & Co.-Conn.

of

1114 Avenue of the Americas, New York, New York, 10036, United States of America

hereby apply for the grant of a Standard Patent for an invention entitled:

Ethoxylated N,N-bis-phosphonomethyl 2-(hydroxy) ethylamine N-oxides and water soluble salts thereof

which is described in the accompanying complete specification.

Details of basic application(s):-

Number	Convention Country	Date
451062	United States of America	15 December 1989

The address for service is care of DAVIES & COLLISON, Patent Attorneys, of 1 Little Collins Street, Melbourne, in the State of Victoria, Commonwealth of Australia.

DATED this TWENTY NINTH day of NOVEMBER 1990

To: THE COMMISSIONER OF PATENTS

....
a member of the firm of
DAVIES & COLLISON for
and on behalf of the
applicant(s)

Davies & Collison, Melbourne

M 023025 291190

Ref. 04-7576
USSN 451,062

COMMONWEALTH OF AUSTRALIA

PATENTS ACT 1952

DECLARATION IN SUPPORT OF CONVENTION OR NON-CONVENTION APPLICATION FOR A PATENT

Insert title of invention.

In support of the Application made for a patent for an invention entitled: "ETHOXYLATED N,N-BIS-PHOSPHONOMETHYL2-(HYDROXY) ETHYLAMINE N-OXIDES AND WATER-SOLUBLE SALTS THEREOF"

Insert full name(s) and address(es) of declarant(s) being the applicant(s) or person(s) authorized to sign on behalf of an applicant company.

I ~~We~~ Steven T. Trinker

of W.R. Grace & Co.-Conn.

of 1114 Avenue of the Americas
New York, New York 10036
United States of America

Cross out whichever of paragraphs 1(a) or 1(b) does not apply

1(a) relates to application made by individual(s)

1(b) relates to application made by company; insert name of applicant company.

do solemnly and sincerely declare as follows :–

1. (a) XXXXXXXXXXXXXXXXXXXXXXXXX

or (b) I am authorized by

W.R. Grace & Co.-Conn.

the applicant............. for the patent to make this declaration on its ~~XXX~~ behalf.

Cross out whichever of paragraphs 2(a) or 2(b) does not apply

2(a) relates to application made by inventor(s)

2(b) relates to application made by company(s) or person(s) who are not inventor(s); insert full name(s) and address(es) of inventors.

2. (a) XXXXXXXXXXXXXXXXXXXXXXXXXXXXX

or (b)
1. Chih Ming HWA
of 1041 Nightingale Drive, Palatine, Illinois 60067
2. John Arthur KELLY
of 1005 Abbey Drive, Crystal Lake, Illinois 60014
3. Mita ADHYA
of 984 Hampton Park, Barrington, Illinois 60010
All of United States of America

~~XX~~ are the actual inventorS.......... of the invention and the facts upon which the applicant............ is ~~XX~~ entitled to make the application are as follows :–

State manner in which applicant(s) derive title from inventor(s)

The actual inventors assigned the invention to the said applicant.

Cross out paragraphs 3 and 4 for non-convention applications. For convention applications, insert basic country(s) followed by date(s) and basic applicant(s).

3. The basic application............ as defined by Section 141 of the Act was ~~XXX~~ made

in United States of America on the 15 December 1989

by Chih Ming Hwa, John Arthur Kelly and Mita Adhya

in .... on the ....

by ....

in .... on the ....

by ....

4. The basic application.......... referred to in paragraph 3 of this Declaration was ~~XXX~~ the first application.......... made in a Convention country in respect of the invention the subject of the application.

Insert place and date of signature.

Declared at Columbia, MD U.S.A. this 15th day of August, 1990

Signature of declarant(s) (no attestation required)

Steven T. Trinker

Steven T. Trinker
Assistant Secretary

Note: Initial all alterations.

DAVIES & COLLISON, MELBOURNE and CANBERRA.

(12) **PATENT ABRIDGMENT** (11) Document No. **AU-B-67049/90**
(19) **AUSTRALIAN PATENT OFFICE** (10) Acceptance No. **622359**

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(54) Title
ETHOXYLATED N,N-BIS-PHOSPHONOMETHYL 2-(HYDROXY) ETHYLAMINE N-OXIDES AND WATER SOLUBLE SALTS THEREOF

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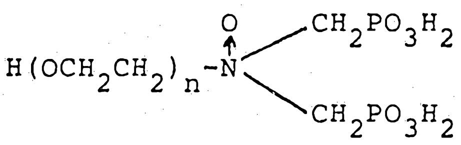
(71) Applicant(s)
W.R. GRACE & CO.-CONN.

(72) Inventor(s)
CHIH MING HWA; JOHN ARTHUR KELLY; MITA ADHYA

(74) Attorney or Agent
DAVIES COLLISON CAVE , 1 Little Collins Street, MELBOURNE VIC 3000

(57) Claim

1. A compound of the formula



$$H(OCH_2CH_2)_n-\overset{\overset{O}{\uparrow}}{N}\begin{matrix} CH_2PO_3H_2 \\ CH_2PO_3H_2 \end{matrix}$$

where n is an integer from 2 to 5 and water soluble salts thereof.

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COMPLETE SPECIFICATION

NAME & ADDRESS
OF APPLICANT:

W. R. Grace & Co.-Conn.
1114 Avenue of the Americas
New York New York 10036
United States of America

NAME(S) OF INVENTOR(S):

Chih Ming HWA
John Arthur KELLY
Mita ADHYA

ADDRESS FOR SERVICE:

DAVIES & COLLISON
Patent Attorneys
1 Little Collins Street, Melbourne, 3000.

COMPLETE SPECIFICATION FOR THE INVENTION ENTITLED:

Ethoxylated N,N-bis-phosphonomethyl 2-(hydroxy) ethylamine N-oxides and water soluble salts thereof

The following statement is a full description of this invention, including the best method of performing it known to me/us:-

FIELD OF THE INVENTION

This invention relates to organic phosphonate compounds and more particularly the organic phosphonates which can be used as water treatment agents.

BACKGROUND OF THE INVENTION

Much recent research has concerned development of organic water treatment agents for use in scale or corrosion control. Organic corrosion inhibitors which can reduce reliance on the traditional inorganic inhibitors are particularly sought. Among the organic agents successfully employed for water treatment are numerous organic phosphonates. These compounds may generally be employed without detrimentally interfering with other commercial water treatment additives. Phosphonic acid compounds have also been used in other fields for such purposes as flame retardants, plasticizers, lubricants and surfactants.

U.S. Patent No. 3,474,133 discloses that certain organo-phosphono-amine oxide compounds can be prepared by oxidizing organo-phosphono amine with a suitable oxidizing agent. For instance ethanol bis(dihydrogen phosphonomethyl) amine can be reacted with H_2O_2 to yield ethanol bis(dihydrogen phosphonomethyl) amine oxide (i.e. $HOCH_2CH_2N(O)\ (CH_2PO_3H_2)_2$); and tris(dihydrogen phosphonomethyl) amine can be reacted with H_2O_2 to yield tris(dihydrogen phosphonomethyl) amine oxide (i.e. $ON(CH_2PO_3H_2)_3$). It is disclosed that the organo-phosphono amine oxides have utility in practically all fields of organic chemistry wherein their acidic or salt and/or amine oxide properties can be utilized; and the various utilities indicated for the compounds in such fields include utility as sequestering or chelating agents, water treating agents, stabilizers for peroxy compounds and corrosion inhibitors. In particular, the acids and water soluble salts of the tris(phosphono lower alkylidene) amine oxides are reported to exhibit the property of being effective sequestering agents for metal ions in alkaline mediums. For example, the penta sodium salt of tris(dihydrogen phosphonomethyl) amine oxide is reported to sequester calcium ions in alkaline media in over a mole per mole basis.

U.S. Patent No. 3,336,221 to Ralston discloses that compounds having a methylphosphonic acid (or alkali metal or ammonium salts thereof) bonded to a nitrogen atom (e.g. pentasodium amino tri(methyl phosphonate) and phosphonomethyl ethanolamines) are threshold active compounds, useful for inhibiting the precipitation of various scale-forming compounds (e.g. calcium carbonate).

U.S. Patent No. 3,214,454 to Blaser et al discloses certain acylation products of phosphorous acid (e.g. hydroxyethylidene diphosphonic acid) and use thereof as complex formers for metal ions (e.g. calcium). Delay of calcite precipitation by the use of substoichiometrical amounts of the compounds is disclosed, as is the comparative effectiveness of certain products in preventing scale formation (e.g. in boilers, tubes, etc.).

While as indicated above, various phosphonates have proved useful for particular water treatment applications, many of them nevertheless have important disadvantages when treating water under certain conditions. For example, many phosphonates such as tri(methylphosphonic acid) are not chlorine resistant and thus degrade in the presence of free chlorine which is commonly used as a disinfectant or biocide in many aqueous systems. Other phosphonates including tris(phosphono lower alkylidene) amine oxide compounds such as tris(dihydrogen phosphonomethyl) amine oxide are considered very sensitive to calcium hardness and are prone to form calcium phosphonate precipitates. There is thus a continuing need for safe and effective water treating agents which are sufficiently versatile to be used when substantial calcium and/or free chlorine is present in the water to be treated.

SUMMARY OF THE INVENTION

Ethoxylated N,N-bis-phosphonomethyl 2-(hydroxy) ethylamine N-oxides and their water soluble salts are disclosed in accordance with this invention. These compounds are particularly useful as water treatment agents which can be employed for the control of scale and/or corrosion.

It is an object of this invention to provide a water treatment agent useful for corrosion and/or scale control.

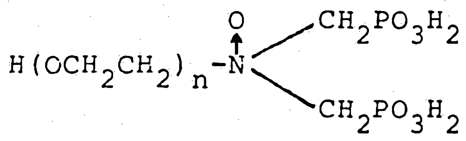
It is another object of this invention to provide a water treatment agent which is considered calcium insensitive.

It is yet another object of this invention to provide a water treatment agent which is resistant to free chlorine in water.

These and other objects and advantages of the present invention will become apparent from the detailed description of the invention which follows.

DETAILED DESCRIPTION OF THE INVENTION

This invention relates to the certain ethoxylated N,N-bis-phosphonomethyl 2-(hydroxy)ethylamine N-oxides and their water soluble salts. The ethoxylated N,N-bis-phosphonomethyl 2-(hydroxy)ethylamine N-oxides of this invention include those which may be represented by the general formula



where n is an integer from 2 to 5. The ethoxylated N,N-bis-phosphonomethyl 2-(hydroxy)ethylamine N-oxides (e.g. N,N-bis-phosphonomethyl 2-(hydroxyethoxy)ethylamine N-oxide) may be prepared by conventional oxidation of the trisubstituted nitrogen of the corresponding tertiary phosphonomethyl amine (e.g. N,N-bis-phosphonomethyl 2-(hydroxyethoxy)ethylamine) with a suitable oxidizing agent. Suitable oxidizing agents are generally oxidizing agents which contain an O-O linkage (peroxide compound) and have oxidizing action. Suitable oxidizing agents are considered to include hydrogen peroxide, substituted peroxides and additional compounds of hydrogen peroxide such as the peroxide of sodium and the peroxide of potassium, urea percompounds, percarbonates, perborates, persulfates and the peracids such as persulfuric acid, peracetic acid, peroxymonophosphoric acid and the like as well as their water-soluble salt compounds such as sodium, potassium, ammonium and organic amine salts. In general, the oxidation process is usually carried out in an aqueous medium.

Hydrogen peroxide is the preferred oxidizing agent. Reference is made to Hoh et al. "Hydrogen Peroxide Oxidation of Tertiary Amines", The Journal of the American Oil Chemists' Society, Vol. LV, No. 7, pp 268-271 (July 1963) and Lake et al. "Recent Advances in Fatty Amine Oxides. Part I. Chemistry and Preparation", The Journal of the American Oil Chemists' Society, Vol. 40, No. 11, pp. 628-631 (November 1963) for discussion of such oxidations. In general, a solution of the tertiary amine may be advantageously reacted at a pH of about 10 with about 20% excess hydrogen peroxide. It is preferred to use concentrations of hydrogen peroxide above about 2% by weight of the reaction medium.

The water soluble salts are readily prepared from the phosphonomethyl amine oxide by neutralizing the phosphonic acid group with a stoichiometric amount of a base or salt that contains essentially the desired cation or by conversion of the ethoxylated N,N-bis-phosphonomethyl 2-(hydroxy)-ethylamine to a salt form prior to its oxidation to the amine oxide. Bases and salts of acids such as those containing an alkali metal, alkaline earth metal, zinc, aluminum, ammonia and amines are especially suited, with sodium and potassium salts being preferred. For example, to make a sodium salt, a free acid of N,N-bis-phosphonomethyl 2-(hydroxyethoxy)ethylamine N-oxide can be neutralized with a stoichiometric amount of a base containing sodium cation, such as sodium hydroxide.

Other bases or salts which can be reacted with the free acids to produce salt compounds of the instant invention include the inorganic alkali metal salts, oxides and hydroxides such as Na_2O, Na_2CO_3, KOH, K_2O, LiOH, Li_2CO_3, CsOH, Cs_2CO_3, other inorganic salts and hydroxides such as $Al(OH)_3$, $Al_2(SO_4)_3$, $Al(NO_3)_3$ and $ZnSO_4$, and amines, particularly low molecular weight amines (i.e. amines having a molecular weight below about 300), and more particularly the alkyl amines, alkylene amines and alkanol amines containing not more than 2 amine groups such as ethyl amine, diethylamine, propyl amine, propylene diamine, hexylamine, 2-ethyl hexylamine, N-butylethanol amine, triethanolamine and the like.

The tertiary phosphonomethyl amine, N,N-bis-phosphonomethyl 2-(hydroxyethoxy)ethylamine which is useful as a reagent for preparing the preferred compounds of the instant invention can be prepared by the known reaction of a nitrogenous material (i.e. 2-(hydroxyethoxy)ethylamine) with a compound containing a carbonyl group (i.e. formaldehyde) and orthophosphorous acid.

For the foregoing methods of preparation, reaction conditions such as temperatures, pH and time for reaction can be varied with the optimum conditions for the reactions being readily ascertained by those skilled in the art. Reference is made to U. S. Patent No. 3,429,914, which is hereby incorporated herein in its entirety by reference, for a discussion of the preparation of organo-phosphono amines and organo-phosphono-amine oxides.

The ethoxylated N,N,-bis-phosphonomethyl 2-(hydroxy) ethylamine N-oxides of this invention, especially N,N-bis-phosphonomethyl 2-(hydroxyethoxy)ethylamine N-oxide (i.e. n is 2) and its water soluble salts, are considered useful in water treatment applications. It has been found that compounds of this invention can be used to inhibit corrosion of metal in aqueous systems as well as to inhibit the formation of scale in such systems. Thus, both the formation of scale and the corrosion of metals can be inhibited in an aqueous system by addition of the compounds of this invention to the system water. Moreover compounds of this invention have been found calcium insensitive and resistant to free chlorine in aqueous solution. In addition metal surfaces can be passivated by the ethoxylated N,N-bis-phosphonomethyl 2-(hydroxy)ethylamine N-oxides. Accordingly the compounds of this invention are considered particularly versatile water treatment agents.

Practice of the invention will become further apparent from the following non-limiting examples.

EXAMPLE I

A solution of 2-(hydroxyethoxy) ethylamine (52.5 grams, 0.5 mole) in water (10 grams) was cooled in an ice-water bath. Phosphorous acid (99%, 82.0 grams, 1 mole) was added slowly to the solution, followed by concentrated hydrochloric acid (37%, 29.6 grams, 0.3 mole). The resultant solution was slowly heated to reflux and formaldehyde (37%, 86.08 grams, 1.07 mole) was added dropwise to the refluxing solution. The solution was allowed to reflux for 13 hours. Analysis of the P^{31}NMR spectra of the product indicates N,N-bis-phosphonomethyl 2-(hydroxyethoxy) ethylamine.

EXAMPLE II

To a solution of N,N-bis-phosphonomethyl 2-(hydroxyethoxy) ethylamine (53.8%, 120.19 grams, 0.2206 mole), sodium hydroxide solution (50%, 82 grams, 1.025 mole) was added dropwise until pH 10.1 was reached. Hydrogen peroxide solution (35.7%, 24.27 grams, 0.255 mole) was added dropwise to the resultant solution. The solution was allowed to stir for 17 hours at ambient temperature. Analysis of the P^{31}NMR spectra of the product indicates N,N-bis-phosphonomethyl 2-(hydroxyethoxy) ethylamine N-oxide.

EXAMPLE III

Calcium sensitivity for the phosphonomethyl amine oxide N,N-bis-phosphonomethyl-2-(hydroxyethoxy) ethylamine N-oxide, was tested using a cloud point test wherein the phosphonomethyl amine oxide was respectively added to a 250-ml beaker containing a hard water solution having a temperature of 60°C, having a pH of 8.3, and containing 500 ppm calcium ion (as $CaCO_3$) and 0.005M borate buffer. Over 100 ppm of N,N-bis-phosphonomethyl-2-(hydroxyethoxy) ethylamine N-oxide, was added without reaching a cloud point (i.e. a point at which the solution becomes turbid). Accordingly the N,N-bis-phosphonomethyl-2-(hydroxyethoxy) ethylamine N-oxide was considered "calcium insensitive".

It is noted that not all phosphonates, nor even all organo phosphono amine compounds exhibit the calcium insensitivity of N,N-bis-phosphonomethyl-2(hydroxyethoxy) ethylamine N-oxide. For example, the addition of about 5 ppm amino tri(methylphosphonic acid) N-oxide or the addition of about 7 ppm hydroxyethylidene diphosphonic acid each produces a cloud point in the cloud point test described in Example III.

EXAMPLE IV

A test solution was formulated to approximate a 4-fold concentrate of Chicago tap water. The water had an initial pH of about 8.5. Two mild steel coupons were weighed and suspended for three days in an air-sparged sample of the solution at 54°C. The steel coupons were then removed and reweighed, and an average corrosion rate (in mils per year) over the three days was calculated on the basis of coupon weight loss. The results are provided in Table A below (Run 1). Three additional runs (Runs 2, 3 and 4) were made using the same procedure except that 15 ppm, 30 ppm and 45 ppm N,N-bis-phosphonomethyl-2-(hydroxyethoxy)ethylamine N-oxide were respectively added to the test solution. The calculated coupon corrosion rates for these runs are also shown in Table A below.

TABLE A

Run	Additive	Additive Concentration (ppm)	Corrosion Rate (mpy)
1.	None	___	48.0
2.	N,N-Bis-phosphonomethyl-2-(hydroxyethoxy) ethylamine N-oxide	15	13.4
3.	N,N-Bis-phosphonomethyl-2-(hydroxyethoxy) ethylamine N-oxide	30	3.1
4.	N,N-Bis-phosphonomethyl-2-(hydroxyethoxy) ethylamine N-oxide	45	2.9

There was no pH control during the test of this example and the final pH of the test solutions after the three day test ranged from about 8.8 to 9.5.

EXAMPLE V

A potentiodynamic polarization test was conducted for demonstrating passivation by a solution of 30 ppm N,N-bis-phosphonomethyl-2-(hydroxyethoxy)ethylamine N-oxide. In this test a disc of 1010 mild steel was polished to 600 grit finished, ultrasonicly cleaned in soap water, and rinsed with acetone. The solution was subjected to argon deaeration to achieve an oxygen concentration of less than 0.5 ppm. The solution was adjusted to a pH of 8.5 by using sodium hydroxide or hydrochloric acid and heated to 55°C by a water bath. The disc surface is reduced for 200 seconds at -1 volt against saturated calomel electrode. During the potentiodynamic polarization measurements, the potential is swept at 1 millivolt per second.

The tabularized results for these runs are shown in Table B below.

TABLE B

Potential (E) (Volts/Saturated Calomel Electrode)	Current Density (I) (Amperes/Square Meter)
	N,N-Bis-phosphonomethyl -2-(hydroxyethoxy) ethylamine N-oxide (30 ppm)
-0.850	0.357
-0.826	0.207
-0.802	0.042
-0.778	0.031
-0.750	0.103
-0.726	0.134
-0.702	0.201
-0.678	0.234
-0.654	0.271
-0.630	0.286
-0.602	0.300
-0.578	0.319
-0.554	0.325
-0.546	0.325
-0.538	0.326
-0.530	0.317
-0.522	0.312
-0.514	0.303
-0.506	0.309
-0.498	0.288
-0.490	0.285
-0.482	0.284
-0.474	0.276
-0.466	0.291
-0.458	0.295
-0.450	0.283
-0.434	0.305
-0.426	0.321
-0.418	0.344
-0.410	0.347
-0.402	0.368

An interval of relatively constant current density over a range of potential is considered indicative of passivation. The current density over the potential range of -0.498 to -0.450 is considered indicative of passivation of metal surfaces in the presence of N,N-bis-phosphonomethyl-2-(hydroxyethoxy) ethylamine N-oxide.

EXAMPLE VI

A two ppm solution of N,N-bis-phosphonomethyl-2-(hydroxyethoxy)ethylamine N-oxide in zero hardness water was heated for 24 hours at 60°C. The amount of organic phosphonate which was converted to orthophosphate was then determined. Additional runs (runs 2 and 3) were made using the same solution except that 10 ppm and 20 ppm of NaOCl were respectively added prior to heating. The results are shown in Table C below.

TABLE C

Run	Additive	NaOCl Added (ppm)	% Conversion
1	N,N-Bisphosphonomethyl--2-(hydroxyethoxy) ethylamine N-oxide	0	0.4
2	N,N-Bisphosphonomethyl -2-(hydroxyethoxy) ethylamine N-oxide	10	4.6
3	N,N-Bisphosphonomethyl -2-(hydroxyethoxy) ethylamine N-oxide	20	5.4

For comparison, N,N-bis-phosphonomethyl ethanolamine and amino tri(methylphosphonic acid) showed 100% and 93% conversion, respectively, under the above testing conditions in the presence of 10 ppm NaOCl. Unlike N,N-bis-phosphonomethyl ethanolamine and amino tri(methylphosphonic acid), the N,N-bis-phosphonomethyl-2-(hydroxyethoxy) ethylamine N-oxide of the instant invention is chlorine resistant.

EXAMPLE VII

The ability of the calcium insensitive phosphonomethyl amine oxide, N,N-bis-phosphonomethyl-2-(hydroxyethoxy) ethylamine N-oxide, to also inhibit calcium carbonate formation was demonstrated using a threshold inhibitor test. In this test 800 ml of a test solution containing 400 ppm calcium (as Ca) and 400 ppm bicarbonate (as HCO_3) in a 1000 ml beaker was stirred with a magnetic stir bar and heated using a stainless steel immersion heater to 49°C. The pH was monitored during heating and kept at pH 7.15 with addition of dilute HCl. After the temperature of 49°C was achieved, 0.1 N NaOH was added to the test solution at a rate of 0.32 ml/min using a syringe pump and the rise in pH was monitored. A decrease or plateau in the rate of pH increase is observed when calcium carbonate starts to precipitate, and the pH at which this decrease or plateau is observed is termed the critical pH. The critical pH for the test solution is shown in Table D below along with the total milliequivalents per liter of hydroxide (as NaOH) added to reach the critical pH.

The procedure was repeated using test solution to which 5 ppm of the calcium insensitive N,N-bis-phosphonomethyl-2-(hydroxyethoxy)ethylamine N-oxide was added. A run was also made using amino tri(methylphosphonic acid) N-oxide which as indicated above is considered calcium sensitive when compared to N,N-bis-phosphonomethyl-2-(hydoxyethoxy)ethylamine N-oxide. The results are shown in Table D below.

TABLE D

Run	Additive	Critical pH	NaOH added to reach critical pH (meq/l)
1	Blank (without treatment)	7.69	0.48
2	N,N-Bis-phosphonomethyl-2-(hydroxyethoxy) ethylamine N-oxide	8.96	2.78
3	Amino tri(methylphosphonic acid) N-oxide	8.50	1.34

As shown in Table D, use of the phosphonomethyl amine oxide of the present invention raised the critical pH and generally resulted in substantially more sodium hydroxide addition before the critical pH was reached. This phosphonomethyl amine oxide is thus an effective threshold inhibitor, capable of inhibiting calcium carbonate precipitation.

EXAMPLE VIII

Scale formation was further tested using an apparatus comprising a covered 28-liter basin, a centrifugal pump which withdraws liquid from the bottom of the basin and circulates it through tubing respectively to a needle valve which allows flow control, a flow meter which allows flow measurement, a glass housing containing an immersion heater for heating the liquid which is returned to the basin. A cooling coil is provided in the basin and is connected such that tap water may be circulated through the cooling coil. The liquid temperature is controlled using a thermoregulator which activates a solenoid valve which controls the flow of tap water through the coil. A pH probe is also located in the basin and is operably connected to a pH controller which in turn controls a pair of solenoid valves which respectively control flow of 0.5 N NaOH and 0.2 N H_2SO_4 from 1-liter containers to the basin.

Five liters of test solution containing 600 ppm total hardness (as $CaCO_3$) was transfered to the basin and circulated at a flow rate of 1.4 ft. per second using the centrifugal pump. The pH was controlled within the range of 8.0-8.2 and the variable transformer was turned on such that the heat flux for the immersion heater was 10.9 KBTU per square foot per hour. The cooling coil was operated such that the outlet water from the basin was controlled at 60°C. After six hours the power transformer and the pH controller were turned off and the pH probe was removed from the basin. The water in the basin was cooled rapidly by resetting the thermoregulator to provide tap water circulation through the cooling coil. A sample of test solution was removed from the basin when it had cooled to 35°C, and it was analyzed for total hardness. The results are shown in Table E below. The reduction in total hardness was considered indicative of the scale formation in the system.

The run was repeated using the above procedure except that 2 ppm N,N-bis-phosphonomethyl-2-(hydroxyethoxy) ethylamine N-oxide was added to the solution prior to heating. The total hardness of the test solution at the conclusion of these runs are shown in Table E below, as is the reduction in total hardness, and the calculated inhibition of scale formation.

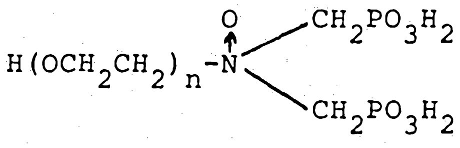
TABLE E.

Run	Additive	Test Solution Total Hardness (ppm) Start	End	Change	Calculated Scale Inhibition %
1	Blank (without treatment)	600	134	466	———
2	N,N-Bis-phosphonomethyl-2-(hydroxyethoxy)ethylamine N-oxide (2ppm)	600	580	20	95.7

The Examples encompass particular embodiments of the invention. Other embodiments will become apparent to those skilled in the art from a consideration of the specification or practice of the invention disclosed herein. It is understood that modifications and variations may be produced without departing from the spirit and scope of the novel concepts of this invention. It is further understood that the invention is not confined to the particular formulations and examples herein illustrated, but it embraces such modified forms thereof as come within the scope of the following claims.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS.

1. A compound of the formula



$$H(OCH_2CH_2)_n-\overset{O}{\overset{\uparrow}{N}}(CH_2PO_3H_2)_2$$

where n is an integer from 2 to 5 and water soluble salts thereof.

2. A compound according to Claim 1 wherein n is 2.

3. A compound according to Claim 1 wherein the compound is selected from N,N-bis-phosphonomethyl-2-(hydroxyethoxy)ethylamine N-oxide and its sodium and potassium salts.

4. A compound according to claim 1, substantially as hereinbefore described with reference to the examples.

Dated this 13th day of January, 1992
W.R.GRACE & CO.-CONN
By its Patent Attorneys
DAVIES COLLISON CAVE