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SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO

E. _____ S. _____

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO



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Referencia: Expediente No. 03.106.165

Solicitud de Patente de Invención titulada: "Procedimiento para la
preparación de alquilcarbamatos de alquil-n-(3-dimetilamino) "

Solicitante: Bayer Cropscience AG

SOLICITUD DE AGILIZACIÓN DEL TRÁMITE

ÁLVARO CORREA ORDÓÑEZ, mayor de edad y vecino de Bogotá D.C., identificado tal como
aparece al pie de mi firma, en mi carácter de apoderado de la sociedad en referencia, solicito
respetuosamente que se agilice el trámite para el expediente de la referencia.

Atentamente,

ÁLVARO CORREA ORDÓÑEZ

C.C.No.79.143.366 de Usaquén

T.P.20.281

BOGOTABOXUDMS-156913-v1-P2003_000004-Memorial_de_agilización.DOC

Anexos:

DIVISIÓN DE NUEVAS CREACIONES

RADICACIÓN EXPEDIENTE No. 03-106165

EL EXTRACTO DE LA PRESENTE SOLICITUD DE PATENTE DE INVENCION

FUE PUBLICADO EN LA GACETA DE PROPIEDAD INDUSTRIAL No 536 DE

FECHA 30 DE ENERO DE 2004 EL TÉRMINO HÁBIL SEÑALADO POR LA LEY

EXPIRA EL 28 DE ABRIL DE 2004

NOTA: Dentro del plazo de los seis (6) meses siguientes a partir de la fecha de publicación en la gaceta de Propiedad Industrial, deberá solicitar y cancelar el examen de patentabilidad so pena de que la solicitud caiga en abandono, de acuerdo con lo establecido en el Artículo 44 de la Decisión 486/2000.

SE PRESENTARON OPOSICIONES POR PARTE DE TERCEROS.

SI _____

NO X

1

3,513,241

FUNGICIDAL AND FUNGISTATIC N-(DIALKYL-AMINOALKYL) - CARBAMIC ACID AND THIO-CARBAMIC ACID ESTERS

Georg-Alexander Hoyer and Ernst Albrecht Pieroh, Berlin, Germany, assignors to Schering AG Berlin & Bergkamen, Berlin, Germany

No Drawing. Filed Dec. 11, 1967, Ser. No. 689,310

Claims priority, application Germany, Dec. 17, 1966, Sch 39,978

Int. Cl. A01n 7/00, 9/12, 9/20

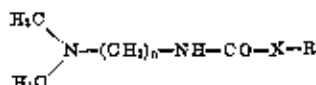
U.S. Cl. 424-300

1 Claim

ABSTRACT OF THE DISCLOSURE

Compounds containing N-(dialkylaminoalkyl)-carbamic acid esters and/or N-(dialkylaminoalkyl)-thiocarbamic acid esters are used either per se or in conjunction with other ingredients for use as fungicides or fungistats.

It has been found that compounds of the general formula:



in which R is an aliphatic hydrocarbon radical, preferably containing 1 to 8 carbon atoms, X oxygen or sulfur, and n an integer from 1 to 5, exhibit a fungicidal and fungistatic action and therefore are suitable preferably for the control of phytopathogenic fungi.

The invention relates to fungicidal and fungistatic agents containing N-(dialkylaminoalkyl)-carbamic acid esters and respectively N-(dialkylaminoalkyl)-thiocarbamic acid esters.

It is to be noted that compounds wherein in the above stated general formula, R represents a straight-chain or branched, saturated or unsaturated hydrocarbon radical with 1 to 8 carbon atoms, as for example, methyl, ethyl, propyl, butyl, pentyl, hexyl, octyl, isopropyl, isobutyl, tertiary butyl, allyl, propargyl, or butyne-(1)-yl-(3), have a particularly good and effective action for said control.

The compounds to be used according to the invention are surprisingly distinguished by an excellent action against *Pythium ultimum* which, in part, exceeds the effect of similarly acting products that have become known for this. The effect as disinfecting steep to promote germination of the seed is also considerable and better than that of heretofore known agents.

Agents which contain one or more of the aforementioned compounds can therefore find application, for example, in agriculture or horticulture as disinfecting steeps for seed treatment, for seed row treatment as so-called drill-in agent or for general soil treatment and the like.

The partly new active ingredients can be produced by methods known in the art. For example, the dimethylaminomethyl carbamic acid esters wherein n=1 are prepared by reaction of the respective carbamic acid ester with formaldehyde and dimethylamine in water at room temperature or elevated temperature. The other esters wherein n=2 to 5 may be obtained by the reaction of a dimethylaminoalkyl amine with a chloroformic acid ester or chloroformic acid thioester in a suitable solvent, possibly in the presence of an acid acceptor. As solvents, there may be used, for example, inert organic liquids, such as ether or hydrocarbons. Suitable acid acceptors are tertiary organic amines, namely triethylamine or pyridine, etc., inorganic bases, namely alkali hydroxides or carbonates, etc., or also the amine required for the reaction, which is then used in correspondingly higher quantity. The reaction proceeds smoothly in a temperature range from about

2

0 to 100° C., but can be carried out also at higher or lower temperatures. The conversion may be conducted as a one-phase or two-phase reaction, the latter with the use of non-miscible liquids, such as water and an organic solvent.

Compounds to be preferably used according to the present invention are, for example, the following:

Name of compound	Physical constant
10 N-(γ-dimethylaminoethyl)-carbamic acid propyl ester. N-(γ-dimethylaminoethyl)-carbamic acid isobutyl ester. N-(γ-dimethylaminoethyl)-carbamic acid allyl ester.	B.P. (18 mm.) 139-141° C. n _D ²⁰ =1.4490. B.P. (0.55 mm.) 100-102° C. n _D ²⁰ =1.4180. B.P. (30 mm.) 158-158° C. n _D ²⁰ =1.4599.
15 N-(γ-dimethylaminoethyl)-thiocarbamic acid-3-ethyl ester. N-(δ-dimethylaminobutyl)-carbamic acid butyl ester. N-(δ-dimethylaminobutyl)-carbamic acid ethyl ester.	Undistillable oil. n _D ²⁰ =1.4985. B.P. (18 mm.) 132-138° C. n _D ²⁰ =1.4470. B.P. (18 mm.) 140-143° C. n _D ²⁰ =1.4500.
20 N-(γ-dimethylaminopropyl)-thiocarbamic acid-3-propyl ester. N-(γ-dimethylaminopropyl)-carbamic acid butyne(1)-yl-(3) ester.	Undistillable oil. n _D ²⁰ =1.4440. B.P. (0.55 mm.) 123-124° C. n _D ²⁰ =1.4640.

The active ingredients may be used singly or as mixtures of several ingredients. If desired, other fungicides, nematocides, herbicides, disinfectants or other pest control agents may be added, depending on the purpose intended. Advantageously, the active ingredients are used in the form of suitable preparations, such as powders, scatter materials, granulates, solutions, emulsions or suspensions, with addition of liquid and/or solid vehicles or diluents and also of wetting, adhesive, emulsifying and/or dispersing aids.

Suitable liquid vehicles are water, mineral oils, or other organic solvents, such as xylol (xylene), chlorobenzene, cyclohexanol, cyclohexanone, dioxane, acetonitrile, acetic ester, dimethyl formamide and dimethyl sulfoxide, etc.

As solid vehicles, there may be used lime, kaolin, chalk, talcum, attaclay and other clays, as well as natural or synthetic silica, i.e., silicic acid.

Among surface-active substances that may be used are salts of the lignin sulfonic acids, salts of alkylated benzene sulfonic acids, sulfonated acid amines and their salts, polyethoxylated amines and alcohols.

If the active ingredients are to be used for seed disinfection, there also may be added dyes, such as new fuchsin, etc., to give the steeped seed a distinctly visible coloration.

The proportion of ingredients in the agent may vary within wide limits, the exact concentration of the ingredient used for the agents depending mainly on the quantity in which the agents are to be used for soil or seed treatment, etc. For example, the agents contain between about 1 and 80 percent by weight, preferably between about 20 and 50 percent by weight of active ingredient and about 99 to 20 percent by weight of liquid or solid vehicle and possibly up to 20 percent by weight of surface-active substances in the form of an emulsion or of a dry disinfectant.

To promote germination of the seed, the agents are applied either before sowing directly on the seed or when sowing into the furrow, i.e., so-called drilling in. When treating the soil as such, the agents are advantageously introduced into the upper soil layers to a depth of about 20 cm., such as by hoeing in.

The following illustrative examples will explain the invention.

EXAMPLE 1

Unsteamed compost soil was additionally seeded with mycelium of *Pythium ultimum*. After uniform mixing of the agents with the uninfected soil, there followed without waiting period per concentration the seeding out of 25 grains of narrow peas of the variety "Miracle of Kel-

vedon" in clay dishes holding 1 liter of soil. In the following table, there are stated the number of germinated healthy peas, the root rating (1 to 4) and the weight of the fresh plant after a cultivation time of 3 weeks at 22 to 24° C. CAPTAN (N-trichloromethylthio-tetrahydrophthalimide) was used as a reference agent. The preparations were a powder containing 20% by weight of active ingredient.

Preparation:	Active ingredients per liter of soil, mg.	Number of healthy peas	Root rating	Weight of fresh plant, g.
N-(γ-dimethylamino-propyl)-carbamic acid propyl ester.....	50 100 200	21 24 28	4 4 4	13 17 18
N-(γ-dimethylaminobutyl)-thiocarbamic acid-S-ethyl ester.....	50 100 200	20 17 20	4 4 4	13 12 14
N-(β-dimethylaminoethyl)-carbamic acid butyl ester.....	50 100 200	10 20 24	1 3 4	20 24 22
N-(γ-dimethylaminopropyl)-carbamic acid allyl ester.....	50 100 200	16 21 20	3 4 4	20 25 22
N-(γ-dimethylaminopropyl)-carbamic acid isobutyl ester.....	50 100 200	18 18 22	3 3 2	19 20 24
N-(γ-dimethylaminopropyl)-carbamic acid butyne-(1)-yl-(3) ester.....	50 100 200	9 12 20	1 1 2	4 6 17
CAPTAN.....	50 100 200	8 10 18	1 1 1	2 5 10
Steamed soil (3 control tests):				
A.....		21	4	10
B.....		24	4	16
C.....		24	4	17
Untreated soil (3 control tests):				
A.....		0	1	1
B.....		2	1	1
C.....		1	1	1

Note.—Root rating:
4=White roots without fungus necrosis.
3=White roots, slight fungus necrosis.
2=Brown roots, more advanced fungus necrosis.
1=Advanced fungus necrosis, roots rotted.

EXAMPLE 2

This example illustrates the drill-in method in sowing cotton.

Clay dishes holding 2 liters of soil were filled with unsteamed compost soil. For 20 cm. of furrow, the agents were scattered in the form of a powder containing 20 percent by weight of active ingredient. There then followed per concentration, the sowing of 25 grains of delinted cotton. In the table, there is stated the number of germinated healthy cotton seedlings and the fresh plant weight after cultivation time of 20 days at 22 to 24° C. As a reference agent MANEB (manganoethylene-bis-dithiocarbamate) was used.

Preparation:	Mg. of active ingredients per 20 cm. of furrow	Number of healthy plants	Weight of fresh plant, g.
N-(dimethylaminoethyl)-carbamic acid butyl ester.....	10 20 40 80	21 19 21 18	28 40 42 40
N-(γ-dimethylaminopropyl)-carbamic acid allyl ester.....	10 20 40 80	22 20 26 21	40 40 49 49
N-(γ-dimethylaminopropyl)-carbamic acid isobutyl ester.....	10 20 40 80	12 18 24 23	28 36 46 45
MANEB.....	10 20 40 80	12 0 11 19	24 22 30 30
Steamed soil (3 control tests):			
A.....		21	50
B.....		17	54
Untreated soil (3 control tests):			
A.....		9	22
B.....		10	28

EXAMPLE 3

The drill-in method was used in the sowing of cotton for the control of *Pythium ultimum*.

Steamed compost soil was seeded with mycelium of *Pythium ultimum* and then clay dishes holding 2 liters were filled with this soil. The stated active ingredient quantity was scattered as a 20% powder per 20 cm. of furrow. Then followed by concentration, the sowing of 25 grains of delinted cotton. In the table there are given the number of germinated healthy cotton seedlings and the weight of the fresh plant after a cultivation time of 25 days at 22 to 24° C. The reference agent in those examples was CAPTAN (N-trichloro-methylthio-tetrahydrophthalimide).

Preparation:	Active ingredient per 20 cm. of furrow, mg.	Number of healthy peas	Weight of fresh plant, g.
N-(β-dimethylaminobutyl)-carbamic acid butyl ester.....	10 20 40 80	16 23 22 28	34 50 46 40
N-(γ-dimethylaminopropyl)-carbamic acid propyl ester.....	10 20 40 80	16 17 21 24	34 33 43 47
N-(γ-dimethylaminopropyl)-thiocarbamic-S-ethyl ester.....	10 20 40 80	17 21 21 14	30 46 44 21
CAPTAN.....	10 20 40 80	4 7 15 7	6 18 20 16
Steamed soil (3 control tests):			
A.....		18	27
B.....		20	30
C.....		18	24
Untreated soil (3 control tests):			
A.....		0	9
B.....		4	4
C.....		8	18

EXAMPLE 4

Unsteamed compost soil was additionally seeded with mycelium of *Pythium ultimum*. Peas of the variety "Miracle of Kelvedon" steeped with 50-percentual product formulations, were set out 2 to 3 cm. deep in clay dishes holding 0.5 liter of soil; per concentration 25 grains. After a cultivation time of 14 days at 22 to 24° C., the number of germinated healthy peas was recorded. Comparison was made with MANEB (manganoethylene-bis-dithiocarbamate) and TMTD (tetramethyl-thiuram-disulfide).

Preparation:	0.5 g. active ingredient per kg. of seed	Number of germinated healthy peas	1.5 g. active ingredient per kg. of seed
N-(γ-dimethylaminopropyl)-carbamic acid propyl ester.....	25	21	21
N-(γ-dimethylaminobutyl)-carbamic acid ethyl ester.....	25	23	23
N-(γ-dimethylaminopropyl)-carbamic acid allyl ester.....	14	22	22
N-(γ-dimethylaminopropyl)-carbamic acid butyne-(1)-yl-(3)-ester.....	14	21	21
N-(β-dimethylaminobutyl)-carbamic acid butyl ester.....	10	17	17
N-(γ-dimethylaminopropyl)-thiocarbamic acid-S-propylester.....	20	23	23
MANEB.....	10	11	11
TMTD.....	13	10	10
Steamed soil, seed not steeped (4 control tests):			
A.....		22	22
B.....		20	20
C.....		22	22
D.....		20	20
Untreated soil, seed not steeped (4 control tests):			
A.....		1	1
B.....		2	2
C.....		0	0
D.....		3	3

From the foregoing examples taken in conjunction with the description of the invention it will be noted that

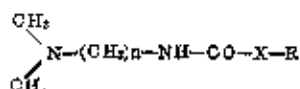
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fungicides and fungistats are provided per se or in conjunction with other ingredients and fillers acting as adjuvants to effect the desired result.

What is claimed is:

I. A method for seed and soil treatment which comprises treating said seed and soil with an effective amount of a fungicidal and fungistatic agent, said agent comprising:

- (a) from 1 to 80 percent by weight of a compound having the formula



where R is an aliphatic hydrocarbon containing 1 to 8 carbon atoms, X is oxygen or sulfur, and n is an integer from 1 to 5;

- (b) up to 20 percent by weight of a surface active substance selected from the group consisting of lignin sulfonic acid salts, salts of alkylated benzene

6

sulfonic acids, sulfonated acid amides, salts of said acid amides, polyethoxylated amines and polyethoxylated alcohols and;

- (c) from 99 to 20 percent by weight of a fungicidal diluent.

References Cited

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OTHER REFERENCES

Chem. Abstracts, vol. 65, p. 18528f (1966).

ALBERT T. MEYERS, Primary Examiner
V. D. TURNER, Assistant Examiner

U.S. Cl. X.R.

260-455, 482

[54] SALTS OF THIOCARBAMIC ESTERS WITH FUNGICIDAL AND FUNGISTATIC ACTION

[75] Inventors: Georg-Alexander Hoyer; Ernst Albrecht Pieroh, both of Berlin, Fed. Rep. of Germany

[73] Assignee: Schering AG, Berlin and Bergkamen, Fed. Rep. of Germany

[21] Appl. No.: 724,820

[22] Filed: Sep. 20, 1976

Related U.S. Application Data

[60] Continuation of Ser. No. 109,073, Nov. 10, 1970, abandoned, which is a division of Ser. No. 746,711, Jul. 18, 1968, Pat. No. 3,649,674.

[51] Int. Cl.⁴ C07C 153/09; A01N 9/12

[52] U.S. Cl. 260/455 A; 424/300

[58] Field of Search 260/455 A

[56] References Cited

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Primary Examiner—Lewis Gotts

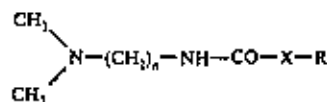
Assistant Examiner—Robert C. Whittenbaugh

Attorney, Agent, or Firm—Michael J. Striker

[57]

ABSTRACT

Salts of carbamic and thiocarbamic esters are used having fungicidal and fungistatic action, said salts having the general formula



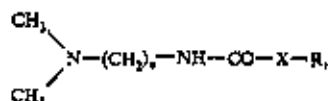
wherein R is an aliphatic hydrocarbon radical with preferably 2 to 4 carbon atoms, X is oxygen or sulfur, and n is an integer from 2 to 4, with an inorganic or organic acid.

3 Claims, No Drawings

SALTS OF THIOCARBAMIC ESTERS WITH FUNGICIDAL AND FUNGISTATIC ACTION

This is a continuation of application Ser. No. 109,073, filed Nov. 10, 1970 and now abandoned, which is a division of Ser. No. 746,711 filed July 18, 1968 and now U.S. Pat. No. 3,649,674.

It has been found that salts of compounds of the general formula



wherein

R is an aliphatic hydrocarbon radical with preferably 2 to 4 carbon atoms,

X is oxygen or sulfur and n is an integer from 2 to 4, with any inorganic or organic acids exhibit a fungicidal and fungistatic action and therefore are suitable preferably for the control of phytopathogenic fungi.

In the above stated general formula, the radical R may have, among others, the following meaning: straight-chain or branched, saturated or unsaturated aliphatic hydrocarbon radical, such as ethyl, propyl, butyl, isopropyl, tertiary butyl, allyl.

As suitable acids in connection with the invention there enter into consideration any inorganic or organic acids. Examples are generally mineral acids, mono- or poly-carboxylic acids and sulfonic acids. Of the above the following may be specifically named: Hydrochloric acid, sulfuric acid, formic acid, propionic acid, valeric acid, oxalic acid, malonic acid, succinic acid, cyanoacetic acid, chloroacetic acid, dichloroacetic acid, trifluoroacetic acid, benzoic acid, furane-2-carboxylic acid, p-toluenesulfonic acid, methanesulfonic acid, thioglycolic acid and citric acid, among others.

The salts according to the invention display an excellent fungicidal and fungistatic action and herein are surprisingly superior, to the best heretofore known compounds of the same direction of action. In particular, germination exchangers, such as Pythium blight, are controlled. Another advantage is their excellent plant tolerance. In addition, their good solubility in water and their fitness for the production of dry disinfectants should be stressed, as some of the salts are crystalline substances and others highly viscous oils.

They can therefore be used in agriculture or in horticulture predominantly for general soil treatment, as disinfectants for seed treatment or for furrow treatment as till-in material.

The salts according to the invention may be used alone or as mixtures with one another or with other plant protecting or pest controlling agents, e.g., with insecticides or nematocides, when the simultaneous control of these or other pests is desired, or with fungicides to enlarge the spectrum of action. Application of the invention is effected advantageously as powder, dust, granulate, solution, emulsion or suspension, among others, with addition of solid or liquid diluents or vehicles and possible of adhesion, wetting, emulsification or dispersion aids. Use in the form of their aqueous solutions is likewise possible.

Suitable liquid vehicles for use with the invention are water, mineral oils or other organic solvents, as for example, xylol, chlorobenzene, cyclohexanol, cyclo-

hexanone, dioxane, acetonitrile, acetic ester, dimethyl formamide and dimethyl sulfoxide, among others.

Solid vehicles such as lime, attapulgite and other clays, kaolin, talcum as well as natural or synthetic silica among others, are suitable for use.

Surface-active substances may be used; such as salts of ligninsulfonic acids, salts of alkylated benzenesulfonic acids, sulfonated acid amides and their salts, polyethoxylated amines and alcohols.

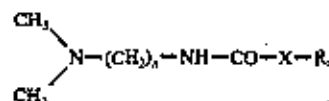
If the active substances are to be used for seed disinfection, dyes, as for example New Fuchsin among others, may be admixed, to give the disinfected seed material a clearly visible color.

The production of the various forms of preparation is carried out in a manner known in the art; such as, by grinding or mixing processes.

To promote germination of the seed, the agents are applied in a well known manner either before sowing directly onto the seed material or upon sowing into the furrow (so called tillage). For treatment of the soil in itself, the agents are advantageously introduced into the upper soil layers to a depth of about 20 cm, by the use, for example of a rotary hoe.

It is to be noted that the salts according to the invention are believed new and have not been described in the literature before. The production of the compounds herein is accomplished by methods for example as follows:

(a) a compound of the general formula



wherein

R is an aliphatic hydrocarbon radical with preferably 2 to 4 carbon atoms,

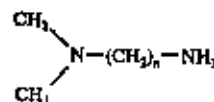
X, oxygen or sulfur and

n, an integer from 2 to 4, is neutralized with the desired acid hereinabove indicated in the usual manner or,

(b) the acid radical of any salt of the named compounds is exchanged for the desired acid radical by double conversion of two salts, or

(c) the ester radical R' of any salt of the named compounds is exchanged for the desired ester radical R by ester interchange, or

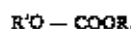
(d) a compound of the general formula



or the salt thereof with a hydrohalic acid is reacted with chloroformic esters of the general formula



or carbonic esters of the general formula



wherein the reactions described under (c) and (d) the symbols R and n have the above-mentioned meaning and R' is any hydrocarbon radical.

Reactions (a), (b) and (d) above are preferably carried out with the use of a solvent, such as water, or organic

solvents, such as ether, alcohol, hydrocarbons and hydrocarbon halides. For reaction (c) the alcohol with the desired ester radical is used in excess.

Reactions (a) and (d) may be carried out without solvent.

The reaction temperatures may be selected as desired and depend on the type of the reaction partners. They are approximately within the range of from -20° to 100° C. The isolation of the salts formed is likewise effected according to methods known such as by suction-filtering the resultant crystals formed, by decanting the supernatant solutions from the precipitated oils, or by concentrating the solutions to dryness.

The following illustrative but not restrictive examples will explain some production processes used in accordance with the desired products of the invention.

N-(3-dimethylammoniopropyl)-thiocarbamic acid-S-ethyl ester chloride

14.25 g (0.075 mole) of N-(3-dimethylaminopropyl)-thiocarbamic acid-S-ethyl ester are dissolved in 250 ml of absolute ether. While stirring and cooling, dry HCl gas is introduced. After 1 hour the product is suction-filtered. One obtains 14.1 g (83.4% of the theory) of the Mp. 87° - 93° C. After reprecipitation from isopropanol/ether one obtains a product of the Mp. 93° - 94° C.

N-(3-dimethylammoniopropyl)-carbamic acid propylester-hydrogen sulfate

11.0 g (0.0585 mole) of N-(3-dimethylaminopropyl)-carbamic acid propylester are dissolved in 200 ml of ether. While stirring and cooling, 3.2 ml (0.06 mole) of concentrated sulfuric acid are added in drops. Agitation is continued for 2 hours at room temperature, the ether is decanted, the oil is washed with ether and dried in vacuum. One obtains 15.0 g (89.7% of the theory) of the Mp. 44° - 50° C.

N-(3-dimethylammoniopropyl)-carbamic acid propylester benzoate

14.1 g (0.075 mole) of N-(dimethylaminopropyl)-carbamic acid propylester are dissolved in 200 ml of ether. While stirring, 9.15 g (0.075 mole) of benzoic acid in 30 ml of tetrahydrofuran are added in drops. Agitation is continued for 2 hours, the solvent is distilled, and the oil dried in vacuum. One obtains 23.1 g (99.4% of the theory) of $nD_{20} = 1.5070$.

N-(3-dimethylammoniopropyl)-carbamic acid ethyl ester chloride

27.2 g (0.1 mole) of N-(3-dimethylammoniopropyl)-carbamic acid ethyl ester hydrogen sulfate are dissolved in 50 ml of water and while stirring slowly admixed with a solution of 24.4 g (0.1 mole) of $BaCl_2 \cdot 2H_2O$ in 60 ml of water. Agitate for 15 minutes, admix with 100 ml of ethanol, suction filter the precipitated $BaSO_4$, treat the aqueous solution with active carbon, filter, concentrate, and drag in vacuum.

One obtains 14.7 g of oil of $nD_{20} = 1.4900$.

N-(3-dimethylammoniopropyl)-carbamic acid butyl ester chloride

21.0 g (0.1 mole) of N-(3-dimethylammoniopropyl)-carbamic acid ethyl ester chloride are suspended in 100 ml of n-butanol. Dry HCl gas is introduced into the suspension for a short time and the mixture is heated for 6 hours at 100° to 115° C while stirring, the hydrochloride

ride going into solution and some ml of liquid distilling off. Then concentrate and drag the residue in vacuum. One obtains 18.0 g of $nD_{20} = 1.4750$.

N-(3-Dimethylammoniopropyl)-carbamic acid butyl ester chloride

4.6 g (0.2 g-atom) of sodium are dissolved in 200 ml of n-butanol. To this add 21.0 g (0.1 mole) of N-(3-dimethylammoniopropyl)-carbamic acid ethyl ester chloride and boil the mixture for 6 hours with reflux. Concentrate, admix with water, ether out, dry the ether phase over $MgSO_4$, introduce dry HCl gas while stirring and cooling, decant the ether solution, and drag the remaining oil in vacuum. One obtains 13.1 g of $nD_{20} = 1.4735$.

N-(3-dimethylammoniopropyl)-thiocarbamic acid-S-ethyl ester chloride

12.25 g (0.1 mole) of chlorothioformic acid-S-ethyl ester are dissolved in 50 ml of absolute ether. While stirring, 10.2 g (0.1 mole) of N,N-dimethyl propendiamine-(1,3) in 100 ml of ether are slowly added in drops at 30° to 36° C. After 1 hour of further agitation, suction-filter. One obtains 22.0 g of the melting point 90° - 94° C.

N-(3-dimethylammoniopropyl)-carbamic acid ethyl ester chloride

20.4 g (0.2 mole) of N,N-dimethylpropanediamine-(1,3) are boiled with 80 ml of diethyl carbonate for 3 hours with reflux. Then one distills the mixture and obtains 5.2 g of N-(3-dimethylaminopropyl)-carbamic acid ethyl ester, $Bp_{16} 123^{\circ}$ C, $nD_{20} = 1.4480$. The product is dissolved in 100 ml of absolute ether, dry HCl gas is introduced, the ether solution is decanted, and the remaining oil is dragged in vacuum. One obtains 5.9 g of $nD_{20} = 1.4915$.

The other compounds herein following can be obtained in an analogous manner.

The following table shows additional salts according to the invention.

Name of Compound	Physical constant
1. N-(3-dimethylammoniopropyl)-carbamic acid ethyl ester chloride	$nD_{20} 1.4915$
2. N-(3-dimethylammoniopropyl)-carbamic acid ethyl ester benzoate	$nD_{20} 1.5080$
3. N-(3-dimethylammoniopropyl)-carbamic acid propyl ester chloride	$nD_{20} 1.4865$
4. N-(3-dimethylammoniopropyl)-carbamic acid propyl ester formate	$nD_{20} 1.4600$
5. N-(3-dimethylammoniopropyl)-carbamic acid propyl ester propionate	$nD_{20} 1.4390$
6. N-(3-dimethylammoniopropyl)-carbamic acid propyl ester valerate	$nD_{20} 1.4440$
7. N-(3-dimethylammoniopropyl)-carbamic acid propylester hydrogenoxalate	Mp. $112-115$ deg. C
8. N-(3-dimethylammoniopropyl)-carbamic acid propylester malonate	$nD_{20} 1.4675$
9. N-(3-dimethylammoniopropyl)-carbamic acid polyester succinate	$nD_{20} 1.4700$
10. N-(3-dimethylammoniopropyl)-carbamic acid propylester cyanacetate	$nD_{20} 1.4662$
11. N-(3-dimethylammoniopropyl)-carbamic acid propylester chloroacetate	$nD_{20} 1.4740$
12. N-(3-dimethylammoniopropyl)-carbamic acid propylester dichloroacetate	$nD_{20} 1.4673$
13. N-(3-dimethylammoniopropyl)-carbamic acid propylester trifluoroacetate	$nD_{20} 1.4245$
14. N-(3-dimethylammoniopropyl)-carbamic furane-2-carboxylate	$nD_{20} 1.4860$
15. N-(3-dimethylammoniopropyl)-carbamic acid propylester-p-toluenesulfonate	$nD_{20} 1.5000$
16. N-(3-dimethylammoniopropyl)-carbamic acid butylester chloride	$nD_{20} 1.4729$
17. N-(3-dimethylammoniopropyl)-thiocarbamic	