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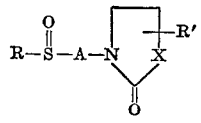
N-(ALKYLSULFINYL) ETHYL-PYRROLIDINONES AND OXAZOLIDINONES

Richard A. Hickner and Edgar E. Moore, Midland, Mich., assignors to The Dow Chemical Company, Midland, Mich., a corporation of Delaware

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8 Claims

ABSTRACT OF THE DISCLOSURE

New compounds having the structure

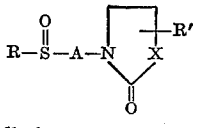


wherein R is an alkyl group of 1 to 18 carbon atoms, R′ is H or an alkyl group of from 1 to 6 carbon atoms, X is —O— or —CH_2—, and A is an alkylene group of about 2 or 3 carbon atoms.

All these compounds have antimicrobial activity. Those in which R contains 1 to 8 carbons are useful as plasticizers, while those where R contains 8 to 18 carbons are useful as nonionic surfactants. The pyrrolidinone derivatives are quite surface active, while the oxazolidinone derivatives are less surface active, but are foam boosters.

SUMMARY OF THE INVENTION

The instant invention comprises new compounds having the structure



wherein R is an alkyl group of 1 to about 18 carbon atoms, R′ is H or an alkyl group of from 1 to about 6 carbon atoms, X is —O— or —CH_2—, and A is an alkylene group of 2 or 3 carbon atoms.

These compositions are prepared by the oxidation of the corresponding thioether with an oxidizing agent. The thioethers can be prepared by the free radical addition of a thiol to the appropriate vinyl or allyl compound, or by the reaction of an N-(2- or 3-haloalkyl-2-pyrrolidinone or -axazolidinone with a thiol in the presence of an alkali metal hydroxide. Both processes are known to those skilled in the art.

The oxidizing agents that are suitable for making the compounds of this invention include peracids, such as persulfuric, perchromic, peracetic, perbenzoic and monoperphthalic acid; aqueous hydrogen peroxide; and dilute permanganates, such as potassium permanganate and sodium permanganate.

The ratio of thioether/oxidizing agent should be from a molar equivalent to about a 10% molar excess of the oxidizer. If more than 10 percent excess of the oxidizer is used, some of the thioether is oxidized to the sulfone.

The reaction should be carried out from about 0° to 100° C., and preferably from about 30° to 60° C.

It is generally desirable to have an inert diluent present to act as a heat sink. Materials such as dioxane, the lower aliphatic ketones and the lower aliphatic alcohols are suitable.

The compounds of the invention have antimicrobial activity. Those in which R contains 1 to 8 carbons, and preferably 1 to 4 carbons, are useful as plasticizers for

synthetic resins, such as polyvinyl alcohol, ethyl cellulose, and the like, while those where R contains 8 to 18 carbons are useful as nonionic surfactants. The pyrrolidinone derivatives are quite surface active, while the oxazolidinone derivatives are less surface active, but are foam boosters.

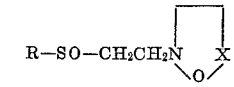
SPECIFIC EMBODIMENTS

The following are specific examples of the method of preparation and use of the new compounds disclosed herein.

Examples 1 to 6

In a typical procedure for preparing the sulfoxides, one-half mole of the thioether in 200 ml. of methanol is heated to 50–55° and one-half mole of 30% aqueous hydrogen peroxide is added during about a two-hour period. The mixture is stirred an additional hour and the solvent removed at reduced pressure. The products are white solids quite soluble in alcohol. Typical compounds so prepared are listed in Table I.

TABLE I



	R	X	M.P., deg.
1	n-C_8	CH_2	58–60
2	n-C_{10}	CH_2	72–74
3	n-C_{12}	CH_2	80–81.5
4	n-C_{14}	CH_2	86–87.5
5	n-C_{10}	O	90–92
6	n-C_{12}	O	98–100

Example 7.—Preparation of 3-(3-ethylsulfinyl)propyl-5-methyl-2-oxazolidinone

8.1 g. (0.043 mole) of 3-(3-ethylthio)propyl-5-methyl-2-oxazolidinone in 50 ml. of methanol was oxidized as in the above examples with 4.9 g. of 30% aqueous hydrogen peroxide. The resulting product was a viscous orange liquid weighing 8.3 g.

Other compounds, such as 3-[(3-pentylsulfinyl)-propyl]-5-hexyl-2-oxazolidinone; 1-[(2-octadecyl-sulfinyl)-ethyl]-4-propyl-2-pyrrolidinone; 1-[(2-t-dodecylsulfinyl)-ethyl]-2-pyrrolidinone and 1-[(2-t-octylsulfinyl)-propyl]-2-pyrrolidinone can be made by oxidizing their thioethers in a similar way.

Example 8

The product of Example 7 was used as a plasticizer for polyvinyl alcohol. 5 g. of the PVA was dissolved in 50 g. of H_2O. Eleven grams of this stock solution and 0.25 g. of the above sulfoxide were mixed to give a clear solution. 10 ml. were cast on a glass plate. After the water evaporated, a tough film remained which retained its flexibility on standing. An identical batch was also cast without the plasticizer. Upon standing for the same length of time, it became quite brittle.

Compounds such as 1-[(2-pentylsulfinyl)-ethyl]-2-pyrrolidinone, 3-[(3-heptylsulfinyl)-propyl]-4-ethyl-2-oxazolidinone and 1-[(2-butylsulfinyl)-ethyl]-2-pyrrolidinone are similar in their effects.

Various of the compounds of this invention were tested as antimicrobial agents. The chemicals tested were diluted in a suitable solvent. They were then diluted to the desired concentration in warm melted agar, poured into Petri dishes, and the agar allowed to solidify.

Droplets of the test organism were applied to the sterile agar, and the plates incubated.

The results are given in Table II. The recorded concentrations are the lowest concentrations that prevented growth of the organisms.

TABLE II

Microbiological activity of R—SO—CH_2CH_2N(X)C=O

Compound		In vitro microbiological test							
		P.p.m. to give inhibition							
R	X	*S. aur.*	*Can alb.*	*Myco phlei.*	*Ath. foot*	*Bact. subt.*	*P pull.*	*Asp. terr.*	*Rhiz. nig*
n-C_{10}	CH_2	500	500	500	500	500	500	500	-------
n-C_{12}	CH_2	100	100	500	100	500	500	500	500
n-C_{10}	O	500	500	500	500	500	500	500	50
n-C_{12}	O	500	500	500	10	100	100	100	50

Various of the compounds of this invention were tested for their surface activity.

Surface tension was determined on 0.1% aqueous solutions with a surface tensiometer. Interfacial tension was the surface tension between a Nujol mineral oil and a 0.19% aqueous solution. Foam height was determined with a Ross-Miles apparatus, while wetting time was determined on a Draves apparatus. These tests are all well known in the art.

The results are recorded on Table III:

TABLE III

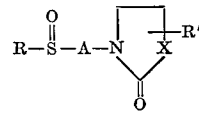
Surface activity of R—SO—CH_2CH_2N(X)C=O

R	X	Surface tension [1]	Interfacial tension [1]	Foam height, mm.		Wetting time, min.
				0 min.	5 min.	
C_8	CH_2	34.0	16.5	2.0	0.0	3.12
C_{10}	CH_2	28.9	3.2	180	14	0.2
C_{12}	CH_2	29.7	2.0	160	117	0.1
C_{14}	CH_2	32.2	6.0	15	13	1.50
C_{10}/C_{12} [2]	CH_2	29.8	3.0	185	120	0.15
C_{12}	O	33.6	9.0	0	0	>5.0

[1] Dynes/cm.
[2] 50/50.

We claim:

1. A compound having the structure



wherein R is an alkyl group of 1 to about 18 carbon atoms, R′ is H or an alkyl group of from 1 to 6 carbon atoms, X is —O— or —CH_2—, and A is an alkylene group of 2 or 3 carbon atoms.

2. The compound of claim **1** wherein R is an alkyl group of 10 to 14 carbon atoms and R′ is H.

3. The compound of claim **2** wherein X is CH_2.

4. The compound of claim **1** wherein R is an alkyl group of 1 to 4 carbon atoms.

5. The compound of claim **2** wherein X is O.

6. The compound of claim **4** wherein X is O.

7. The compound of claim **2** wherein A is ethylene.

8. The compound of claim **4** wherein A contains 3 carbon atoms and R′ is 1.

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ALEX MAZEL, Primary Examiner

R. V. RUSH, Assistant Examiner

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