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REACTIONS WITH PROPANESULTONE-(1, 3)

by

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REACTIONS WITH PROPANESULTONE-(1, 3)

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Propanesultone- (1,3) (1) reacts with p-anisidine, m-nitroaniline, m-acetamidoaniline, 2-aminopyrimidine or glycine and give the aminosulphonic acids (2 a-e), dehydration of (2 a-e) yields (3 a-e). (3e) reacts with thionylchloride to give (4), which condenses with aniline, benzylamine hydrazine hydrate and ammonia to give rise to (5 a-d), respectively.

The reactions of sultones were the subject of our previous investigation[1,2,3]. The present work deals with aminolysis of propanesultone-(1,3) (1) with primary amines to give the corresponding N-substituted aminopropane-1-sulphonic acid (2 a-d). The compounds (2 a-e) were converted to sultames (3 a-e) by treatment with boiling phosphorus oxychloride.

Propanesultone-(1,3) (1) reacted with p-anisidine m-nitroaniline, m-acetamidoaniline, 2-aminopyrimidine or glycine in boiling n-butanol or by fusion at 180°C and yielded 3 N-substituted aminopropane-1-sulphonic acids (2 a-d). The structure of (2 a-e) were proved from infrared spectra which showed absorptions characteristic of sulphonic acids in the region 600-630 cm^{-1} and the characteristic bands of SO_2 in sulphonic acid at 1150 and 1050 cm^{-1}.

Treatment of (2 a-d) with boiled phosphorus oxychloride yielded the corresponding sultames (3 a-d). The infrared spectra of (2 a-d) lack the frequency bands in the region 630-600 cm^{-1} which excluded the presence of SO_3H group, in stead it shows the characteristic sultam bands at 1270-1290 cm^{-1} (4).

When the sultam (3e) was allowed to boil with thionylchloride it yielded the acid chloride (4). Treatment of (4) with aniline, benzylamine, hydrazine hydrate or ammonia yielded the amides (5 a-d), respectively.

EXPERIMENTAL

Action of primary amines on propanesultone -(1,3) (1) *formation of* (2*a-d-t*).

A solution of (1) (0.01 mole) and primary amines namely p-anisidine, m-nitroaniline, m-acetamidoaniline and 2- aminopyrimidine (0.01 mole) in 40 ml. of n-butanol was heated under reflux for five hr. The products (2 a-d) that separated on cooling were crystallized from acetic acid. (c.f. Table 1)

Action of glycine on (1), *formation of* (2*e*)

A mixture of (1) (0.01 mole) and glycine (0.01 mole) was heated in an oil bath at 180°C for 2 hr. After cooling, the reaction mixture was washed with ethanol and the product was crystallized from acetic acid, to give (2 e) as colourless crystals, soluble in water insoluble in most of the organic solvents. m.p. 270°C (c.f. Table 1).

Table 1

Compd.	Formula	m.p.°C	Analysis		Calcd. / Found	
			C	H	N	S
2a	$C_{10}H_{15}N_4S$	261	48.98	6.12	5.71	
			49.13	6.52	5.60	
2b	$C_9H_{12}N_2O_5S$	180	41.54	4.62	10.77	12.30
			41.31	4.49	10.80	12.12
2c	$C_{11}H_{16}N_2O_4S$	185	48.53	5.88	10.29	
			48.62	5.90	10.30	
2d	$C_7H_{11}N_3S_3O$	250	38.71	5.07	19.35	
			38.62	4.84	19.28	
2e	$C_5H_{11}NO_5S$	270	30.46	5.58	7.1	16.24
			30.84	5.98	6.98	16.31

Action of phosphorus oxychloride on (2*a-e*), *formation of* (3 *a-e*).

One gram of 3N-substituted aminopropane -1- sulphonic acid (2 a-e) was added to 10 ml of $POCl_3$. The mixture was boiled under reflux for 4 hours. The reaction mixture, after cooling, was added dropwise, while stirring, to 100 ml of ice-cold water. The product which separated out was filtered off, washed with water, dried and crystallized from ethanol to give (3 a-e) (c.f. Table 2).

Table 2

Compd.	Formula	m.p.°C	Analysis		Calcd / Formula	
			C	H	N	S
3a	$C_{10}H_{15}NO_3S$	122			6.16	13.92
					6.20	13.92
3b	$C_9H_{10}N_2O_4S$	135			11.57	13.22
					11.55	13.18
3c	$C_{11}H_{14}N_2O_3S$	127			11.02	12.60
					10.99	12.82
3d	$C_7H_9N_3O_2S$	186	42.22	4.52	21.11	
			42.23	4.45	21.15	
3e	$C_5H_9NO_4S$	86	33.52	5.03	7.82	17.88
			33.42	5.10	7.72	18.01

Preparation of (5*a-c*).

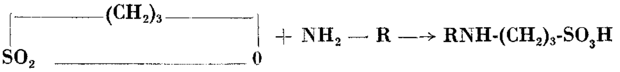
A mixture of (3e) (0.01 mole) and thionylchloride 10 ml. was heated under reflux for 2hr. The thionylchloride was evaporated in vacuum. The residue was washed with pet. ether 40-60°C to get rid of thionylchloride. A solution of (0.02 mole) aniline, benzylamine or, hydrazine hydrate in 10 ml ether was added to the residue. After one night the ether evaporated in vacuum and the residue was washed with water and crystallized from methanol. (c.f. Table 3).

Preparation of (5 d)

A mixture of (3e) (0.01 mole) and thionylchloride 10 ml was heated under reflux for 2hr. The thionylchloride was evaporated under reduced pressure. The residue, after washing with boiling pet. ether at 40-60°C, was treated with a solution of 2.5 ml 30 % NH_4OH in 10 ml ether. The reaction mass kept over night, the ether was evaporated in vacuum and the residue washed, with water, dried and crystallized from methanol to give (5 d) as collourless crystals (c.f. Table 3).

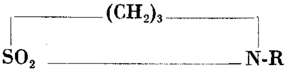
Table 3

			Analysis		Calcd / Found	
Compd.	Formula	m.p.°C	C	H	N	S
5a	$C_{11}H_{14}N_2O_3S$	170	51.97	5.51	11.02	12.60
			52.02	5.42	10.9	12.67
5b	$C_{11}H_{16}N_2O_3S$	93	53.73	5.97	10.45	
			53.82	6.01	10.51	
5c	$C_5H_{11}N_3O_3S$	118			21.76	16.58
					21.80	16.37
5d	$C_5H_{10}N_2O_3S$	105	33.71	5.62	15.73	17.98
			33.43	5.71	15.84	18.04



(1) (2)

2,3	R
a	CH_4OCH_3(p)
b	$C_6H_4NO_2$(m)
c	$C_6H_4NHCOCH_3$(m)
d	2-pyrimidyl
e	CH_2-COOH



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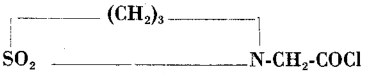

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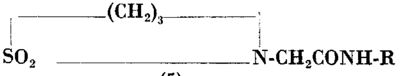

(5)

5	R
a	C_6H_5
b	CH_2-C_6H_5
c	NH_2
d	H

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