Commun. Fac. Sci. Üniv. Ank. Serie B
V. 32, pp. 55 - 61 (1986)

STUDY ON THE INFRARED SPECTRA OF SULTAMS

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(Received in June 1986, Accepted in September 1986)

ABSTRACT

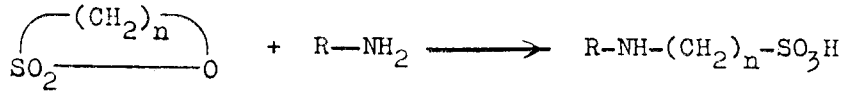
Infrared spectra show bands at 1300-1310 $cm^{-1}$and 1270-1290 cm $^{-1}$characteristic of the sultam ring, whether the sultam is six membered or five-membred ring.

INTRODUCTION

It has been suggested that the band in the rigion of 1270-1290 $cm^{-1}$is characteristic of the six-membered sultam ring, whether the ring is saturated or unsaturated (Doss, 1972). Further supporting proof for this bands assignment had also been reported depending on the transformation of the sultam ring into pyrrol ring, and consequently lacking the characteristic sultam band at 1270-1290 cm^{-1} (Doss, 1971).

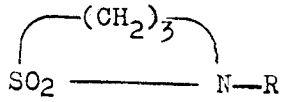
In the present work the authers would like to add other proofs supporting this observation depending on the change of N-substituted-amino-sulphonic acids into sultams. We would like also to proof that the bands in the region of 1270-1290 $cm^{-1}$is characteristic for the five-membered sultam ring. Eight model substances containing sultam ring were prepared. The sultams (3b-g) and (5) were prepared through cyclization of the N-substituted-amino sulphonic acids (2b-g) and (4) by the action of $POCl_3$. The amino sulphonic acids were prepared by reaction of the corresponding sultone (la or 1b) with amino compounds. The compounds (2a,f), (3a) and (4) were prepared according to published procedurs. (Helferich, 1960; Zeid, 1973; 1983).

ISSN 0570-1414, A. Üniv. Basımevi.



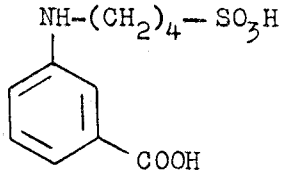
(1) a, n=3
b, n=4

(2) n=3

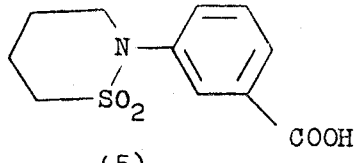


(3)

2,3	R
a	C_6H_5
b	p-ClC_6H_4
c	m-ClC_6H_4
d	p-IC_6H_4
e	m-BrC_6H_4
f	p-$CH_3C_6H_4$
g	tetradecyl



(4)



(5)

EXPERIMENTAL

3N-Substituted-amino-propane-1-sulphonic acids (2b-e,g)

A solution of equimolecular amounts of propane-1,3-sultone and the amino compound, in n-butanol, is boiled under reflux for 4 hr. The separated crystaline product after cooling was filtered off washed with methanol and recrystalized from acetic acid to give the corresponding 3N-substituted-amino-propane-1-sulphonic acids (2a–g) (cf. table 1.)

N-Substituted-sultams (3b-g), (5)

A solution of the sulphonic acid (2) or (4) (1 gm.) in $POCl_3$ (20 ml.) was boiled under reflux for 5 hr. after cooling, the oily mix-

Table 1

No.	Formula	M.p.°C	Analysis cal./ found				
			C %	H %	N %	S %	Cl %
2b	$C_9H_{12}ClNO_3S$	255	43.28 43.61	4.81 4.62	5.61 5.43	12.82 12.78	14.22 14.02
2c	$C_9H_{12}ClNO_3S$	247	43.28 43.29	4.81 4.98	5.61 5.72	12.82 12.85	14.22 14.61
2d	$C_9H_{12}INO_3S$	199	31.67 31.72	3.52 3.62	4.11 4.01	9.38 9.21	
2e	$C_9H_{12}BrNO_3S$	255	36.73 36.30	4.08 4.27	4.76 4.26		
2g	$C_{17}H_{40}NO_3S$	275	60.35 60.62	11.83 11.92	4.14 4.12	9.47 9.61	

ture was poured onto ice-coled water. The product was filtered off, washed with water, dried and crystalized from methanol to give the corresponding sultams (3a-g), (5) (cf. table 2.)

Table 2

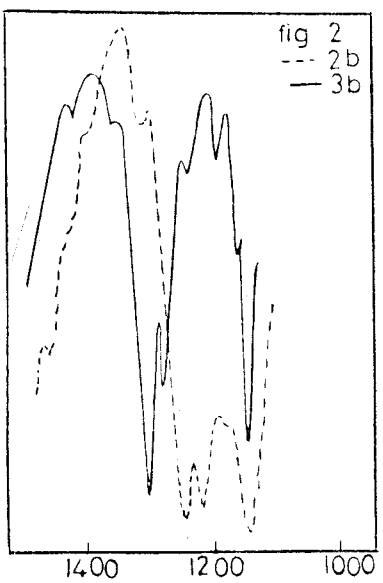
No.	Formula	M.p.°C	Analysis cal./found			
			C %	H %	N %	S %
3b	$C_9H_{10}ClNO_2S$	112	46.65 46.23	4.32 4.04	6.04 6.21	
3c	$C_9H_{10}ClNO_2S$	90	46.65 46.64	4.32 4.41	6.04 6.19	
3d	$C_9H_{10}INO_2S$	126	33.44 33.62	3.10 3.12	4.33 4.62	9.91 10.01
3e	$C_9H_{10}BrNO_2S$	75	39.13 39.19	3.62 3.65	5.07 5.62	
3f	$C_{10}H_{13}NO_2S$	94	56.87 56.42	6.16 6.16	6.64 6.72	15.17 15.20
3g	$C_{17}H_{38}NO_2S$	56	63.75 63.30	11.88 11.81	4.48 4.52	10.00 10.04
5	$C_{11}H_{13}NO_4S$	190	51.76 51.70	5.10 5.21	5.49 5.54	

DISCUSSION

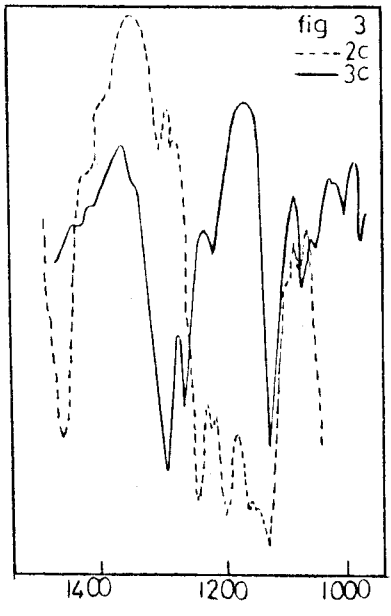
On transformation of 3N-phenylamino-propane-1-sulphonic acid (2a) into N-phenyl-propane-1,3-sultam (3a), its infrared spectrum showed a new band at 1270 cm^{-1}which is considered for the sultam ring. The week SO_2 asy. band in the spectrum of (2a) appeared as a strong band at 1300 cm^{-1} in the spectrum of (3a) (cf. Fig. 1).

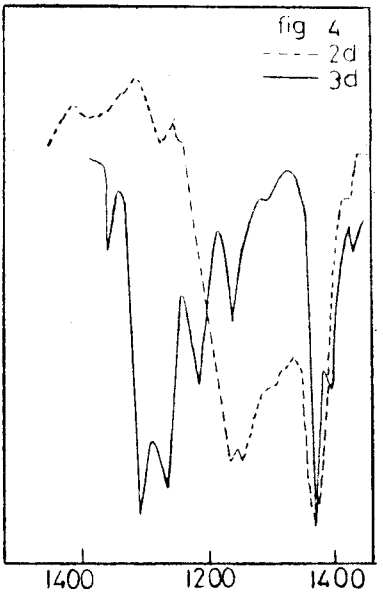
The authers extended their study by transforming 3N-(p-chloro phenyl)-propane-1-sulphonic acid (2b) into N-(p-chlorophenyl)-

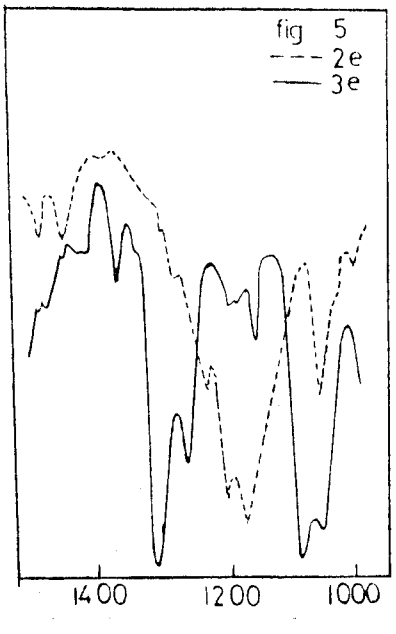
propane-1,3-sultam (3b). The infrared spectra of (3b) showed the sultam band at 1270 cm^{-1}, and similarly the week SO_2 asym. band at 1310 cm^{-1} appeared as strong band at 1300 cm^{-1} (cf. Fig. 2).

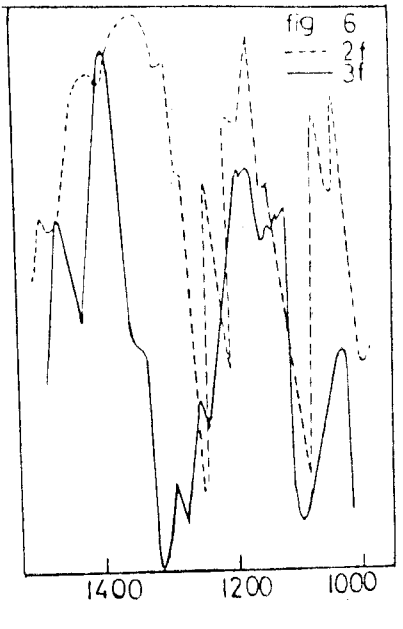


The same results were obtained in case of (3c-f) (cf. Fig. 3-6).



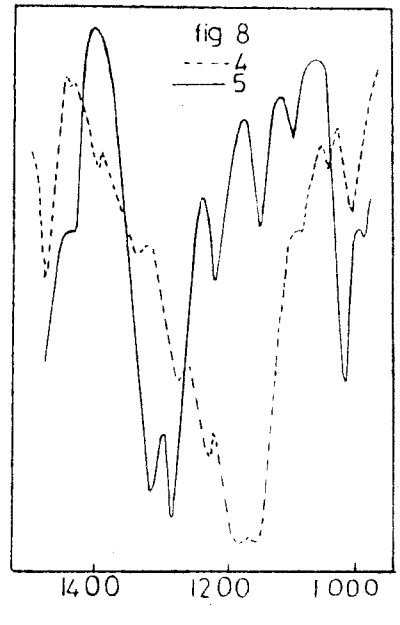






It has been also proved that these bands are also characteristic for N-alkyl-propane-1,3-sultam. On changing the sulphonic acid (2g) to the sultam (3g), the infrared spectrum showed the sultam band at 1270 cm^{-1}. The week band at 1320 cm^{-1} (SO_2 band) in the spectrum of (2g) appeared in the spectrum of (3g) at 1300 cm^{-1} (cf. Fig. 7).

Similarly the infrared spectrum of the six-membered ring sultam (5), with respect to that of the corresponding sulphonic acid (4) has been studied. Thus, it has been found that on transforming 4N-(m-carboxyphenyl)-aminobutane-l-sulphonic acid (4) into N-(m-carboxyphenyl)-butane-1,4-sultam (5), the infrared spectrum showed the sultam band at 1290 cm^{-1}. The week band at 1320 cm^{-1} appeared also as a strong band at 1310 cm^{-1}. (cf. Fig. 8)



From these evidences, it can be conluded that the strong bands at 1300-1310 cm^{-1} and 1270-1290 cm^{-1} are characteristic for the sultam ring whether the ring is six-membered or five-membered.

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