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SOME TRANSFORMATIONS OF N-(2,3-EPOXYPROPYL)-1,8-NAPHTHOSULTAM

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A method of synthesis has been developed for N-(2,3-epoxypropyl)-1,8-naphthosultam and some transformations of this compound were studied.

Keywords: 1,8-naphthosultam, 1-chloro-2,3-epoxypropane, alkylation.

Epoxypropyl derivatives of heterocyclic compounds such as 9H-carbazole and its halo derivatives, 11H-benzo[*a*]carbazole, 7H-benzo[*c*]carbazole, 1,2,3,4-tetrahydro-9H-carbazole, 9H-phenothiazine, 2-chloro-9H-phenothiazine, and indole may be obtained by the action of 1-chloro-2,3-epoxypropane (CEP) on heterocyclic compounds in the presence of alkali and dehydrating agents [1-4]. However, 1,8-naphthosultam does not react with CEP under conditions optimal for obtaining 9-(2,3-epoxypropyl)-9H-carbazole (in excess CEP in the presence of alkali in ratio 1:3, and dehydrating agents at 25-30°C). A study of this reaction showed that the action of KOH on 1,8-naphthosultam at 20-40°C leads to the formation of potassium salt of 1,8-naphthosultam, which does not react with CEP under these conditions and alkylation occurs only upon reaching 60°C. The reaction is completed in 2-2.5 h at 60°C, but only in 20-30 min at 115°C. The product yield reaches 95-98%. The following conditions were developed for obtaining N-(2,3-epoxypropyl)-1,8-naphthosultam (**1**) (see Scheme below).

The action of hydrogen chloride in methanol or hydrochloric acid in dioxane on the compound **1** leads to N-(3-chloropropyl-2-hydroxy)-1,8-naphthosultam (**2**). Then, compounds **1** and **2** were subjected to the action of sodium cyanide. Shaking a solution of compound **1** in methanol with sodium cyanide at room temperature (method A) or heating compound **2** with sodium cyanide in methanol at reflux (method B) gave N-(3-cyanopropyl-2-hydroxy)-1,8-naphthosultam (**3**). In the former case, the yield of product **3** is 85-88% though this reaction requires five days. In the latter case, the reaction time is reduced to 0.5-1.5 h but the yield is only 76.3%. Thus, the preparation of nitrile **3** from epoxy compound **1** is the most applicable.

A detailed study of the reactions of nitrile **3** showed that heating this compound with $POCl_3$ at 90°C for 6 h and chromatographic separation of the reaction products gave N-(2-chloro-3-cyanopropyl)-1,8-naphthosultam (**4**) in about 30% yield and negligible amounts of N-(3-carbamoyl-2-chloropropyl)-1,8-naphthosultam (**6**). The formation of the latter may be explained by saponification of chloronitrile **4** in the presence of hydrochloric acid to amide **6**. The yield of compound **6** is enhanced upon further heating of the reaction mixture and reaches 20% after 12 h, while the yield of chloronitrile **4** decreases to 6%. Amide **6** was also isolated upon the action of $POCl_3$ on N-(3-carbamoylpropyl-2-hydroxy)-1,8-naphthosultam (**5**), obtained by the saponification of nitrile **3** by sodium hydroxide in methanol in the presence of hydrogen peroxide and also by the saponification of chloronitrile **4** using concentrated hydrochloric acid.

Thin-layer chromatography indicated the formation of several products upon treating chloronitrile **4** with sodium hydroxide in methanol or with diethylamine in benzene. However, only one pure compound was isolated upon chromatographic separation of the reaction mixture. The structure of this compound was solved by IR,

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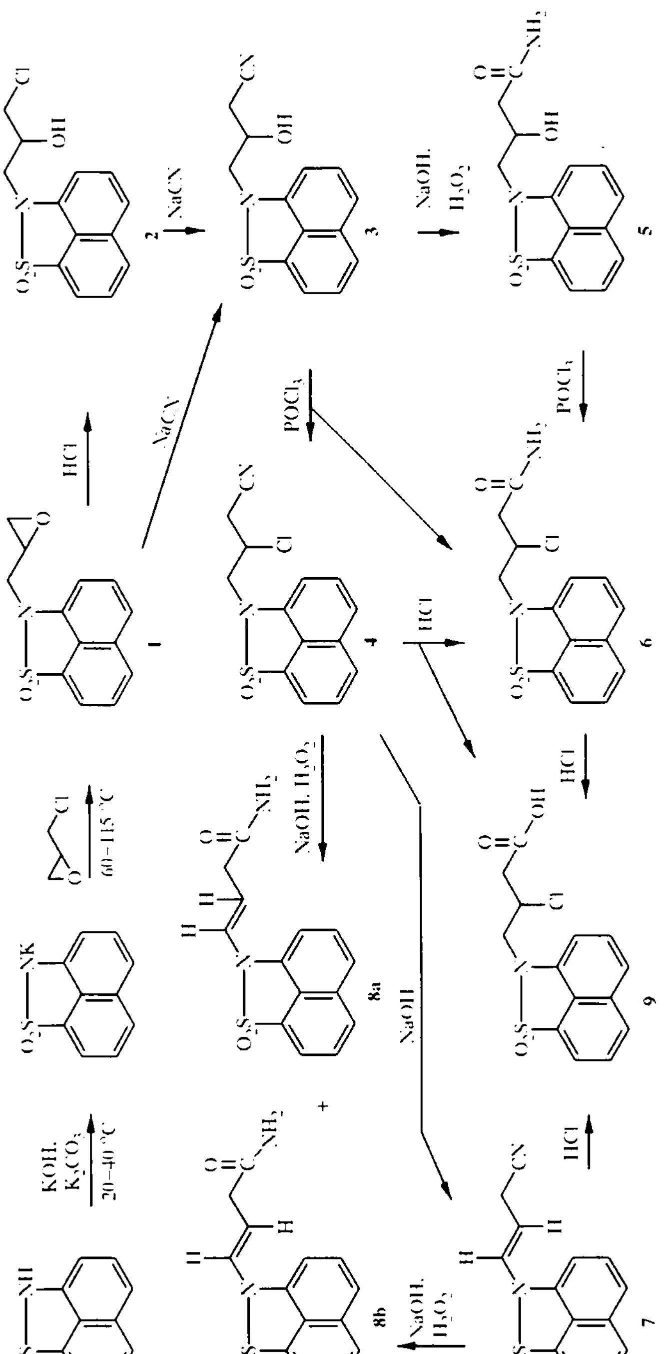
KOH, K2CO3, 20–40 °C
60–115 °C
HCl
NaCN
NaOH, H2O2
POCl3
NaOH
1
2
3
4
5
6
7
8a
8b
9

TABLE 1. The Characteristics of Compounds **1-7**, **8a,b**, and **9**

Compound	Empirical formula	Found, % / Calculated, %				mp, °C (solvent)	Yield	
		C	H	Cl	N		g	%
1	$C_{13}H_{11}NO_3S$	59.7 / 59.75	4.1 / 4.2		5.3 / 5.4	110.5-111.0 (ethanol)	25.0	95.8
2	$C_{13}H_{12}ClNO_3S$	52.5 / 52.4	4.3 / 4.1	12.0 / 11.9	4.7 / 4.7	95.0-96.0 (ethanol)	13.4	90.6
3	$C_{13}H_{12}N_2O_3S$	58.0 / 58.3	4.3 / 4.2		9.4 / 9.7	154.5-155.5 (ethanol)	10.0	86.7
4	$C_{13}H_{11}ClN_2O_2S$	54.7 / 54.8	3.8 / 3.6	11.8 / 11.6	9.2 / 9.1	144.0-145.5 (ethanol)	9.5	31.0
5	$C_{13}H_{10}N_2O_4S$	54.7 / 54.9	4.5 / 4.6		9.1 / 9.2	174.0-175.5 (ethanol)	1.1	36.0
6	$C_{13}H_{11}ClN_2O_3S$	51.8 / 51.8	3.9 / 4.0	10.9 / 10.9	8.5 / 8.6	170.5-171.0 (ethanol)	0.8	24.7
7	$C_{13}H_{10}N_2O_2S$	62.9 / 62.2	3.7 / 3.7		10.7 / 10.4	149.5-150.5 (ethanol)	2.1	77.7
8a	$C_{13}H_{12}N_2O_3S$	58.5 / 58.3	4.5 / 4.2		9.3 / 9.7	158.0-159.5 (ethanol)	1.4	24.3
8b	$C_{13}H_{12}N_2O_3S$	58.8 / 58.3	4.4 / 4.2		9.8 / 9.7	169.0-170.0 (ethanol)	2.2	38.2
9	$C_{13}H_{12}ClNO_3S$	51.2 / 51.6	3.5 / 3.7	11.2 / 10.9	4.7 / 4.3	193.5-195.0 (ethanol/wather)	0.5	30.7

^{1}H NMR, and UV spectroscopy. The analysis of the spectra showed that a small amount of a mixture of isomeric nitriles characterized by an IR absorption band for the CN group at 2240 cm^{-1}, was present in addition to the major product, *trans*-N-(3-cyano-1-propenyl)-1,8-naphthosultam (**7**). Such an absorption is observed at conjugation between the CN-group and double bond that indicates the formation of *cis*- or *trans*-N-(3-cyano-2-propenyl)-1,8-naphthosultam.

Hence, hydrogen atom from $C_{(1)}$, mainly participates in the splitting off hydrogen chloride from chloronitrile **4** and the participation of hydrogen at $C_{(3)}$ is insignificant.

Two compounds were isolated upon treating amide **6** with sodium hydroxide in methanol or with diethylamine in benzene and chromatographic separation of the reaction mixture. IR, ^{1}H NMR, and UV-spectroscopic analysis indicated that these compounds were *cis*-N-(4-carbamoyl-1-propenyl)-1,8-naphthosultam (**8a**) and *trans*-N-(4-carbamoyl-1-propenyl)-1,8-naphthosultam (**8b**). These isomers have IR spectra with absorption bands at 3440, 3200, 1695, 1679, and 1410 cm^{-1} (amide **8a**) and at 3380, 3220, 1680, 1630, and 1406 cm^{-1} (amide **8b**). Several nonplanar deformation vibration bands characteristic of the HC=CH bond are found at 715 cm^{-1} for **8a** (*cis* form) and 940 cm^{-1} for **8b** (*trans* form). The observed bathochromic shift and enhanced absorption intensity in the UV spectra of amides **8a** and **8b** relative to the spectrum of compound **6** indicate the presence of a double bond in amides **8a** and **8b**, while the bathochromic shift with maximum at 500 and 600 cm^{-1} and enhanced intensity of the absorption band in the spectrum of *trans*-isomer **8b** indicate more complete conjugation in the molecule of **8b** in comparison with that of *cis*-isomer **8a**. The ^{1}H NMR spectrum of amide **8b** shows that the coupling constant of the vinyl protons is 16 Hz, which also indicates their *trans* arrangement. The signals of the vinyl protons (N–C<u>H</u>=C<u>H</u>) in the ^{1}H NMR spectrum of *trans*-nitrile **7** and amides **8a** and **8b** are located in the region above 6.0 ppm.

Amide **8b** was obtained at the saponification of nitrile **7** using sodium hydroxide in the presence of hydrogen peroxide as well as at the saponification of chloronitrile **4** also using sodium hydroxide in the presence of hydrogen peroxide. In the latter case, hydrogen chloride is eliminated from chloronitrile **4** and then the resultant nitrile **7** is saponified to give amide **8b**.

Heating chloronitrile **4** and chloramide **6** with concentrated hydrochloric acid at reflux gave N-(3-carboxypropyl-2-chloro)-1,8-naphthosultam (**9**), which is also formed upon heating unsaturated compounds **7**, **8a**, and **8b** with hydrochloric acid at reflux. In this case, hydrogen chloride is initially added to the double bond and the resultant chloro derivatives are saponified to give acid **9**.

TABLE 2. Spectral Characteristics of Compounds **1-7**, **8a,b**, and **9**

Compound	^{1}H NMR spectrum, δ, ppm	IR spectrum, ν, cm^{-1}	UV spectrum, λ_{max}, nm (log ε) (in ethanol)
1	2.77 m (epoxy ring); 3.3 (1H, m, CH); J_{AB} = 17; J_{AX} = 3; J_{BX} = 5 Hz; ν_A = 298.07; ν_B = 330.93 Hz; 6.80-8.25 (6H, m, CH_{arom}); $CDCl_3$	3085, 3000 (=C–H); 2930, 2860 (CH_{aliph}); 1635, 1592, 1492 (C=C); 1310 sh. at 1300, 1293 (SO_2); 1173, 1145 (SO_2); 1220, 930, 895, 860 (epoxy ring); 810, 763 sh. at 750 (out-of-plane def. vibrations of C–H$_{arom}$); 607, 575 (C–S)	
2	2.8-3.2 (1H, m, OH); 3.7-3.82 (2H, d, N–CH_2–CH–CH_2); 3.95-4.1 (2H, d, N–CH_2); 4.15-4.45 (1H, m, CH_2–CH–CH_2); 6.8-8.15 (6H, m, CH_{arom}); $CDCl_3$	3480 (OH); 3070 (CH_{arom}); 2970-2880 (CH_{aliph}); 1310, 1170, 1140 (SO_2)	
3	2.5-2.65 (2H, d, N–CH_2–CH–CH_2); 3.7-3.8 (2H, d, N–CH_2); 4.2-4.45 (1H, m, CH_2–CH–CH_2); 6.5-7.8 (6H, m, CH_{arom}); $CDCl_3$	3465 (OH); 3100, 3080 (CH_{arom}); 2975-2945 (CH_{aliph}); 2270 (CN); 1310, 1170, 1140 (SO_2)	
4	3.1 (2H, d, N–CH_2–CH–CH_2, J = 6 Hz); 4.1-4.3 (2H, m, N–CH_2); 4.5-4.75 (1H, m, CH_2–CH–CH_2); 6.85-8.15 (6H, m, CH_{arom}); $CDCl_3$	3070, 3040 (CH_{arom}); 2970, 2940 (CH_{aliph}); 2270 (CN); 1310, 1175, 1145 (SO_2); 751 (Cl)	211 (4.53); 243.5 (4.28); 340 sh (3.70)
5	2.625 (2H, d, N–CH_2–CH–CH_2, J = 6 Hz); 3.8 (2H, d, N–CH_2, J = 6 Hz); 4.4-4.7 (1H, m, CH_2–CH–CH_2); 6.5-8.0 (8H, m, CH_{arom}, NH_2); CF_3COOD	3400, 3340, 3290, 3200 (OH, amide I); 3070 (CH_{arom}); 2930 (CH_{aliph}); 1675 (amide); 1430 (amide II); 1300, 1175, 1145 (SO_2)	
6	2.45-3.15 (2H, m, N–CH_2–CH–CH_2); 3.86 (2H, d, N–CH_2, J = 6 Hz); 4.25-4.65 (1H, m, CH_2–CH–CH_2); 6.52-8.2 (8H, m, CH_{arom}, NH_2); CF_3COOD	3420, 3716 (amide I); 3075 (CH_{arom}); 2930 (CH_{aliph}); 1685 (amide I); 1410 (amide II); 1300, 1170, 1145 (SO_2); 750 (Cl)	211 (4.54); 243.5 (4.28); 340 sh (3.68)
7	3.275 (2H, dd, CH_2–CN, J = 5.5 Hz); 6.0 (1H, dt, CH=CH–CH_2, J = 16 Hz); 6.5-8.1 (7H, m, CH_{arom}, N–CH=CH); $CDCl_3$	3080, 3040 (–CH=CH–); 2950, 2935 (CH_{aliph}); 2270 (CN); 1675 (–CH=CH–); 1315, 1180, 1145 (SO_2); 950 (–CH=CH-*trans*)	215 (4.62); 249 (4.39); 340 sh (3.68)
8a	3.17 (2H, d, CH_2CONH_2, J = 6 Hz); 6.0-6.5 (2H, m, CH=CH–CH_2); 6.5-8.3 (8H, m, CH_{arom}, NH_2); DMSO-d_6	3440, 3200 (amide I); 3080, 3040 (–CH=CH–); 1695, 1679, 1630 (amide I, –CH=CH–); 1310, 1170, 1135 (SO_2); 715 (–CH=CH-*cis*)	212 (4.58); 244 (4.34); 340 sh (3.71)
8b	3.1 (2H, m, CH_2CONH_2, J = 6 Hz); 6.1 (1H, dt, CH=CH–CH_2, J = 16 Hz); 6.5-8.3 (9H, m, CH_{arom}, N–CH=CH, NH_2); DMSO-d_6	3380, 3220 (amide I); 3080 (–CH=CH–); 2970-2910 (CH_{aliph}); 1680, 1630 (amide I, CH=CH-*trans*); 1406 (amide II); 1325, 1170, 1145 (SO_2); 1070, 940 (–CH=CH-*trans*)	214 (4.60); 248 (4.33); 350 sh (3.68)
9	2.5-2.8 (2H, m, N–CH_2–CH–CH_2); 3.75-3.9 (2H, m, N–CH_2); 4.45-4.75 (1H, m, CH_2–CH–CH_2); 6.5-7.85 (6H, m, CH_{arom}); CF_3COOD	3500-3300 (OH); 3070, 3035 (CH_{arom}); 2940 (CH_{aliph}); 1718, 1700 (C=O); 1300, 1175, 1140 (SO_2)	211 (4.52); 243 (4.28); 340 sh (3.70)

EXPERIMENTAL

The IR spectra were recorded on an UR-20 spectrophotometer using potassium bromide pellets. The ^{1}H NMR spectra were taken on a Tesla BS-487C spectrometer at 80 MHz using tetramethylsilane as the standard. The UV spectra were obtained on a Specord UV-VIS spectrophotometer for ethanolic solutions (c = 1.2·10^{-4} M).

The reaction course and purity of the products were monitored by thin-layer chromatography on Silufol plates. The characteristics of the synthesized products are given in Table 1. The IR, ^{1}H NMR, and UV spectral data are given in Table 2.

N-(2,3-Epoxypropyl)-1,8-naphthosultam (1). A. Mixture of 1,8-naphthosultam (20.5 g, 0.1 mol), powdered potassium hydroxide (6.2 g, 0.11 mol), and CEP (92.5 g, 1 mol) was stirred vigorously at room temperature for 2.5 h. Then, temperature was elevated to 60°C and the mixture was heated and stirred for 2 h, and filtered. The inorganic residue was washed two or three times with CEP. Excess CEP was evaporated off and the remaining crystalline precipitate was recrystallized from ethanol.

B. Mixture of 1,8-naphthosultam (20.5 g, 0.1 mol), powdered potassium hydroxide (6.2 g, 0.11 mol), and CEP (92.5 g, 1 mol) was stirred vigorously at 40°C for 1.5 h. The reaction was then carried out and product isolated as in method A. The yield of compound **1** was 25.5 g (98%).

C. Mixture of 1,8-naphthosultam (20.5 g, 0.1 mol), powdered potassium hydroxide (6.2 g, 0.11 mol), and CEP (92.5 g, 1 mol) was stirred vigorously at 90°C for 2.5 h. The temperature was then raised to 110-115°C. The reaction was completed in 30 min. The product was separated from the reaction mass by treatment with water. The yield of naphthosultam **1** was 25.1 g (96.2%).

N-(3-Chloropropyl-2-hydroxy)-1,8-naphthosultam (2). A. Sample of compound **1** (13.1 g, 0.05 mol) was dissolved in methanol and hydrogen chloride was introduced into the solution under cooling. At the end of the reaction, methanol was distilled off. The residue was treated with ethanol and cooled to 0°C. The formed crystalline precipitate was filtered off and washed with ethanol.

B. Mixture of compound **1** (2.6 g, 0.01 mol) and concentrated hydrochloric acid (d = 1.19) (3 ml) in dioxane (30 ml) was heated at reflux for 0.5 h. The product was then separated as described above to give 1.9 g (64%) of compound **2**. A mixed probe with a sample of compound **2** obtained according to method A did not give melting point depression.

N-(3-Cyanopropyl-2-hydroxy)-1,8-naphthosultam (3). A. Mixture of compound **1** (10.5 g, 0.04 mol) was dissolved in methanol (240 ml), sodium cyanide (2.16 g, 0.044 mol) was added, and the mixture was stirred vigorously at 20-25°C for 120 h. Methanol was distilled off. The residue was extracted with chloroform and then washed with water to neutral reaction. Chloroform was distilled off. The crystalline precipitate was filtered and washed with ethanol.

B. Mixture of compound **2** (3.0 g, 0.01 mol) and sodium cyanide (0.54 g, 0.011 mol) in methanol (70 ml) was heated at reflux for 1 h. Methanol was removed and the residue was treated as in the above procedure to give 2.2 g (76%) of compound **3**. A mixed probe with a sample of **3** obtained using method A did not give melting point depression.

N-(2-Chloro-3-cyanopropyl)-1,8-naphthosultam (4). Mixture of compound **3** (28.8 g, 0.1 mol) and $POCl_3$ (46.1 g, 0.3 mol) was heated at 90°C for 6 h. The reaction mixture was poured onto ice. The resultant oily residue was subjected to chromatography on a column packed with L40/100 silica gel using 1:2 hexane–ether as the eluent. The precipitate was crystallized from ethanol.

N-(3-Carbamoylpropyl-2-hydroxy)-1,8-naphthosultam (5). Mixture of compound **3** (2.9 g, 0.01 mol), methanol (60 ml), powdered NaOH (0.44 g, 0.011 mol), and 25% H_2O_2 (10 ml) was stirred vigorously at room temperature for seven days. The reaction mixture was then cooled to -3°C. The formed crystalline precipitate was filtered off and recrystallized from ethanol.

N-(3-Carbamoyl-2-chloropropyl)-1,8-naphthosultam (6). A. Mixture of compound **4** (3.1 g, 0.01 mol) and concentrated hydrochloric acid (d = 1.19) (40 ml) was heated at 80°C for 4 h. The product was separated by chromatography using 2:3 acetone–hexane as the eluent. The precipitate was crystallized from ethanol.

B. Mixture of compound **3** (14.4 g, 0.05 mol) and $POCl_3$ (23.0 g, 0.15 mol) was heated at 90°C for 12 h. The reaction mixture was poured onto ice. The product was purified by chromatography using 1:4 hexane–ether as the eluent to give 3.5 g (2%) of compound **6** and 1.0 g (6%) of compound **4**. Mixed probes of **4** and **6** with samples obtained as described above did not give melting point depression.

C. Mixture of compound **5** (3.1 g, 0.01 mol) and $POCl_3$ (7.7 g, 0.05 mol) was heated at 90°C for 5 h. The reaction mixture was cooled and poured onto ice. The product was purified by chromatography as described above to give 0.7 g (22%) of compound **6**. A probe mixed with a sample of **6** obtained by method A did not give melting point depression.

***trans*-N-(3-Cyano-1-propenyl)-1,8-naphthosultam (7).** A. Mixture of compound **4** (3.1 g, 0.01 mol) and diethylamine (1.5 g, 0.02 mol) in benzene (60 ml) was heated at reflux for 1 h. The solution was cooled and diethylamine hydrochloride was filtered off. The solvent was removed to give an oil, which was subjected to chromatography using 1:3 hexane–ether as the eluent. The product was crystallized from ethanol.

B. Mixture of compound **4** (3.1 g, 0.01 mol) and powdered NaOH (0.4 g, 0.01 mol) in methanol (50 ml) was stirred vigorously at 40°C. The product was separated as described above to give 1.7 g (63%) of compound **7**. A mixed probe with a sample of compound **7** obtained by method A did not give melting point depression.

***cis*-N-(3-Carbamoyl-1-propenyl)-1,8-naphthosultam (8a) and *trans*-N-(3-Carbamoyl-1-propenyl)-1,8-naphthosultam (8b).** A. Mixture of compound **6** (6.5 g, 0.02 mol) and diethylamine (3.0 g, 0.04 mol) in benzene (300 ml) was heated at reflux for 1.5 h. The solution was cooled and the precipitated diethylamine hydrochloride was filtered off. The solvent was removed and the oily product was subjected to chromatography with 3:2 hexane–acetone as the eluent. At first the product **8a** was isolated, then product **8b**. The products were crystallized from ethanol.

B. Mixture of compound **6** (3.3 g, 0.01 mol) and powdered NaOH (0.4 g, 0.01 mol) in methanol (70 ml) was stirred at 40°C for 30 min. Products **8a** and **8b** were isolated as described above. The yield of **8a** was 0.5 g (17%) and the yield of **8b** was 0.9 g (31%).

C. Mixture of compound **4** (9.2 g, 0.03 mol), methanol (150 ml), powdered NaOH (1.3 g, 0.033 mol), and 25% H_2O_2 (20 ml) was stirred vigorously for 10 days at room temperature. Methanol was removed and the oily product was subjected to chromatography as described above to give 2.1 g (24%) of compound **8b**.

D. Mixture of compound **7** (1,35 g, 0.005 mol), methanol (30 ml), powdered NaOH (0.22 g, 5.5 mol), and 25% H_2O_2 (3 ml) was stirred intensely at room temperature for 8 days. Methanol was distilled off. The obtained oily residue was chromatographed as described above to give 0.8 g (56%) of compound **8b**.

Samples of **8b** obtained by methods C and D, and mixed with a sample of **8b** obtained by method A did not give melting point depression.

N-(3-Carboxypropyl-2-chloro)-1,8-naphthosultam (9). A. Mixture of compound **4** (1.5 g, 0.005 mol) and concentrated hydrochloric acid (d = 1.19) (30 ml) was heated at reflux for 8 h. After cooling, the oily product was subjected to chromatography using 1:1 hexane–acetone as the eluent. The precipitate was crystallized from 4:1 ethanol–water.

B. Mixture of compound **6** (1.6 g, 0.005 mol) and concentrated hydrochloric acid (30 ml) was heated at reflux for 6.5 h to give 0.8 g (49%) of compound **9**.

C. Mixture of compound **7** (1.35 g, 0.005 mol) and concentrated hydrochloric acid (30 ml) was heated at reflux for 9 h to give 0.45 g (28%) of compound **9**.

D. Mixture of compound **8a** (0.6 g, 0.002 mol) and concentrated hydrochloric acid (15 ml) was heated at reflux for 8 h to give 0.3 g (46%) of compound **9**.

E. Mixture of compound **8b** (0.6 g, 0.002 mol) and concentrated hydrochloric acid (15 ml) was heated at reflux for 8 h to give 0.25 g (38%) of compound **9**.

The product was isolated in methods B-E as in method A. Samples of **9** obtained by methods B-E mixed with a sample obtained by method A did not give melting point depression.

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