Aluminum Chloride-Promoted Aminolysis of *N*-Tosyl Lactams

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The ring-opening reactions of lactams by nucleophilic attack provide a convenient route to ω-substituted carboxylic acid derivatives. Owing to the low reactivity of the endocyclic carbonyl, the presence of an electron-withdrawing group on the lactam nitrogen is generally necessary, and *N*-acyl or *N*-alkoxycarbonyl lactams may be cleaved by hydroxide[1-4] and alkoxide ions,[5-7] hydrazine,[8] Grignard reagents,[8-10] organolithium compounds,[9,11] and nucleophilic amines.[2,12]

In this context we have recently reported[13] that the aminolysis of lactams may be extended to weakly basic or sterically hindered amines by aluminum chloride catalysis. The products obtained were, however, dependent on the nature of the electrophile. Thus, good to excellent yields of ring-opened products were obtained with *N*-benzoyl-, *N*-pivaloyl-, or *N*-*tert*-butoxycarbonyl lactams, whereas *N*-acetyl lactams, for example, gave transacylated products.

In the search for a general method for the aminolysis of lactams we reasoned that aluminum chloride catalysis could be used to ensure reaction with amines of low nucleophilicity, and that in order to direct attack to the lactam carbonyl group, the activating electron-withdrawing group should have low electrophilicity and should complex as little as possible with the Lewis acid. An arylsulfonyl group appeared to be an interesting candidate in view of the relatively low Lewis basicity of the sulfonyl group and its steric shielding by the aromatic ring.

We here report the results of a preliminary study on the scope of the reaction of *N*-tosyl lactams with amines in the presence of aluminum chloride.

As shown in Table 1 a variety of primary and secondary amines **1** react with *N*-tosyl lactams **2** to afford the desired ring-opened products **3** in good to excellent yield. Only in the case of *N*-methyl-4-nitroaniline did reaction fail to occur (entry 8). The results obtained with the weakly basic *N*-methylaniline (entries 6, 9) and with sterically-hindered amines such as *tert*-butylamine (entries 5, 10) and diisopropylamine (entry 7) are particularly noteworthy. The nature of the lactam ring also plays a role since more vigorous conditions are required as the size of the ring increases, as demonstrated by entries 1–4. In none of the reactions examined did aminolysis occur in the absence of $AlCl_3$.

The choice of the tosyl group proved to be particularly interesting since only ring-opened products **3** were obtained, which, in addition afford the possibility of selective chemical manipulation. Note, that compared with the results observed with the Boc group,[13] better yields in ring-opened products are obtained.

Thus, as shown in Scheme 2, the amide group of **3e**, for example, is selectively reduced by Red-Al or $LiAlH_4$ to give **4**, and the sulfonyl group may be subsequently cleaved to afford the diamine **5**. Treatment of **3e** with 6 N HCl selectively hydrolyzed the amide function to afford **6**, while treatment of **3e** with 48% HBr gave the amino acid **7**.

In conclusion, the aluminum chloride-promoted aminolysis of *N*-tosyl lactams is applicable to a variety of primary and secondary amines, including those of low nucleophilicity. The reaction is regioselective and proceeds in high yield. This, in combination with selective functional-group transformations and recently described[6,9]

Scheme 1

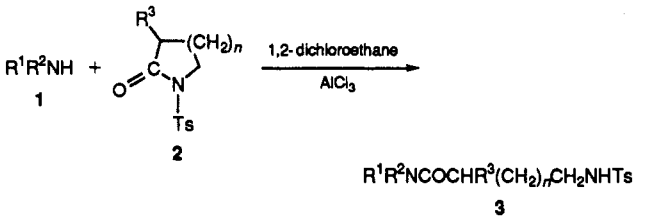
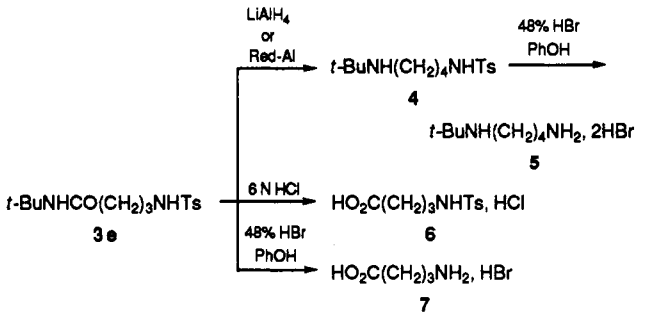


Table 1. Aluminum Chloride-Promoted Reactions of Amines 1 with Lactams 2

entry	amine 1 R¹	amine 1 R²	lactam 2 *n*	lactam 2 R³	ratio 1/2/$AlCl_3$	time, h	temp, °C	yield, % 3[a]
1	THIQ[b]		1	H	2.5/1/1.3	2.5	25	95 **3a**
2	THIQ[b]		2	H	2.5/1/1.3	1	90	89 **3b**
3	THIQ[b]		3	H	2.5/1/1.3	2.5	90	95 **3c**
4	THIQ[b]		9	H	3.5/1/2.3	18	90	83 **3d**
5	t-Bu	H	1	H	2.5/1/1.3	2.5	25	94 **3e**
6	Ph	Me	1	H	2.5/1/1.3	18	25	84 **3f**
7	i-Pr	i-Pr	1	H	2.5/1/1.3	6.5	25	65 **3g**
8	*p*-NO_2Ph	Me	1	H	3.5/1/2.3	16	90	0
9	Ph	Me	1	Me	2.5/1/1.3	18	25	64 **3h**
10	tBu	H	1	Me	2.5/1/1.3	6.5	90	85 **3i**

[a] Yield, based on lactam, of pure isolated product. [b] THIQ: 1,2,3,4-tetrahydroisoquinoline.

Scheme 2



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procedures for the regioselective substitution of lactams, should provide a useful method for the preparation of a variety of ω-amino acid and diamine derivatives.

Experimental Section

General Procedures. Melting points were determined using a Mel-Temp apparatus and are uncorrected. ^{1}H NMR spectra of $CDCl_3$ solutions were recorded at 200.13 or 250.13 MHz on Bruker AC 200 or WM 250 spectrometers. ^{13}C NMR spectra were recorded at 20.15, 50.32, and 62.89 MHz on Bruker AC 80, AC 200, or WM 250 spectrometers. All infrared spectra were obtained in 1,2-dichloroethane solution on a Perkin-Elmer Model 225 IR. C, H, N, S microanalyses were performed using a Perkin-Elmer 2400. Analytical TLC were performed with Aldrich precoated silica gel plates on polyester (0.2-mm layer) containing a fluorescent indicator. Column chromatography was performed using silica gel (70–230 mesh, SDS). 1,2-dichloroethane was distilled from P_2O_5. $AlCl_3$ was freshly sublimed before use. Commercial reagents were used without further purification.

Standard Procedure for Ring-Opening Reactions. A solution of amine 1 (10 mmol, 2.5 equiv) in 1,2-dichloroethane (10 mL) was added dropwise to a stirred suspension of $AlCl_3$ (5.2 mmol, 1.3 equiv) in the same solvent (20 mL) with ice-bath cooling. The reaction mixture was allowed to warm to room temperature and a solution of *N*-tosyl lactam **2** (4 mmol, 1 equiv) (prepared by treating the corresponding lactam with *tert*-butyllithium in THF and tosyl chloride at −78 °C) in 1,2-dichloroethane (10 mL) added over 10 min. The mixture was stirred at room temperature or 90 °C and monitored by IR and TLC. A mixture of ice and H_2O was added and the organic phase collected, washed with brine, and dried ($MgSO_4$). The products were separated by chromatography on silica gel using $CHCl_3$/MeOH as eluent.

***N*-[4-(3,4-Dihydro-1*H*-isoquinolin-2-yl)-4-oxobutyl]-4-methylbenzenesulfonamide (3a)**: pale yellow oil; IR (cm^{-1}) 3363, 1641, 1598, 1585, 1327, 1162; ^{1}H NMR ($CDCl_3$) δ 7.77 (m, 2 H), 7.28 (m, 2 H), 7.26 (m, 4 H), 5.68 (q-like, 1 H), 4.75 and 4.61 (2 s, 1.2 H and 0.8 H), 3.85 and 3.66 (2 t, J = 5.97 Hz, 0.8 H and 1.2 H), 3.06 (q, J = 6.16 Hz, 2 H), 2.91 and 2.88 (2 t, J = 5.87 Hz, 2 H), 2.5 and 2.49 (2 t, J = 6.84 Hz, 2 H), 2.43 (s, 3 H), 1.9 (q-like, 2 H); ^{13}C NMR ($CDCl_3$) δ 171.36 and 171.30 (2 s, 1 C), 142.91 (s), 136.91 (s), 134.77 and 133.94 (2 s, 1 C), 133.15 and 132.20 (2 s, 1 C), 129.43 (s), 128.64 and 128.07 (2 s, 1 C), 126.83 (s), 126.43 (s), 126.22 and 125.96 (2 s, 1 C), 46.99 and 44.12 (2 s, 1 C), 43.00 and 39.70 (2 s, 1 C), 42.85 (s), 30.72 and 30.49 (2 s, 1 C), 29.14 and 28.28 (2 s, 1 C), 24.13 (s), 21.28 (s). Anal. Calcd for $C_{20}H_{24}N_2SO_3$: C, 64.49; H, 6.49; N, 7.25; S, 8.61. Found: C, 64.38; H, 6.43; N, 7.47; S, 8.67.

***N*-[5-(3,4-Dihydro-1*H*-isoquinolin-2-yl)-5-oxopentyl]-4-methylbenzenesulfonamide (3b)**: pale yellow oil; IR (cm^{-1}) 3354, 1641, 1600, 1586, 1332, 1162; ^{1}H NMR ($CDCl_3$) δ 7.73 (m, 2 H), 7.29–7.09 (m, 6 H), 4.96 (q-like, 1 H), 4.68 and 4.57 (2 s, 1.1 H and 0.9 H), 3.78 and 3.63 (2 t, J = 5.96 Hz, 0.9 H and 1.1 H), 2.92 (q, J = 6.23 Hz, 2 H), 2.84 and 2.81 (2 t, J = 6.08 Hz, 2 H), 2.39 (s, 3 H), 2.37 and 2.36 (2 t, J = 6.93 Hz, 2 H), 1.70–1.50 (m, 4 H); ^{13}C NMR ($CDCl_3$) δ 171.28 (s), 143.02 (s), 136.85 (s), 134.86 and 133.83 (2 s, 1 C), 133.30 and 132.27 (2 s, 1 C), 129.44 (s), 128.74 and 128.08 (2 s, 1 C), 126.90 (s), 126.77 and 126.47 (2 s, 1 C), 126.44 and 126.39 (2 s, 1 C), 126.21 and 125.87 (2 s, 1 C), 47.08 and 44.07 (2 s, 1 C), 42.98 and 39.57 (2 s, 1 C), 42.50 (s), 32.53 and 32.30 (2 s, 1 C), 29.29 and 28.30 (2 s, 1 C), 28.94 (s), 24.89 (s), 21.30 (s). Anal. Calcd for $C_{21}H_{26}N_2SO_3$: C, 65.26; H, 6.78; N, 7.25; S, 8.30. Found: C, 65.34; H, 6.74; N, 7.12; S, 8.34.

***N*-[6-(3,4-Dihydro-1*H*-isoquinolin-2-yl)-6-oxohexyl]-4-methylbenzenesulfonamide (3c)**: pale yellow oil; IR (cm^{-1}) 3354, 1641, 1603, 1586, 1327, 1162; ^{1}H NMR ($CDCl_3$) δ 7.73 (m, 2 H), 7.29–7.11 (m, 6 H), 4.96 (q-like, 1 H), 4.70 and 4.58 (2 s, 1.2 H and 0.8 H), 3.80 and 3.63 (2 t, J = 5.96 Hz, 0.8 H and 1.2 H), 2.97–2.79 (m, 4 H), 2.40 (s, 3 H), 2.35 and 2.34 (2 t, J = 7.29 Hz, 2 H), 1.67–1.24 (m, 6 H); ^{13}C NMR ($CDCl_3$) δ 171.62 (s), 143.00 (s), 136.94 (s), 134.92 and 133.88 (2 s, 1 C), 133.38 and 132.40 (2 s, 1 C), 129.46 (s), 128.74 and 128.07 (2 s, 1 C), 126.86 (s), 126.74 and 126.48 (2 s, 1 C), 126.39 and 126.34 (2 s, 1 C), 126.17 and 125.88 (2 s, 1 C), 47.13 and 44.04 (2 s, 1 C), 43.02 and 39.53 (2 s, 1 C), 42.66 (s), 33.18 and 32.98 (2 s, 1 C), 29.31 and 28.33 (2 s, 1 C), 28.99 (s), 25.97 (s), 24.02 and 23.94 (2 s, 1 C), 21.31 (s). Anal. Calcd for $C_{22}H_{28}N_2SO_3$: C, 65.97; H, 7.05; N, 6.99; S, 8.01. Found: C, 66.08; H, 6.94; N, 6.94; S, 7.98.

***N*-[12-(3,4-Dihydro-1*H*-isoquinolin-2-yl)-12-oxododecyl]-4-methylbenzenesulfonamide (3d)**: yellow solid; mp 98–99 °C; IR (cm^{-1}) 3361, 1641, 1602, 1587, 1333, 1162; ^{1}H NMR ($CDCl_3$) δ 7.72 (m, 2 H), 7.26 (m, 2 H), 7.13 (m, 4 H), 5.04 (br t, J = 6.01 Hz, 1 H), 4.70 and 4.59 (2 s, 1.1 H and 0.9 H), 3.80 and 3.65 (2 t, J = 6.01 Hz, 0.9 H and 1.1 H), 2.86 (m, 4 H), 2.39 (s, 3 H), 2.37 (m, 2 H), 1.66–1.17 (m, 18 H); ^{13}C NMR ($CDCl_3$) δ 172.08 and 171.96 (2 s, 1 C), 142.95 (s), 136.93 (s), 134.97 and 133.91 (2 s, 1 C), 133.45 and 132.52 (2 s, 1 C), 129.43 (s), 128.75 and 128.10 (2 s, 1 C), 126.89 (s), 126.66 and 126.48 (2 s, 1 C), 126.34 and 126.28 (2 s, 1 C), 126.12 and 125.85 (2 s, 1 C), 47.22 and 44.01 (2 s, 1 C), 43.04 and 39.44 (2 s, 1 C), 43.03 (s), 33.65 and 33.50 (2 s, 1 C), 29.19 (s), 28.84 and 28.35 (2 s, 1 C), 26.30 (s), 25.07 and 24.97 (2 s, 1 C), 21.33 (s). Anal. Calcd for $C_{28}H_{40}N_2SO_3$: C, 69.38; H, 8.32; N, 5.78; S, 6.61. Found: C, 69.47; H, 8.24; N, 5.73; S, 6.65.

***N*-*tert*-Butyl-4-[(toluene-4-sulfonyl)amino]butyramide (3e)**: white solid: mp 90 °C; IR (cm^{-1}) 3425, 3200, 3363, 1671, 1599, 1516, 1330, 1161; ^{1}H NMR ($CDCl_3$) δ 7.71 (m, 2 H), 7.27 (m, 2 H), 5.57 (br s, 1 H), 5.48 (t, J = 6.15 Hz, 1 H), 2.93 (q, J = 6.18 Hz, 2 H), 2.40 (s, 3 H), 2.16 (t, J = 6.74 Hz, 2 H), 1.76 (m, 2 H), 1.30 (s, 9 H); ^{13}C NMR ($CDCl_3$) δ 171.65 (s), 143.08 (s), 136.76 (s), 129.49 (s), 126.83 (s), 51.12 (s), 42.48 (s), 34.15 (s), 28.51 (s), 24.89 (s), 21.30 (s). Anal. Calcd for $C_{15}H_{24}N_2SO_3$: C, 57.67; H, 7.74; N, 8.97; S, 10.26. Found: C, 57.60; H, 7.79; N, 8.93; S, 10.29.

***N*-Methyl-*N*-phenyl-4-[(toluene-4-sulfonyl)amino]-butyramide (3f)**: orange solid; mp 86 °C; IR (cm^{-1}) 3353, 1652, 1596, 1497, 1330, 1162; ^{1}H NMR ($CDCl_3$) δ 7.78 (m, 2 H), 7.49–7.18 (m, 5 H), 7.21 (m, 2 H), 5.25 (br t, J = 5.75 Hz, 1 H), 3.31 (s, 3 H), 2.97 (q, J = 6.23 Hz, 2 H), 2.48 (s, 3 H), 2.16 (t, J = 6.57 Hz, 2 H), 1.77 (m, 2 H); ^{13}C NMR ($CDCl_3$) δ 172.37 (s), 143.55 (s), 142.89 (s), 136.97 (s), 129.69 (s), 129.40 (s), 127.80 (s), 127.40 (s), 126.86 (s), 42.82 (s), 37.27 (s), 31.32 (s), 24.37 (s), 21.30 (s). Anal. Calcd for $C_{18}H_{22}N_2SO_3$: C, 62.40; H, 6.40; N, 8.09; S, 9.26. Found: C, 62.36; H, 6.41; N, 8.11; S, 9.17.

***N*,*N*-Diisopropyl-4-[(toluene-4-sulfonyl)amino]-butyramide (3g)**: orange oil; IR (cm^{-1}) 3355, 1717, 1598, 1330, 1161; ^{1}H NMR ($CDCl_3$) δ 7.76 (m, 2 H), 7.31 (m, 2 H), 4.87 (br t, J = 6.40 Hz, 1 H), 3.99–3.71 (m, 2 H), 2.95–2.71 (m, 2 H), 2.64–1.99 (m, 2 H), 2.41 (s, 3 H), 2.39 (d, J = 6.27 Hz, 12 H), 1.80–1.55 (m, 2 H); ^{13}C NMR ($CDCl_3$) δ 169.08 (s), 143.18 (s), 136.61 (s), 129.55 (s), 126.78 (s), 45.44 (s), 41.85 (s), 39.06 (s), 25.16 (s), 21.31 (s), 19.72 (s). Anal. Calcd for $C_{17}H_{28}N_2SO_3$: C, 59.97; H, 8.29; N, 8.23; S, 9.42. Found: C, 60.09; H, 8.28; N, 8.15; S, 9.38.

2,*N*-Dimethyl-*N*-phenyl-4-[(toluene-4-sulfonyl)amino]-butyramide (3h): colorless oil; IR (cm^{-1}) 3342, 1652, 1596, 1497, 1333, 1162; ^{1}H NMR ($CDCl_3$) δ 7.68 (m, 2 H), 7.44–7.15 (m, 7 H), 5.11 (br t, J = 5.83 Hz, 1 H), 3.21 (s, 3 H), 2.80 (m, 2 H), 2.42 (m, 1 H), 2.38 (s, 3 H), 1.92–1.74 and 1.44–1.28 (m, 2 H), 0.90 (d, J = 6.84, 3 H); ^{13}C NMR ($CDCl_3$) δ 175.99 (s), 143.53 (s), 142.99 (s), 136.75 (s), 129.70 (s), 129.41 (s), 127.82 (s), 127.24 (s), 126.89 (s), 41.34 (s), 37.43 (s), 34.23 (s), 32.96 (s), 21.29 (s), 17.98 (s). Anal. Calcd for $C_{19}H_{24}N_2SO_3$: C, 63.31; H, 6.71; N, 7.77; S, 8.90. Found: C, 63.56; H, 6.73; N, 7.84; S, 8.92.

***N*-*tert*-Butyl-2-methyl-4-[(toluene-4-sulfonyl)amino]-butyramide (3i)**: white solid; mp 125 °C; IR (cm^{-1}) 3424, 3353, 1674, 1599, 1513, 1327, 1162; ^{1}H NMR ($CDCl_3$) δ 7.71 (m, 2 H), 7.28 (m, 2 H), 5.67 (br s, 1 H), 5.25 (t, J = 6.36 Hz, 1 H), 2.89 (q, J = 6.41 Hz, 2 H), 2.40 (s, 3 H), 2.33–2.19 (m, 1 H), 1.86–1.61 and 1.54–1.37 (m, 2 H), 1.30 (s, 9 H), 1.04 (d, J = 6.86 Hz, 3 H); ^{13}C NMR ($CDCl_3$) δ 175.11 (s), 143.22 (s), 136.45 (s), 129.54 (s), 126.80 (s), 50.91 (s), 40.95 (s), 38.38 (s), 33.56 (s), 28.45 (s), 21.31 (s), 17.76 (s). Anal. Calcd for $C_{16}H_{26}N_2SO_3$: C, 58.87; H, 8.03; N, 8.58; S, 9.82. Found: C, 58.88; H, 8.14; N, 8.57; S, 9.80.

***N*-[4-(*tert*-Butylamino)butyl]-4-methylbenzenesulfonamide (4)**. Reduction by $LiAlH_4$ or Red-Al was carried out under the same conditions.[14] A mixture of 111 mg of $LiAlH_4$ (2.9 mmol) and 0.73 g of **3e** (2.3 mmol) in 30 mL of THF was heated at reflux for 19.5 h. The mixture was then cooled and water was added

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to neutralize the excess reducing agent. After usual workup (filtration, removal of the solvent), 100% of the *N*-tosyl diamine **4** was obtained (91% using Red-Al): pale yellow solid; mp 65–66 °C; IR (cm^{-1}) 3354, 3294, 1599, 1327, 1162; ^1H NMR ($CDCl_3$) δ 7.69 (m, 2 H), 7.23 (m, 2 H), 2.83 (t, J = 5.86 Hz, 2 H), 2.48 (t, J = 5.82 Hz, 2 H), 2.37 (s, 3 H), 1.60–1.39 (m, 4 H), 1.10 (s, 9 H); ^{13}C NMR ($CDCl_3$) δ 142.51 (s), 137.43 (s), 129.31 (s), 126.77 (s), 50.60 (s), 42.97 (s), 41.67 (s), 28.71 (s), 28.29 (s), 28.45 (s), 21.28 (s). Anal. Calcd for $C_{15}H_{26}N_2SO_2$: C, 60.37; H, 8.78; N, 9.39; S, 10.74. Found: C, 60.43; H, 8.90; N, 9.34; S, 10.65.

***N*-1-*tert*-Butylbutane-1,4-diamine, 2HBr (5).** A mixture of 0.36 g of **4** (1.2 mmol), 0.35 g of phenol (3.7 mmol), and 4 mL of 48% HBr was heated at reflux for 12 h. After the mixture was cooled, washed twice with EtOAc, and evaporated, an orange residue was obtained. This residue was washed with THF to afford the hydrobromide diamine **5** quantitatively: white solid; mp 203–204 °C; IR (cm^{-1}) 3428; ^1H NMR (D_2O) δ 2.97 (br t, J = 6.04 Hz, 2 H), 2.64 (m, 2 H), 1.92–1.47 (m, 4 H), 1.20 (s, 9 H); ^{13}C NMR (D_2O) δ 50.09 (s), 41.52 (s), 39.72 (s), 29.32 (s), 28.12 (s), 28.38 (s). Anal. Calcd for $C_8H_{22}Br_2N_2$: C, 31.39; H, 7.24; N, 9.15. Found: C, 31.44; H, 7.30; N, 9.21.

4-(*p*-Tolylamino)butyric Acid, HCl (6). An amount of 0.35 g of **3e** (1.1 mmol) was added to 14 mL of 6 N HCl and 5 mL of EtOH and heated at reflux for 48 h. The reaction mixture was concentrated quantitatively giving **6**, which was purified by column chromatography using $CHCl_3/CH_3OH$ (95/5) as eluent: white solid; mp 167–168 °C; IR (cm^{-1}) 3200, 1735, 1600, 1332, 1159; ^1H NMR (DMSO) δ 11.18 (br s, 1 H), 7.78 (m, 2 H), 7.50 (m, 2 H), 7.22 (br s, 1 H), 2.82 (q, J = 6.87 Hz, 2 H), 2.49 (s, 3 H), 2.31 (t, J = 7.29 Hz, 2 H), 1.69 (m, 2 H); ^{13}C NMR (DMSO) δ 174.08 (s), 142.63 (s), 137.69 (s), 129.73 (s), 126.60 (s), 41.97 (s), 30.73 (s), 24.61 (s), 21.06 (s). Anal. Calcd for $C_{11}H_{16}ClNSO_4$: C, 44.97; H, 5.49; N, 4.77; S, 10.92. Found: C, 44.93; H, 5.44; N, 4.84; S, 10.84.

4-Aminobutyric Acid, HBr (7). The reaction conditions are the same as above (compound **5**): orange solid; mp 215 °C; IR (cm^{-1}) (KBr) 3420, 1641; ^1H NMR (D_2O) δ 3.01 (t, J = 7.61 Hz, 2 H), 2.48 (t, J = 7.45 Hz, 2 H), 1.91 (q-like, 2 H); ^{13}C NMR (D_2O) δ 179.38 (s), 41.11 (s), 33.03 (s), 24.38 (s). Anal. Calcd for $C_4H_{10}BrNO_2$: C, 26.10; H, 5.48; N, 7.61. Found: C, 26.38; H, 5.54; N, 7.52.

Supplementary Material Available: Compound characterization data, complete with NMR peak assignments (6 pages). This material is contained in libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.