

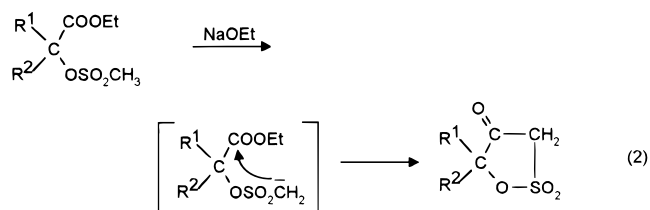
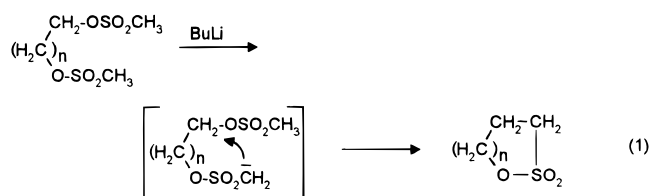
Synthesis of 4-Amino-5*H*-1,2-oxathiole 2,2-Dioxides by Cyclization of Cyanohydrin Mesylates. New Routes to β -Amino and β -Keto Sulfonic Acids

László Poszavác and Gyula Simig*

Chemical Research Division, EGIS Pharmaceuticals Ltd.,
P.O. Box 100, H-1475 Budapest, Hungary

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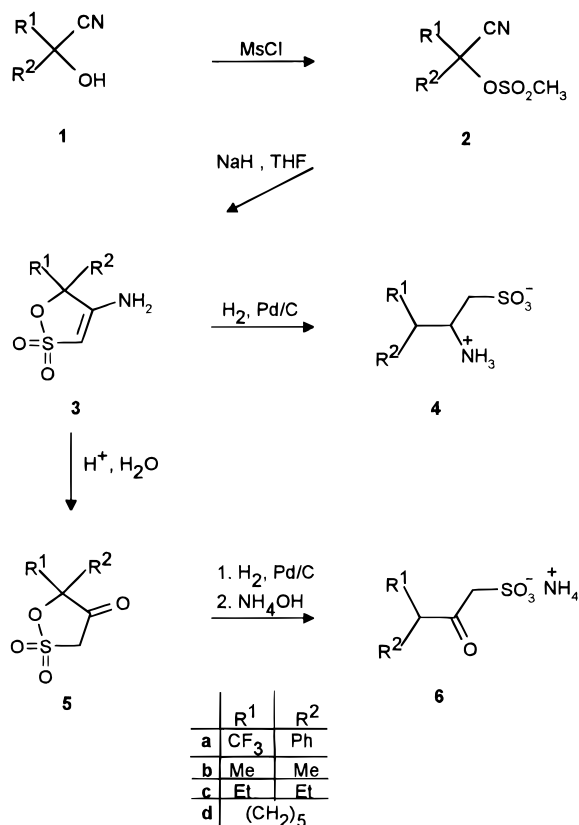
Alkanesulfonamides and alkanesulfonates can be conveniently deprotonated at the α -position and the reactions of the α -metalated species with electrophiles provide a wide variety of substituted alkanesulfonic acid derivatives.^{1–4} Intramolecular alkylation or acylation of sulfonate group-stabilized carbanions has been reported to lead to 1,2-oxathiolane 2,2-dioxides (sultones, eq 1)⁵ and the corresponding 4-ones⁶ (eq 2), respectively.



We report here the preparation of 4-amino-5*H*-1,2-oxathiole 2,2-dioxides (**3**) from cyanohydrin mesylates (**2**) based on the intramolecular addition of an α -sulfonate ester carbanion to the nitrile group. Compounds **3** are useful intermediates for the synthesis of β -amino and β -ketosulfonic acids **4** and **6** (Scheme 1).

We have recently described the anomalous reactions of α -(tosyloxy)- α -(trifluoromethyl)phenylacetone nitrile with sodium borohydride in different solvents.⁷ The formation of unexpected products (deacylation, reduction of the nitrile group) has been attributed to the presence of the trifluoromethyl group. In continuation of these studies we observed that the reaction of the corresponding mesyloxy derivative (**2a**) with sodium borohydride in *tert*-butyl alcohol afforded 1,2-oxathiole **3a**. Both ¹H NMR and ¹³C NMR data provide the chemical shift, multiplicity, and integration for the assigned structure and it was also confirmed by single crystal X-ray diffraction. The enamine structure of compound **3a** is in agreement with earlier observations demonstrating the dominance of the

Scheme 1



enamine tautomer in the presence of an unsaturated electron-withdrawing group attached to the β -carbon.⁸

The formation of compound **3a** in this reaction can be rationalized by assuming sodium borohydride promoted deprotonation of the sulfonate ester moiety at the α -position and subsequent addition to the nitrile group. The electron-withdrawing effect of the trifluoromethyl group prevents the replacement of the mesylate by the hydride anion, resulting in the unexpected reaction pathway. We reasoned, however, that the discovered cyclization reaction should not be limited to trifluoromethyl derivatives when using the weakly nucleophilic sodium hydride as the base. Indeed, treatment of cyanohydrin mesylates **2a–d** with sodium hydride in THF afforded 1,2-oxathioles **3a–d** in good yield. To the best of our knowledge, compounds **3** are the first 4-amino substituted 1,2-oxathioles, which may find interesting applications in organic synthesis.

In the current work we hydrogenated compounds **3b–d** in the presence of palladium–charcoal catalyst, producing β -amino sulfonic acids (2,2-disubstituted taurines) **4b–d** in good yields. The only known alternative general synthetic route to 2-substituted taurines applies α -amino acids as the starting compounds.^{9,10} Surprisingly, similar hydrogenation of enamine **3a** did not produce the corresponding **4a** as the single product, but a mixture of **4a** diastereomers and **6a** as indicated by the ¹H NMR spectrum.

Hydrolysis of compounds **3** furnished 1,2-oxathiolan-4-ones **5**. Synthesis of 1,2-oxathiolan-4-ones by cycliza-

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tion of (α -carboxyethyl)alkyl alkanesulfonates (e.g. eq 2) has been described in the patent literature.⁶ The results obtained in the course of hydrogenation of compounds **3** prompted us to attempt the reductive ring opening of derivatives **5**. Thus, catalytic hydrogenation of β -keto γ -sulfones **5** afforded β -keto sulfonic acids **6** (isolated as ammonium salts) in good yields. Alternative methods for the synthesis of β -keto sulfonic acids involve sulfonation of ketones¹¹ or α -halo ketones.¹²

Our results show the synthetic usefulness of 1,2-oxathioles **3a–d**. The overall transformations **1** $\rightarrow$ **4** and **1** $\rightarrow$ **6** can be regarded as the addition of methanesulfonic acid α -carbanion to a nitrile (R^1R^2CHCN) corresponding to the starting cyanohydrin **1**, followed by the reduction or hydrolysis of the resulting imine, respectively. Further work is in progress to explore the chemistry of these versatile intermediates.

Experimental Section

Melting points are uncorrected. Infrared spectra were recorded as KBr pellets. ¹H NMR spectra were recorded at 200, 250, or 400 MHz and ¹³C NMR spectra at 50.3, 62.9, or 100.6 MHz, as indicated. All unspecified reagents were from commercial resources.

Structure Determination of 3a by X-ray Crystallography. The X-ray measurements were carried out by Prof. A. Kálmán and Mr. Gy. Argay (Central Research Institute for Chemistry, Hungarian Academy of Sciences). $C_{10}H_8F_3NO_3S$; $M_r = 279.233$; crystallized from ethyl acetate as colorless crystals. The orthorhombic cell parameters and calculated cell volume are $a = 16.605$ (2) Å, $b = 9.594$ (1) Å, $c = 7.462$ (1) Å, $V = 1188.8$ (5) Å³. The acentric space group is $Pna2_1$. Intensity data were collected on an Enraf Nonius CAD4 diffractometer. The structure was solved by direct methods and refined on F_o^2 values to $R_1 = \Sigma|F_o| - |F_c|/\Sigma F_o = 0.0306$ for 2396 observations.¹³

3,3,3-Trifluoro-2-(mesyloxy)-2-phenylpropionitrile (2a). To a cold (-10°C) solution of 3,3,3-trifluoro-2-hydroxy-2-phenylpropionitrile (trifluoroacetophenone cyanohydrin, **1a**, 10.1 g, 50 mmol)¹⁴ in pyridine (50 mL) was added mesyl chloride (11.6 mL, 17.0 g, 150 mmol) dropwise. After 2 days at rt the mixture was poured into ice–water (200 mL), acidified with 9 N aqueous H_2SO_4 solution, and extracted with CH_2Cl_2 (3×150 mL). After drying ($MgSO_4$) the solvent was evaporated and the resulting crude oil was purified by distillation to give **2a** (9.1 g, 65%) as a pale yellow oil: bp 119 – 121°C (4.0 mmHg); ¹H NMR ($CDCl_3$, 250 MHz) δ 7.80–7.70 (2H, m), 7.65–7.45 (3H, m), 3.27 (3H, s); ¹³C NMR ($CDCl_3$, 62.9 MHz) δ 131.9, 129.2, 127.9, 127.0, 122.0, 120.2 (q, $J = 287.1$ Hz), 117.9, 40.5. Anal. Calcd for $C_{10}H_8F_3NO_3S$: C, 43.01; H, 2.89; N, 5.02; S, 11.48. Found: C, 43.16; H, 2.93; N, 5.07; S, 11.47.

2-Methyl-2-(mesyloxy)propionitrile (2b). (The compound is mentioned in the patent literature,¹⁵ no data available.) To a cold (-10°C) solution of 2-hydroxy-2-methylpropionitrile¹⁶ (acetone cyanohydrin, **1b**, 17.0 g, 200 mmol) and triethylamine (55.8 mL, 40.5 g, 400 mmol) in CH_2Cl_2 (100 mL) was added mesyl chloride (31.0 mL, 45.8 g, 400 mmol) over a period of 30 min. The reaction was completed by stirring for an additional 2 h at rt. The mixture was poured into ice–

water (150 mL) and extracted with CH_2Cl_2 (2×100 mL). After drying ($MgSO_4$) the solvent was evaporated and the residual was distilled to give **2b** (17.8 g, 55%) as a pale yellow oil: bp 113 – 115°C (10 mmHg); ¹H NMR ($CDCl_3$, 250 MHz) δ 3.19 (3H, s), 1.91 (6H, s); ¹³C NMR ($CDCl_3$, 62.9 MHz) δ 118.1, 74.8, 39.8, 28.0. Anal. Calcd for $C_5H_9NO_3S$: C, 36.80; H, 5.56; N, 8.58; S, 19.65. Found: C, 36.76; H, 5.54; N, 8.59; S, 19.82.

2-Ethyl-2-(mesyloxy)butyronitrile (2c). This compound was prepared analogously to **2a**, starting from 2-ethyl-2-hydroxybutyronitrile¹⁷ (**1c**, 22.6 g, 200 mmol), mesyl chloride (17.0 mL, 25.2 g, 220 mmol), and pyridine (50 mL). The reaction mixture was kept at rt for 3 days. The resulting crude oil was distilled to give **2c** (20.1 g, 53%) as a pale yellow oil: bp 119 – 121°C (2.6 mmHg); ¹H NMR ($CDCl_3$, 200 MHz) δ 3.19 (3H, s), 2.15 (4H, q, $J = 7.3$ Hz), 1.13 (6H, t, $J = 7.3$ Hz). ¹³C NMR ($CDCl_3$, 50.3 MHz) δ 116.5, 83.5, 39.4, 30.5, 7.4. Anal. Calcd for $C_7H_{13}NO_3S$: C, 43.96; H, 6.85; N, 7.32; S, 16.77. Found: C, 44.02; H, 7.03; N, 7.37; S, 16.76.

1-Cyano-1-(mesyloxy)-cyclohexane (2d). This compound was prepared analogously to **2a**, starting from 1-cyanocyclohexanol (**1d**, 25.0 g, 200 mmol),¹⁸ mesyl chloride (17.0 mL, 25.2 g, 220 mmol), and pyridine (50 mL). The reaction mixture was kept at 0 – 5°C for 3 days. The resulting crude oil was purified by distillation to give **2d** (22.9 g, 56%) as a colorless oil: bp 139 – 140°C (2.6 mmHg); ¹H NMR ($CDCl_3$, 200 MHz) δ 3.20 (3H, s), 2.45–2.25 (2H, m), 2.18–1.50 (7H, m), 1.50–1.20 (1H, m); ¹³C NMR ($CDCl_3$, 50.3 MHz) δ 117.5, 79.8, 40.2, 36.7, 23.8, 22.2. Anal. Calcd for $C_8H_{13}NO_3S$: N, 6.89; S, 15.77. Found: N, 6.86; S, 15.91.

4-Amino-5-phenyl-5-(trifluoromethyl)-5H-1,2-oxathiole 2,2-Dioxide (3a). A solution of **2a** (8.38 g, 30 mmol) in THF (20 mL) was added to a suspension of NaH (1.33 g, 59.8%, 33 mmol) in THF (30 mL) at -15°C , during 30 min. The mixture was stirred at 0°C for further 30 min. It was poured into ice–water (50 mL) and extracted with EtOAc (3×40 mL). After drying ($MgSO_4$) the solvent was evaporated and the resulting crude powder was recrystallized from 2-propanol to give **3a** (5.95 g, 71%) as a colorless solid: mp 105 – 107°C ; ¹H NMR ($CDCl_3$, 250 MHz) δ 7.70–7.60 (2H, m), 7.55–7.45 (3H, m), 5.78 (1H, s), 4.55 (2H, bs); ¹³C NMR ($CDCl_3$, 50.3 MHz) δ 151.9, 131.0, 130.8, 129.9, 129.4, 126.4, 122.2, (q, $J = 286.5$ Hz), 87.0, 86.3 (q, $J = 31.3$ MHz). Anal. Calcd for $C_{10}H_8F_3NO_3S$: C, 43.01; H, 2.89; N, 5.02; S, 11.48. Found: C, 43.15; H, 3.10; N, 4.92; S, 11.84. [The reduction of **2a** (1.12 g, 4 mmol) with $NaBH_4$ (0.31 g, 8 mmol) in *tert*-butyl alcohol at 60°C for 3 h gave **3a** in 62% yield.]

4-Amino-5,5-dimethyl-5H-1,2-oxathiole 2,2-Dioxide (3b). This compound was prepared analogously to **3a**, starting from **2b** (9.79 g, 60 mmol) in THF (20 mL) and NaH (1.98 g, 80%, 66 mmol) in THF (20 mL). The crude crystalline product was recrystallized from 2-propanol to give **3b** (8.52 g, 87%) as a colorless solid: mp 179 – 180°C ; ¹H NMR ($CDCl_3$, 250 MHz) δ 6.71 (2H, bs), 5.32 (1H, s), 1.53 (6H, s); ¹³C NMR ($DMSO-d_6$, 62.9 MHz) δ 161.1, 87.4, 84.0, 25.7. Anal. Calcd for $C_5H_8NO_3S$: C, 36.80; H, 5.56; N, 8.58; S, 19.65. Found: C, 37.00; H, 5.77; N, 8.35; S, 19.74.

4-Amino-5,5-diethyl-5H-1,2-oxathiole 2,2-Dioxide (3c). This compound was prepared analogously to **3a**, starting from **2c** (11.48 g, 60 mmol) and NaH (2.65 g, 59.8%, 66 mmol) in THF (40 mL). The resulting crude powder was purified by recrystallization from 2-propanol to give **3c** (9.51 g, 83%) as a colorless solid: mp 162 – 164°C ; ¹H NMR ($DMSO-d_6$, 200 MHz) δ 6.62 (2H, bs), 5.42 (1H, s), 2.00–1.60 (4H, m), 0.84 (6H, t, $J = 7.4$ Hz); ¹³C NMR ($DMSO-d_6$, 50.3 MHz) δ 157.26, 94.17, 87.00, 29.82, 7.28. Anal. Calcd for $C_7H_{13}NO_3S$: C, 43.96; H, 6.85; N, 7.32; S, 16.77. Found: C, 43.76; H, 6.76; N, 7.23; S, 16.53.

4-Amino-1-oxa-2-thiaspiro[4.5]dec-3-ene 2,2-Dioxide (3d). This compound was prepared analogously to **3a**, starting from **2d** (12.20 g, 60 mmol) and NaH (2.65 g, 59.8%, 66 mmol) in THF (40 mL) at rt. The resulting crude powder was purified

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by recrystallization from 2-propanol to give **3d** (8.15 g, 67%) as a colorless solid: mp 200–201 °C; ^1H NMR (DMSO- d_6 , 50.3 MHz) δ 6.67 (2H, bs), 5.29 (1H, m), 2.10–1.80 (2H, m), 1.80–1.35 (7H, m), 1.35–1.03 (1H, m); ^{13}C NMR (DMSO- d_6 , 50.3 MHz) δ 160.91, 89.25, 83.95, 33.37, 23.76, 21.52. Anal. Calcd for $\text{C}_8\text{H}_{13}\text{NO}_3\text{S}$: N, 6.89; S, 15.77. Found: N, 6.78; S, 15.57.

($\pm$)-2-Amino-3-methylbutanesulfonic Acid (4b**).^{9–10}** A solution of **3b** (1.63 g, 10 mmol) in MeOH (20 mL) was hydrogenated in the presence of 10% Pd/C (0.3 g) at rt under 2.5×10^5 Pa H_2 for 8 h. After removal of the catalyst by filtration and evaporation of the solvent, the residue was recrystallized from a mixture of 2-propanol and isopropyl ether to give **4b** (1.30 g, 78%) as a colorless compound: mp >250 °C; ^1H NMR (DMSO- d_6 , 250 MHz) δ 7.78 (3H, bs), 3.20 (1H, ddd, $J = 10.9$ Hz, $J = 2.1$ Hz, $J = 5.6$ Hz), 2.83 (1H, dd, $J = 2.1$ Hz, $J = 14.2$ Hz), 2.62 (1H, dd, $J = 10.9$ Hz, $J = 14.2$ Hz), 1.92 (1H, m), 0.91 (3H, d, $J = 6.9$ Hz), 0.88 (3H, d, $J = 6.9$ Hz); ^{13}C NMR (DMSO- d_6 , 50.3 MHz) δ 54.08, 49.42, 30.06, 18.47, 17.42. Anal. Calcd for $\text{C}_5\text{H}_{13}\text{NO}_3\text{S}$: C, 35.91; H, 7.84; N, 8.38; S, 19.17. Found: C, 35.89; H, 7.86; N, 8.28; S, 18.87.

($\pm$)-2-Amino-3-ethylpentanesulfonic Acid (4c**).** This compound was prepared analogously to **4b**, starting from **3c** (1.91 g, 10 mmol). The resulting crude powder was recrystallized from a mixture of 2-propanol and isopropyl ether to give **4c** (1.43 g, 73%) as a colorless compound: mp >250 °C; ^1H NMR (DMSO- d_6 , 200 MHz) δ 7.71 (3H, bs), 3.51–3.40 (1H, m), 2.71 (1H, dd, $J = 2.4$, 14.5 Hz), 2.58 (1H, dd, $J = 10.6$, 14.5 Hz), 1.60–0.95 (4H, m), 0.86 (3H, t, $J = 7.2$ Hz), 0.84 (3H, t, $J = 7.2$ Hz); ^{13}C NMR (DMSO- d_6 , 50.3 MHz) δ 50.47, 49.16, 43.01, 21.40, 11.47, 11.28. Anal. Calcd for $\text{C}_7\text{H}_{17}\text{NO}_3\text{S}$: C, 43.05; H, 8.77; N, 7.17; S, 16.42. Found: C, 42.92; H, 8.70; N, 7.14; S, 16.34.

($\pm$)-2-Amino-2-cyclohexylethanesulfonic Acid (4d**).** This compound was prepared analogously to **4b**, starting from **3d** (2.03 g, 10 mmol). Water (40 mL) was added to the reaction mixture in order to dissolve the solid precipitate. After removal of the catalyst by filtration and evaporation of the solvent the residue was triturated with petroleum ether (10 mL, bp 80–100 °C) to give **4d** (1.84 g, 89%) as a colorless compound: mp >250 °C; ^1H NMR (DMSO- d_6 , 400 MHz) δ 7.79 (3H, bs), 3.19 (1H, ddd, $J = 1.9$, 5.9, 11.1 Hz), 2.79 (1H, dd, $J = 1.9$, 14.3 Hz), 2.57 (1H, dd, $J = 11.1$, 14.1 Hz), 1.80–1.65 (3H, m), 1.65–1.50 (3H, m), 1.25–0.80 (5H, m); ^{13}C NMR (DMSO- d_6 , 50.3 MHz) δ 53.32, 50.83, 40.35, 28.22, 27.70, 25.79, 25.70, 25.38. Anal. Calcd for $\text{C}_8\text{H}_{17}\text{NO}_3\text{S}$: N, 6.76; S, 15.47. Found: N, 6.83; S, 15.34.

5-(Trifluoromethyl)-4-oxo-5-phenyl-1,2-oxathiolane 2,2-Dioxide (5a**).** A solution of **3a** (5.60 g, 20 mmol) in a mixture of MeOH (20 mL) and concentrated HCl (3.0 mL) was stirred for 2 h at rt. After evaporation to dryness the residue was triturated with water (20 mL) and the crystalline precipitate filtered and washed with water to give **5a** (4.01 g, 71%) as a colorless solid: mp 152–154 °C (aqueous 2-propanol); IR (KBr) 1781 cm^{-1} ; ^1H NMR (CDCl_3 , 250 MHz) δ 7.77–7.70 (2H, m), 7.60–7.40 (3H, m), 4.14 (1H, d, $J = 17.5$ Hz), 4.04 (1H, d, $J = 17.5$ Hz); ^{13}C NMR (DMSO- d_6 , 100.6 MHz) δ 162.00, 130.76, 130.60, 130.22, 129.10, 126.18, 122.08 (q, $J = 286.1$ Hz), 93.58, 85.74 (q, $J = 30.9$ MHz). Anal. Calcd for $\text{C}_{10}\text{H}_7\text{F}_3\text{O}_4\text{S}$: C, 42.86; H, 2.52; S, 11.44. Found: C, 42.83; H, 2.50; S, 11.50.

5,5-Dimethyl-4-oxo-1,2-oxathiolane 2,2-Dioxide (5b**).** This compound was prepared analogously to **5a**, starting from **3b** (9.80 g, 60 mmol), MeOH (30 mL), and concentrated HCl (5.0 mL). The solid residue was triturated with petroleum ether (25 mL, bp 80–100 °C) to give **5b** (6.50 g, 66%) as a colorless solid: mp 59–60 °C (diethyl ether); lit.⁶ mp 59 °C; IR (KBr) 1763 cm^{-1} ; ^1H NMR (CDCl_3 , 250 MHz) δ 3.94 (2H, s), 1.67 (6H, s); ^{13}C NMR (CDCl_3 , 62.9 MHz) δ 200.7, 95.4, 51.7, 24.0. Anal. Calcd for $\text{C}_5\text{H}_8\text{O}_4\text{S}$: C, 36.58; H, 4.91; S, 19.53. Found: C, 36.60; H, 5.02; S, 19.21.

5,5-Diethyl-4-oxo-1,2-oxathiolane 2,2-Dioxide (5c**).** This compound was prepared analogously to **5a**, starting from **3c** (3.83 g, 20 mmol), concentrated HCl (3.0 mL), and MeOH (20

mL) to give **5c** (2.68 g, 70%) as a yellow oil: IR (KBr) 1767 cm^{-1} ; ^1H NMR (CDCl_3 , 200 MHz) δ 3.89 (2H, s), 2.15–1.85 (4H, m), 1.03 (6H, t, $J = 7.5$ Hz); ^{13}C NMR (CDCl_3 , 100.6 MHz) δ 201.07, 102.45, 53.37, 28.30, 7.49. Anal. Calcd for $\text{C}_7\text{H}_{12}\text{O}_4\text{S}$: C, 43.74; H, 6.29; S, 16.68. Found: C, 43.30; H, 6.31; S, 16.77.

1-Oxa-4-oxo-2-thiaspiro[4.5]decane 2,2-Dioxide (5d**).** This compound was prepared analogously to **5a**, starting from **3d** (4.07 g, 20 mmol), concentrated HCl (3.0 mL), and MeOH (20 mL). The crude solid was triturated with water (20 mL) to give **5d** (3.53 g, 86%) as a colorless solid: mp 82–83 °C (aqueous 2-propanol); IR (KBr) 1763 cm^{-1} ; ^1H NMR (CDCl_3 , 200 MHz) δ 3.95 (2H, s), 2.20–2.00 (2H, m), 1.90–1.50 (7H, m), 1.50–1.20 (1H, m); ^{13}C NMR (CDCl_3 , 50.3 MHz) δ 200.57, 98.32, 52.22, 32.09, 24.03, 20.80. Anal. Calcd for $\text{C}_8\text{H}_{12}\text{O}_4\text{S}$: C, 47.05; H, 5.92; S, 15.70. Found: C, 46.82; H, 5.94; S, 15.74.

Ammonium 4,4,4-Trifluoro-2-oxo-3-phenylbutanesulfonate (6a**).** This compound was prepared by hydrogenation analogously to **4b**, starting from **5a** (2.80 g, 10 mmol). The resulting crude oil was dissolved in ether and a solution of NH_3 in EtOH was added. Crystalline ammonium salt separated which was recrystallized from a mixture of EtOH and Et_2O to give **6a** (2.71, 91%): mp 193–194 °C; IR (KBr) 1718 cm^{-1} ; ^1H NMR (D_2O , 400 MHz) δ 7.60–7.50 (3H, m), 7.50–7.40 (2H, m), 5.23 (1H, q, $J = 8.5$ Hz), 4.15 (1H, d, $J = 14$ Hz), 3.88 (1H, d, $J = 14$ Hz); ^{13}C NMR (D_2O , 50.3 MHz) δ 192.7, 127.7, 127.3, 127.0, 125.0, 121.4 (q, $J = 279.7$ Hz), 58.2, 57.4 (q, $J = 25.6$ Hz). Anal. Calcd for $\text{C}_{10}\text{H}_{12}\text{F}_3\text{NO}_4\text{S}$: N, 4.68; S, 10.71. Found: N, 4.76; S, 10.83.

Ammonium 3-Methyl-2-oxo-butanefulfonate (6b**).** This compound was prepared by hydrogenation analogously to **4b**, starting from **5b** (1.64 g, 10 mmol). The resulting ammonium salt was recrystallized from a mixture of 2-propanol and isopropyl ether to give **6b** (1.75, 90%): mp >130 °C (slow decomposition). IR (KBr) 1701 cm^{-1} ; ^1H NMR (D_2O , 400 MHz) δ 4.15 (2H, s), 3.05–2.90 (1H, m), 1.11 (3H, d, $J = 7.0$ Hz), 1.09 (3H, d, $J = 7.0$ Hz); ^{13}C NMR (D_2O , 50.3 MHz) δ 211.71, 61.93, 42.98, 18.89. Anal. Calcd for $\text{C}_5\text{H}_{13}\text{NO}_4\text{S}$: C, 32.78; H, 7.15; N, 7.64; S, 17.50. Found: C, 32.44; H, 7.06; N, 7.52; S, 17.61.

Ammonium 3-Ethyl-2-oxo-pentanesulfonate (6c**).** This compound was prepared by hydrogenation analogously to **4b**, starting from **5c** (1.91 g, 10 mmol). The resulting ammonium salt was purified by trituration with isopropyl ether (15 mL) to give **6c** (1.74, 83%): mp >100 °C (slow decomposition). IR (KBr) 1702 cm^{-1} ; ^1H NMR (DMSO- d_6 , 400 MHz) δ 7.08 (4H, bs), 3.58 (2H, s), 3.10–2.90 (1H, m), 1.60–1.45 (2H, m), 1.42–1.30 (2H, m), 0.76 (6H, t, $J = 7.4$ Hz); ^{13}C NMR (DMSO- d_6 , 50.3 MHz) δ 206.49, 64.28, 51.79, 22.58, 11.34. Anal. Calcd for $\text{C}_7\text{H}_{17}\text{NO}_4\text{S}$: C, 37.79; H, 8.11; N, 6.63; S, 15.18. Found: C, 38.06; H, 8.22; N, 6.57; S, 15.38.

Ammonium 2-Cyclohexyl-2-oxoethanesulfonate (6d**).** This compound was prepared by hydrogenation analogously to **4b**, starting from **5d** (2.04 g, 10 mmol). The resulting ammonium salt was crystallized from a mixture of 2-propanol and isopropyl ether to give **6d** (1.68, 75%): mp 182–183 °C; IR (KBr) 1698 cm^{-1} ; ^1H NMR (DMSO- d_6 , 400 MHz) δ 7.09 (4H, t, $J = 51.2$ Hz, $^+\text{NH}_4$), 3.61 (2H, s), 3.00–2.85 (1H, m), 1.90–1.75 (2H, m), 1.75–1.55 (3H, m), 1.30–1.05 (5H, m); ^{13}C NMR (DMSO- d_6 , 100.6 MHz) δ 206.13, 63.15, 48.18, 28.12, 25.76, 25.36. Anal. Calcd for $\text{C}_8\text{H}_{17}\text{NO}_4\text{S}$: C, 43.03; H, 7.67; N, 6.27; S, 14.36. Found: C, 42.72; H, 7.62; N, 6.16; S, 14.54.

Supporting Information Available: ^1H NMR and ^{13}C NMR spectra for compounds **2**, **3**, **4b–d**, **5**, and **6** (39 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.