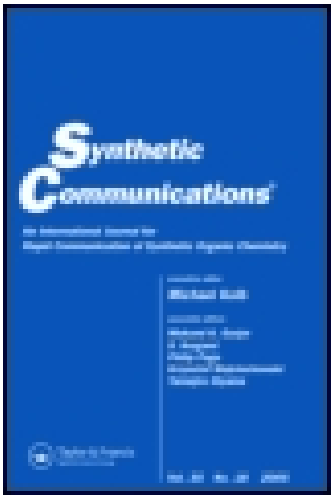

Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/lsyc20

Synthesis of 7-Sulfamoyl-substituted 2-Oxo-2,3,4,5-tetrahydro-1H-benzo[b]azepines

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Published online: 24 Nov 2006.

To cite this article: Mikhail V. Dorogov, Sergey A. Ivanovsky, Maria Y. Khakhina, Dmitry V. Kravchenko, Sergey E. Tkachenko & Alexandre V. Ivachtchenko (2006) Synthesis of 7-Sulfamoyl-substituted 2-Oxo-2,3,4,5-tetrahydro-1H-benzo[b]azepines, Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry, 36:23, 3525-3535, DOI: 10.1080/00397910600943493

To link to this article: http://dx.doi.org/10.1080/00397910600943493

Synthetic Communications®, 36: 3525–3535, 2006

ISSN 0039-7911 print/1532-2432 online
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Synthesis of 7-Sulfamoyl-substituted 2-Oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepines

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Abstract: Sulfochlorination of 2-oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepine led to regioselective formation of the corresponding 7-chlorosulfonyl derivative. Starting from this reagent, a large number of substituted 2-oxo-7-sulfamoyl-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepines were obtained. This approach is amenable to combinatorial production of the title compounds, which possess promising therapeutic potential.

Keywords: Benzoazepines, combinatorial synthesis, regioselective reaction, sulfonamides

The 2-oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepine heterocycle is a core scaffold of many physiologically active compounds, including protease inhibitors,[1,2] calcium-channel blockers,[3] N-methyl-D-aspartate (NMDA) glycine-site antagonists,[4] integrin antagonists,[5] and growth hormone secretagogues,[6] to name just a few. Beilstein database[7] cites more than 2,600 compounds, including 650 physiologically active ones, which contain this fragment. According to these numerous examples, the 2-oxo-2,3,4,5-

Received in the USA April 20, 2006
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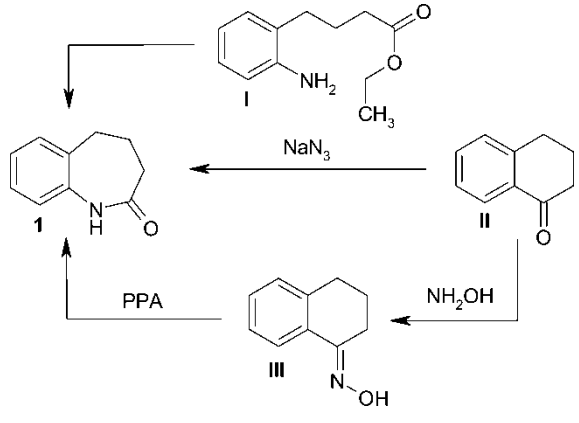
tetrahydro-1*H*-benzo[*b*]azepine heterocycle can be considered as a privileged one. The term *privileged structure* describes structural chemotypes (originally, benzodiazepine-2-ones[8]) that bind to multiple unrelated classes of protein receptors as high affinity ligands.[9,10] Privileged substructures are typically rigid, polycyclic, or heterocyclic systems capable of orienting various substituents in a well-defined three-dimensional space.

To the best of our knowledge, there have been no 7-sulfonyl derivatives of 2-oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepine heterocycle reported to date. This situation prompted us to explore synthetic routes to 2-oxo-7-sulfamoyl-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepines, which can serve as a promising source of bioactive molecules.

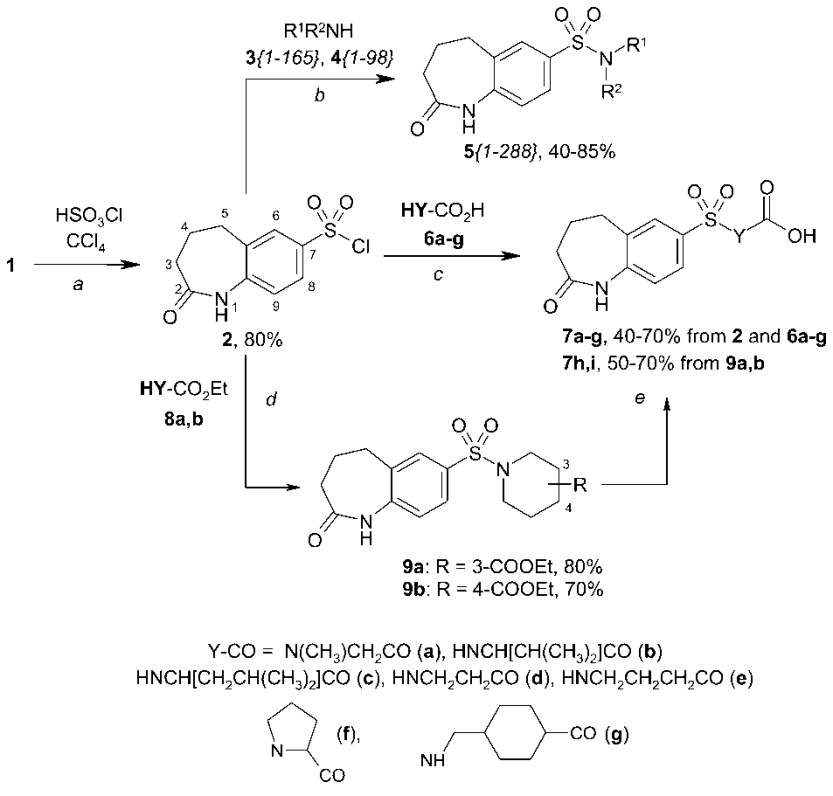
Synthetic routes to 2-oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepine **1**, which was used in this work as an initial reagent, are well documented in literature. For example, compound **1** can be prepared by self-condensation of ethyl 4-(2-aminophenyl)butanoate **I**[11] or by interaction of 1-tetralone (3,4-dihydro-1(2*H*)-naphtalenone) **II** with sodium azide.[12,13] In our opinion, the reaction of 1-tetralone **II** with hydroxylamine followed by the Beckman rearrangement of the resulting oxime **III** is the most suitable method for the preparation of **1** (Scheme 1).[14] This method, which was used in this work, provides optimal yield and is experimentally convenient.

2-Oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepine-7-sulfonyl chloride **2** was prepared in 80% yield by treatment of **1** (1 equiv.) with a mixture containing 5 equiv. of chlorosulfonic acid and the same volume of CCl_4. The reaction was regioselective and led to one major 7-substituted product, as was evidenced from ^{1}H-^{1}H nuclear Overhauser effect spectroscopy (NOESY) 2-D correlation spectra.

A combinatorial library of 265 substituted 2-oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepine-7-sulfonamides **5**{*1–288*} was further synthesized by treatment of chloride **2** with various amines **3** and **4** in the presence of



Scheme 1. Synthesis of 2-oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepine.



Scheme 2. Synthesis of sulfonamide derivatives of 2-oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepine: (*a*) 70°C, 3 h, 80%; (*b*) dioxane, TEA, 60°C, 3 h, 40–85%; (*c*) NaOH, H_2O, 50°C, 1 h, 50–85%; (*d*) 1,4-dioxane, TEA, 60°C, 3 h, 40–85%; and (e) NaOH, H_2O, 100°C, 15 min, 60–85%.

triethylamine (TEA) (1:1.1:1.5) in 1,4-dioxane (Scheme 2). In these parallel reactions, we used 190 primary amines **3**{*1–165*} and 98 secondary amines **4**{*1–98*}, including alkylamines, cycloalkylamines, substituted anilines, benzyl- and phenethylamines, pyrrolidines, piperidines, piperazines, morpholines, and heteroarylamines. Structures of some representative amines are given in Fig. 1.

The structural diversity of the sulfonyl amide derivatives of **1** can be increased through application of various amino acids or their ester derivatives as amine components. Reaction of chloride **2** with amino acids **6a–g** in an aqueous solution of NaOH at 50°C afforded the corresponding carboxylic acids **7a–g** in 40–70% yield. Alternatively, acids **7** can be obtained via the corresponding alkyl carboxylate intermediates in those cases when the corresponding amino esters are more commercially accessible than the free acids. For example, the reaction of chloride **2** with ethyl nipecotate **8a** or ethyl isonipecotate **8b** followed by alkali hydrolysis of the obtained esters **9a,b** led to acids **7h,i**.

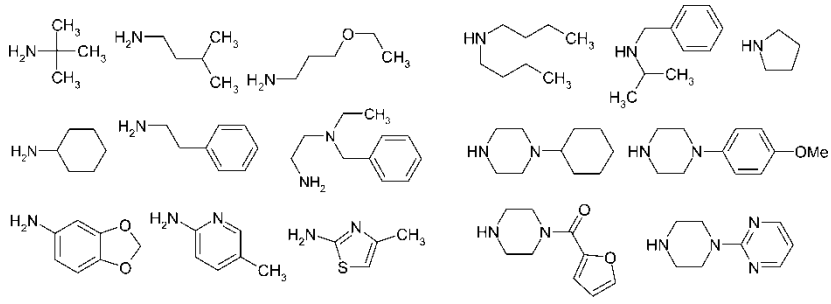
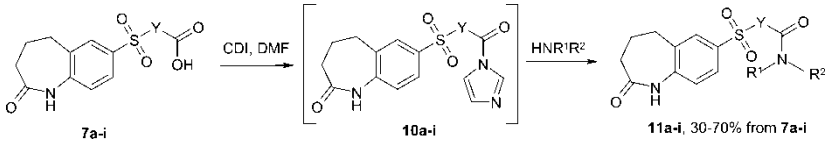


Figure 1. Representative primary **3**{*1–165*} and secondary **4**{*1–98*} amines.

The resulting acids **7a–i** were used as building blocks for parallel synthesis of a small series of carboxamides **11a–i**. The latters were synthesized using 1,1′-carbonyldiimidazole (CDI)-promoted coupling of acids **7a–i** with several arbitrary amines from the corresponding sets **3**{*1–165*} and **4**{*1–98*} (Scheme 3). Reaction of acids **7a–i** with CDI (0.9 equiv.) readily proceeded in dry DMF at 60°C. The resulting imidazolide intermediates **10a–i** were then treated with the corresponding amines to obtain the final products **11a–i** in 30–70% yield (from **7a–i**). In most cases, the desired products precipitated from the reaction mixtures upon addition of water. Purity of the synthesized compounds collected as precipitates was 95% or higher. Given the simple synthesis and purification procedures, the developed approach to carboxamides **11a–i** can be readily applied in a high-throughput combinatorial format. The analytical samples were obtained by recrystallization from ethanol. Structures and yields of the synthesized compounds **11a–i** are given in Table 1.

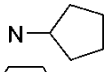
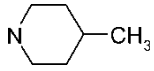
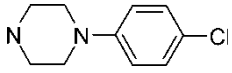
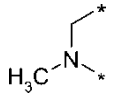
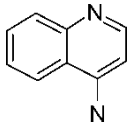
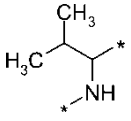
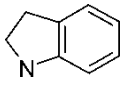
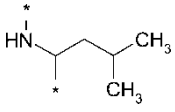
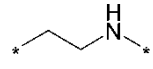
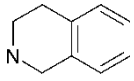
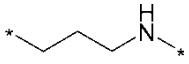
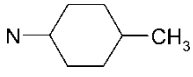
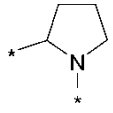
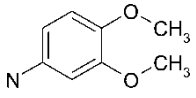
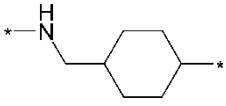
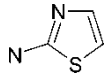
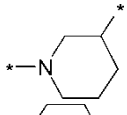
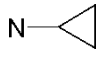
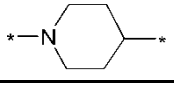
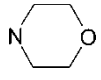
All the synthesized compounds within the combinatorial series **5**{*1–288*}, **7a–i**, and **11a–i** were characterized by ^{1}H NMR, liquid chromatography mass-spectrometry; CDI, 1,1′-carbonyldiimidazole (LCMS), and elemental analysis. The analytical data were in agreement with the suggested structures.

The regioselective character of sulfochlorination of compound **1** has been proven by ^{1}H NMR studies. Thus, ^{1}H NMR spectra of all the synthesized sulfonamides contain three signals corresponding to *abx*-system: two doublets with $J_o = 8.3$ Hz and $J_m = 1.8$ Hz and a doublet of doublets with similar spin–spin coupling constants. This picture corresponds to location of the sulfonyl group in positions 7 or 8. In ^{1}H-^{1}H NOESY spectra of a sample



Scheme 3. Synthesis of carboxamides **11a–i**.

Table 1. Structures and yields of compounds **11a–i**

Compound	Y	NR^1R^2	Yield (%)
5{*1*}	—		75
5{*2*}	—		80
5{*3*}	—		67
11a			52
11b			45
11c			60
11d			67
11e			53
11f			65
11g			54
11h			50
11i			70

compound **5**{*2*} (Fig. 2), we observed a cross-pick, which characterizes an interaction of the H^1 proton of NH group with the H^9 proton of benzene ring. The 1-D NMR signal of this proton is a doublet ($J_o = 8.3$ Hz) of the *abx*-system. Such a picture would be impossible if the sulfonyl group was in position 8. In this case, the signal would be a singlet or a doublet with a

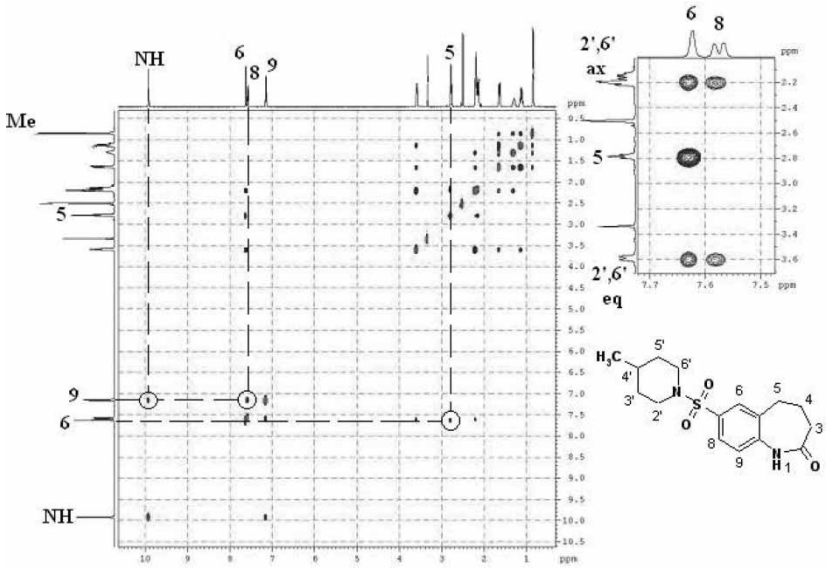


Figure 2. 7-(4-Methyl-piperidine-1-sulfonyl)-1,3,4,5-tetrahydro-benzo[*b*]azepin-2-one **5**{*2*} ^{1}H-^{1}H NOESY spectra.

small coupling constant corresponding to *meta*-interaction H^9-H^7. At the same time, we observed a cross-pick characterizing the *ortho*-interaction H^9-H^8. The actual spectrum also contains a couple of doublet signals ($J_m = 1.8$ Hz), which correspond to *meta*-interacting protons H^6-H^8. One of them gives a cross-pick with a signal of the H^5-proton (2.8 ppm). The observed signals unambiguously prove the location of the sulfonyl group in position 7 of the 2-oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepine heterocycle.

In summary, an efficient way to combinatorial libraries of novel 7-sulfamoyl derivatives of 2-oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepine was developed. The desired compounds were obtained in good to excellent yields with a high level of purity using simple purification procedures. The synthesized compounds have great potential bioactivity and are valuable objects for biological screening.

Structures and ^{1}H NMR spectra of synthesized compounds are available from the authors.

EXPERIMENTAL

General Information

Melting points (°C) were measured with Koeffler melting-point apparatus and are uncorrected. Thin-layer chromatography (TLC) was performed on

aluminum sheets precoated with silica gel (Merck, Kieselgel 60 F-254). ^{1}H NMR spectra were recorded on Bruker AMX-400 using TMS as an internal standard (chemical shifts in parts per million, ppm). LCMS spectra were recorded with PE SCIEX API 150EX liquid chromatograph equipped with a UV detector (λ_{max} 215 and 254 nm) and using a C_{18} column (100 × 4 mm). Elution started with water, ended with acetonitrile/water (95:5, v/v), and used a linear gradient at a flow rate of 0.15 mL/min and an analysis cycle time of 25 min. All solvents and reagents were obtained from commercial sources and used without purification. The parallel solution-phase syntheses of compounds **5**{*1–288*} were accomplished using a laboratory synthesizer CombiSyn-012-3000 on the 50- to 100-mg scale.

2-Oxo-2,3,4,5-tetrahydro-1 *H*-benzo[*b*]azepine-7-sulfonyl chloride (2): 1,3,4,5-Tetrahydro-benzo[*b*]azepin-2-one **1** (0.5 mol) was slowly added to a mixture of chlorosulfonic acid (165 mL, 2.5 mol) and tetrachloromethane (165 mL) at 0°C. The mixture was stirred at 70°C for 3 h, then cooled down, and poured onto crushed ice. The formed precipitate of 2-oxo-2,3,4,5-tetrahydro-1 *H*-benzo[*b*]azepine-7-sulfonyl chloride **2** was collected by filtration, washed with cold water, and purified by crystallization from toluene to give pure **2** in 80% yield, mp 200–202°C (toluene).

General Procedure for Synthesis of 2-Oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepine-7-sulfonic Acid Amides (5{*1–288*})

Sulfonyl chloride **2** (1.0 mmol) was added to a solution of amine **3** or **4** (1.1 mmol) and TEA (1.5 mmol) in dioxane (3 mL) at room temperature. The mixture was stirred for 3 h at 60°C, then cooled down to room temperature, and mixed with water (15 mL). The formed precipitate was collected by filtration, washed with water, and recrystallized from 2-propanol to give pure **5** in 40–85% yields.

Analytical Data for Representative Compounds

2-Oxo-2,3,4,5-tetrahydro-1 *H*-benzo[*b*]azepine-7-sulfonic acid cyclopentylamide 5{*1*}: yield 75%, mp 211–214°C; LCMS m/z 309 (M + 1); ^{1}H NMR (500 MHz): δ 1.4 (m, 4H, $^{3',4'}CH_2$), 1.6 (m, 4H, $^{2',5'}CH_2$), 2.20 (m, 4H, $^{3,4}CH_2$), 2.75 (t, 2H, $^5CH_2$), 3.35 (m, 1H, $^{1'}CH$), 7.05 (d, 1H, $J_{9-8} = 8.3$ Hz, ^9CH), 7.3 (d, 1H, SO_2NH), 7.55 (d, 1H, $J_{6-8} = 1.8$ Hz, ^6CH), 7.60 (dd, 1H, $J_{8-9} = 8.3$ Hz, $J_{8-6} = 1.8$ Hz, ^8CH), 9.75 [s, 1H, C(O)NH]. Anal. calcd. for $C_{27}H_{28}F_3N_3O_2S$: C (58.42%), H (6.54%), N (9.08%). Found: C (58.38%), H (6.58%), N (8.89%).

7-(4-Methyl-piperidine-1-sulfonyl)-1,3,4,5-tetrahydro-benzo[*b*]azepin-2-one 5{*2*}: yield 80%, mp 229–232°C; LCMS m/z 323 (M + 1); ^{1}H NMR

(500 MHz): δ 0.83 (d, 2H, CCH_3), 1.14 (m, 2Hax, $^{3',5'}CH_2$), 1.3 (m, 1H, ^{4}CH), 1.64 (m, 2Heq, $^{3',5'}CH_2$), 2.15 (m, 2Hax, $2NCH_2$), 2.20 (m, 4H, $^{3,4}CH_2$), 2.79 (t, 2H, $^{5}CH_2$), 3.6 (m, 2Heq, $2NCH_2$), 7.15 (d, 1H, $J_{9-8} = 8.3$ Hz, ^{9}CH), 7.52 (dd, 1H, $J_{8-9} = 8.3$ Hz, $J_{8-6} = 1.8$ Hz, ^{8}CH), 7.62 (d, 1H, $J_{6-8} = 1.8$ Hz, ^{6}CH), 9.93 [s, 1H, C(O)NH]. Anal. calcd. for $C_{16}H_{22}N_2O_3S$: C (59.60%), H (6.88%), N (8.69%). Found: C (59.11%), H (6.96%), N (8.61%).

General Procedure for Synthesis of 4-(2-Oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepine-7-sulfonylamino)-carboxylic Acids (7a–g)

2-Oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepine-7-sulfonyl chloride **2** (0.1 mol) was added to a solution of NaOH (0.22 mol) and amino acid **6a–g** (0.11 mol) in water (150 mL) at 30°C. The mixture was stirred at 60°C for 1 h, then cooled down to room temperature, and neutralized with a 10% solution of hydrochloric acid to maintain pH 2–3. The formed precipitate was collected by filtration, washed with water, and recrystallized from MeCN to give pure **7a–g** in 40–70% yield.

Analytical Data for Representative Compounds

2-Methyl-[(2-oxo-2,3,4,5-tetrahydro-1*H*-1-benzo[*b*]azepine-7-yl)-sulfonyl]-aminoacetic acid (7a): yield 55%, mp 237–240°C; LCMS m/z 313 (M + 1); ^{1}H NMR (500 MHz): δ 2.23 (m, 4H, $^{4,3}CH_2$), 2.8 (m, 5H, $^{5}CH_2$, NCH_3), 3.82 (s, 2H, NCH_2), 7.11 (d, 1H, $J_{9-8} = 8.3$ Hz, ^{9}CH), 7.56 (dd, 1H, $J_{8-9} = 8.3$ Hz, $J_{8-6} = 1.8$ Hz, ^{8}CH), 7.59 (d, 1H, $J_{6-8} = 1.8$ Hz, ^{6}CH), 9.75 [s, 1H, C(O)NH], (s, 1H, COOH). Anal. calcd. for $C_{13}H_{16}N_2O_5S$: C (49.99%), H (5.16%), N (8.97%). Found: C (50.06%), H (5.18%), N (8.91%).

3-Methyl-2-(2-oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepine-7-sulfonylamino)-butyric acid (7b): yield 67%, mp 221–222°C; LCMS m/z 341 (M + 1); ^{1}H NMR (500 MHz): δ 0.85 (d, 3H, CH_3), 0.9 (d, 3H, CH_3), 2.0 (m, 1H, CCHC), 2.2 (m, 4H, $^{4,3}CH_2$), 2.8 (m, 2H, $^{5}CH_2$), 3.5 [m, 1H, NCHC(O)], 7.05 (d, 1H, $J_{9-8} = 8.3$ Hz, ^{9}CH), 7.16 [d, 1H, $J_{S(O)NH\text{-}NCH2} = 6.6$ Hz, S(O)NH], 7.58 (m, 2H, $^{6,8}CH$), 9.7 [s, 1H, C(O)NH], 12.1 (s, 1H, COOH). Anal. calcd. for $C_{15}H_{20}N_2O_5S$: C (52.93%), H (5.92%), N (8.23%). Found: C (52.99%), H (5.86%), N (8.28%).

General Procedure for Synthesis of Ethyl 4-(2-Oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepine-7-sulfonylamino)-carboxylates (9a,b)

2-Oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepine-7-sulfonyl chloride **2** (0.2 mol) was slowly added to a solution of ethyl carboxylate **8a,b** (0.22 mol) and

TEA (0.22 mol) in dry DMF (50 mL). The mixture was stirred for 15 h at room temperature and then mixed with 5% solution of hydrochloric acid (200 mL). The formed precipitate was collected by filtration, washed with water, and recrystallized from ethanol to give pure **9a,b** in 70–80% yield.

Analytical Data

1-(2-Oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepine-7-sulfonyl)-piperidine-4-carboxylic acid ethyl ester (8a): yield 80%, mp 165–167°C, LCMS m/z 381 (M + 1); ^{1}H NMR (500 MHz): δ 1.15 (t, 3H, CH_3), 1.7 (m, 2Hax, CCH_2), 1.9 (m, 2Heq, CCH_2), 2.25 [m, 5H, C(O)CH, $^{4,3}CH_2$], 2.4 (m, 2Hax, NCH_2), 2.9 (m, 2H, $^5CH_2$), 3.55 (m, 2Heq, NCH_2), 4.05 (m, 2H, OCH_2), 7.1 (d, 1H, $J_{9-8} = 8.3$ Hz, ^{9}CH), 7.50 (dd, 1H, $J_{8-9} = 8.3$ Hz, $J_{8-6} = 1.8$ Hz, $^{-8}$CH), 7.55 (d, 1H, $J_{6-8} = 1.8$ Hz, ^{6}CH), 9.8 [s, 1H, C(O)NH]. Anal. calcd. for $C_{18}H_{24}N_2O_5$ S: C (56.82%), H (6.36%), N (7.36%). Found: C (56.94%), H (6.38%), N (7.34%).

General Procedure for Synthesis of 4-(2-Oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepine-7-sulfonylamino)-carboxylic Acids (7 h,i).

A suspension of ester **9a,b** (0.1 mol) in a mixture of ethanol (30 mL) and 5% solution of NaOH (100 mL) was stirred at 80°C for 1 h. The unsolved precipitate was filtered off; the clear solution was cooled down to room temperature and acidified with 10% solution of hydrochloric acid to maintain pH 2–3. The formed precipitate was collected by filtration, washed with water, and recrystallized from MeCN to give pure **7h,i** in 55–70% yields.

Analytical Data

1-[(2-Oxo-2,3,4,5-tetrahydro-1*H*-1-benzo[*b*]azepine-7-yl)sulfonyl]-4-piperidinecarboxylic acid (7 h): yield 55%, mp 270–273°C, LCMS m/z 353 (M + 1); ^{1}H NMR (500 MHz): δ 1.65 (m, 2Hax, CCH_2), 1.9 (m, 2Heq, CCH_2), 2.18 [m, 1H, C(O)CH], 2.22 (m, 4H, $^{4,3}CH_2$), 2.5 (m, $2H_{ax}$, NCH_2), 2.8 (m, 2H, $^5CH_2$), 3.5 (m, 2Heq, NCH_2), 7.1 (d, 1H, $J_{9-8} = 8.3$ Hz, ^{9}CH), 7.50 (dd, 1H, $J_{8-9} = 8.3$ Hz, $J_{8-6} = 1.8$ Hz, ^{8}CH), 7.55 (d, 1H, $J_{6-8} = 1.8$ Hz, ^{6}CH), 9.8 [s, 1H, C(O)NH], 12.0 (s, 1H, COOH). Anal. calcd. for $C_{16}H_{20}N_2O_5S$: C (54.53%), H (5.72%), N (7.95%). Found: C (54.58%), H (5.65%), N (7.88%).

General Procedure for Synthesis of 4-(2-Oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepine-7-sulfonylamino)-carboxylic Acid Amides 11a–i

A suspension of carboxylic acid **7a–i** (0.9 mmol) and CDI (0.8 mmol) in 4 mL of dry DMF was stirred at 50–60°C until full dissolution of the initial

material. Then amine **3** or **4** (0.9 mmol) was added to the resulting solution, and the mixture was stirred at 100°C for 2 h. Then the mixture was cooled down to room temperature and mixed with water (20–30 mL). The formed precipitate was collected by filtration, washed with cold water (50 mL), and recrystallized from ethanol to give pure **7a–i** in 30–70% yield.

Analytical Data for Representative Compounds

2-[Methyl-(2-oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepine-7-sulfonyl)-amino]-*N*-quinolin-4-yl-acetamide (11a): yield 52%, mp 254–257°C, LCMS m/z 439 (M + 1); ^{1}H NMR (500 MHz): δ 2.15 (m, 2H, $^4CH_2$), 2.25 (m, 2H, $^3CH_2$), 2.6 (s, 3H, NCH_3), 2.7 (m, 2H, $^5CH_2$), 2.85 (s, 3H, $ArCH_3$), 4.2 (s, 2H, NCH_2), 7.12 (d, 1H, $J_{9-8} = 8.3$ Hz, ^{9}CH), 7.45 (m, 1H, CHAr), 7.65 (m, 3H ^{6,8}CH, CHAr), 7.85 (d, 1H, CHAr), 7.9 (s, 1H, CHAr), 8.2 (d, 1H, CHAr), 9.6 [s, 1H, C(O)NH], 10.0 [s, 1H, C(O)NH]. Anal. calcd. for $C_{22}H_{22}N_4O_4S$: C (60.26%), H (5.06%), N (12.78%). Found: C (60.21%), H (4.98%), N (12.82%).

2-Oxo-2,3,4,5-tetrahydro-1*H*-benzo[*b*]azepine-7-sulfonic acid [1-(2,3-dihydro-indole-1-carbonyl)-2-methyl-propyl]-amide (11b): yield 45%, mp 231–234°C, LCMS m/z 442 (M + 1); ^{1}H NMR (500 MHz): δ 1.0 (m, 6H, $2CH_3$), 1.85 (m, 1H, CCHC) 2.0 (m, 2H, CCH_2), 2.5 (m, 4H, $^{4,3}CH_2$), 3.05 (m, 2H, $^5CH_2$), 3.85 [m, 1H, NCHC(O)], 4.0 (m, 2H, NCH_2), 6.95 (m, 2H, CHAr, ^{9}CH), 7.00 (t, 1H, CHAr), 7.05 [d, 1H, $J_{S(O)NH-NCH2} = 6.6$ Hz, S(O)NH], 7.5 (d, 1H, $J_{6-8} = 1.8$ Hz, ^{6}CH), 7.6 (m, 2H, ^{8}CH, CHAr), 7.85 (d, 1H, CHAr), 9.5 [s, 1H, C(O)NH]. Anal. calcd. for $C_{23}H_{27}N_3O_4S$: C (62.56%), H (6.16%), N (9.52%). Found: C (62.59%), H (6.10%), N (9.48%).

ACKNOWLEDGMENTS

The authors thank Konstantin V. Balakin (ChemDiv, Inc.) for discussion and help in preparation of the manuscript.

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