A New Synthesis of β -Sultams from Pentafluorophenyl (PFP) Sulfonates.

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Supplementary Information

General information

All reactions were carried out under a nitrogen atmosphere in oven-dried vessels. All reagents were purchased from Aldrich or AlfaAesar and were used as received without further purification. The isoxazolidine-sulfonic acid pentafluorophenyl esters were prepared according to the previously described method by Caddick.¹ All reactions were monitored by thin-layer chromatography (TLC) on pre-coated silica gel plates (254 μ m). Flash column chromatography was carried out with Kieselgel 60M 0.04/0.063mm (200-400 mesh) silica gel. ¹H NMR spectra were recorded at 300MHz and ¹³C at 75MHz on a Bruker AMX 300 at ambient temperature in CDCl₃. The chemical shifts (δ) for ¹H and ¹³C are quoted in ppm relative to residual signals of the solvent. Mass spectra were obtained on a VG70-SE mass spectrometer. Infrared spectra were obtained on a Shimadzu FTIR 8700 Spectrophotometer. Melting points were measured with a Gallenkamp apparatus and are uncorrected.

General experimental for the formation of β -sultams:

Isoxazolidine-sulfonic acid pentafluorophenyl ester (0.5 mmol) was added to a N₂ purged round bottom flask containing a stirrer bar. Acetonitrile (7.5 ml) and H₂O (0.5 ml) were added followed by hexacarbonyl molybdenum (131 mg, 0.5 mmol). The mixture was heated to reflux for 16 h at which time the reaction was allowed to cool and the compound adsorbed onto silica. The crude residue was purified by flash column chromatography (gradient solvent system starting at 7 : 3 petroleum ether 40-60 °C : diethyl ether to pure diethyl ether) to give the corresponding β -sultams. Where necessary recrystallisation was achieved using minimal dichloromethane and precipitating with petroleum ether 40-60 °C.

¹ Caddick, S.; Bush, H.D.; *Org. Lett.*, **2003**, 5, 2489.

Entry 1

(2-Methyl-3-naphthalen-2-yl-1,1-dioxo-1λ⁶-[1,2]thiazetidin-4-yl)-methanol

Yield: 58% isolated, white solid

MPt. 161-164 °C

ν_{max} (thin film, cm^{-1}) 3600.9 w, 3058.9 w, 1602.7 w, 1317.3 s, 1147.6 w

δ_{H} (CDCl_3 , 300MHz) 7.88 (m, 4H), 7.55 (dt, $J = 4.5, 3.6 \text{ Hz}$, 2H), 7.43 (dd, $J = 8.6, 1.5 \text{ Hz}$, 1H), 4.71 (dtd, $J = 8.8, 5.5, 2.6 \text{ Hz}$, 1H), 4.56 (d, $J = 8.2 \text{ Hz}$, 1H), 3.96 (ddd, $J = 12.7, 9.3, 5.6 \text{ Hz}$, 1H), 3.44 (ddd, $J = 12.8, 6.9, 5.9 \text{ Hz}$, 1H), 2.83 (s, 3H), 1.80 (t, $J = 6.4, 6.4 \text{ Hz}$, 1H)

δ_{C} (CDCl_3 , 75MHz) 133.2 (Ar), 130.0 (Ar), 129.1 (Ar), 128.0 (Ar), 127.9 (Ar), 126.9 (Ar), 126.3 (Ar), 124.2 (Ar), 75.2 (Ar), 58.8 (CH_2), 55.1 (CH), 30.7 (NCH_3)

LRMS (EI+) 277 (M^+ , 100%),

HRMS (EI+) Calc'd. for $\text{C}_{14}\text{H}_{15}\text{NO}_3$ 277.07726 Found: 277.07690

Entry 2

[3-(4-Methoxy-phenyl)-2-methyl-1,1-dioxo-1λ⁶-[1,2]thiazetidin-4-yl]-methanol

Yield: 53% isolated, white solid

MPt. 94-95 °C

ν_{max} (thin film, cm^{-1}) 3587.4 w, 3062.7 w, 1612.4 m, 1514.0 s, 1317.3 s, 1149.5 s 1033.8 m

δ_{H} (CDCl₃, 300MHz) 7.28 (d, J = 8.6 Hz, 2H), 6.90 (d, J = 8.8 Hz, 2H), 4.58 (ddd, J = 9.5, 8.2, 5.4 Hz, 1H), 4.34 (d, J = 8.1 Hz, 1H), 3.87 (ddd, J = 12.5, 9.7, 5.7 Hz, 1H), 3.79 (s, 3H), 3.36 (dt, J = 12.3, 5.9 Hz, 1H), 2.70 (s, 3H), 2.43 (t, J = 6.3 Hz, 1H)

δ_{C} (CDCl₃, 75MHz) 160.1 (Ar), 128.4 (Ar), 124.4 (Ar), 114.5 (Ar), 75.4 (CH), 58.6 (CH₂), 55.4 (OCH₃), 54.4 (CH), 30.3 (NCH₃)

LRMS (EI+) 257 (M^+ , 23%), 148 (M^+ -Ar, H, 100%)

HRMS (EI+) Calc'd. for C₁₁H₁₅NO₄S 257.07218 Found: 257.0718

Entry 3

[3-(4-Allyloxy-phenyl)-2-methyl-1,1-dioxo-1 λ^6 -[1,2]thiazetidin-4-yl]-methanol

[3-(4-Allyloxy-phenyl)-2-methyl-1,1-dioxo-1 λ^6 -[1,2]thiazetidin-4-yl]-methanol

Yield: 52% isolated, white solid.

MPt. 74-76 °C

ν_{max} (thin film, cm^{-1}) 3587.4 w, 3060.8 w, 2885.3 w, 1610.5 m, 1510.2 s, 1312.4 s, 1147.6 m

δ_{H} (CDCl_3 , 300MHz) 7.27 (d, $J = 8.5$ Hz, 2H), 6.91 (d, $J = 8.8$ Hz, 2H), 6.02 (ddt, $J = 17.3$, 10.5, 5.3, Hz, 1H), 5.39 (ddt, $J = 17.3$, 1.5, 1.5 Hz, 1H), 5.28 (ddt, $J = 10.5$, 1.5, 1.5 Hz, 1H), 4.58 (m, 1H), 4.52 (dt, $J = 5.3$, 1.5 Hz, 2H), 4.34 (d, $J = 8.1$ Hz, 1H), 3.86 (t, $J = 10.8$, 10.7 Hz, 1H), 3.35 (d, $J = 12.3$ Hz, 1H), 2.70 (s, 3H), 2.51 (br s, 1H)

δ_{C} (CDCl_3 , 75MHz) 159.1 (Ar), 132.9 (CH), 128.3 (Ar), 124.6 (Ar), 118.0 (CH_2), 115.2 (Ar), 75.4 (CH), 68.9 (OCH_2), 58.6 (OCH_2), 54.4 (CH), 30.3 (NCH_3)

LRMS (EI+) 283 (M^+ , 48%), 134 (M^+ -Ar, OH, 100%)

HRMS (EI+) Calc'd. for $\text{C}_{13}\text{H}_{17}\text{NO}_4\text{S}$ 283.08782 Found: 283.08788

Entry 4

[3-(4-Bromo-phenyl)-2-methyl-1,1-dioxo-1λ⁶-[1,2]thiazetidin-4-yl]-methanol

[3-(4-Bromo-phenyl)-2-methyl-1,1-dioxo-1λ⁶-[1,2]thiazetidin-4-yl]-
methanol

Yield: 50% isolated, white solid.
MPt. 133-135 °C

ν_{max} (thin film, cm^{-1})

δ_{H} (CDCl_3 , 300MHz) 7.54 (d, $J = 8.4$ Hz, 2H), 7.27 (d, $J = 8.4$ Hz, 2H), 4.62 (ddd, $J = 8.8, 8.5, 5.4$ Hz, 1H), 4.35 (d, $J = 8.2$ Hz, 1H), 3.94-3.79 (m, 1H), 3.46-3.32 (m, 1H), 2.74 (s, 3H), 2.17 (br s, 1H)

δ_{C} (CDCl_3 , 75MHz) 132.3 (Ar), 131.9 (Ar), 128.7 (Ar), 123.2 (Ar), 75.1 (CH), 58.6 (CH_2), 54.5 (CH), 30.6 (NCH_3)

LRMS (EI+) 307 (M^{+81}Br , 5%), 305 (M^{+79}Br , 5%), 288 ($\text{M}^{+}\text{-OH}$, 3%), 196 (M^{+79}Br , 100%), 198 (M^{+81}Br), -Br, CH_2OH , 100%), 131 ($\text{M}^{+}\text{-Ar}$, OH, H, 27%)

HRMS (EI+) Calc'd. for $\text{C}_{10}\text{H}_{12}\text{BrNO}_3\text{S}$ 304.97212 Found: 304.97162

Elemental analysis: Anal.: Calc'd. for $\text{C}_{10}\text{H}_{12}\text{BrNO}_3\text{S}$ C: 39.35, H: 3.97, N: 4.59; Found C: 39.67, H: 4.16, N: 4.1

Entry 5

[3-(2-Chloro-phenyl)-2-methyl-1,1-dioxo-1λ⁶-[1,2]thiazetidin-4-yl]-methanol

[3-(2-Chloro-phenyl)-2-methyl-1,1-dioxo-1λ⁶-[1,2]thiazetidin-4-yl]-methanol

Yield: 47% isolated, white solid.

MPt. 115-116 °C

$\nu_{\max}$ (thin film, cm^{-1}) 3587.4 w, 3062.7 w, 1446.5 w, 1319.2 m,

δ_{H} (CDCl₃, 300MHz) 7.58 (m, 1H), 7.37 (m, 3H), 4.79 (ddd, J = 9.4, 8.2, 4.8 Hz, 1H), 4.58 (d, J = 8.1 Hz, 1H), 3.74 (ddd, J = 12.7, 9.5, 5.9 Hz, 1H), 3.46 (ddd, J = 12.5, 7.4, 4.9 Hz, 1H), 2.80 (s, 3H), 2.22 (t, J = 6.7 Hz, 1H)

δ_{C} (CDCl₃, 75MHz) 133.0 (Ar), 131.3 (Ar), 130.1 (Ar), 129.8 (Ar), 127.6 (Ar), 127.4 (Ar), 74.5 (CH), 58.6 (CH₂), 52.9 (CH), 31.3 (NCH₃)

LRMS (EI+) 261 (M, 10%), 152 (M⁺-Ar)

HRMS (EI+) Calc'd. for C₁₀H₁₂ClNO₃S 261.02264 Found: 261.02297

Elemental analysis: Anal.: Calc'd. for C₁₀H₁₂ClNO₃S C:45.97 H: 4.63 N: 5.37 Found C:46.15 H: 4.66 N: 5.12

Crystal Structure:

Table 1. Crystal data and structure refinement for str0434.

Identification code	str0434	
Chemical formula	C ₁₀ H ₁₂ ClNO ₃ S	
Formula weight	261.72	
Temperature	293(2) K	
Radiation, wavelength	MoK α , 0.71073 Å	
Crystal system, space group	triclinic, P $\bar{1}$	
Unit cell parameters	a = 7.6575(6) Å b = 8.1030(7) Å c = 9.3266(8) Å 563.99(8) Å ³	α = 84.4330(10) ^o β = 84.9250(10) ^o γ = 79.0290(10) ^o
Cell volume	2	
Z	1.541 g/cm ³	
Calculated density	0.514 mm ⁻¹	
Absorption coefficient μ	272	
F(000)	colourless, 0.44 × 0.42 × 0.08 mm ³	
Crystal colour and size	Brucker SMART Apex CCD diffractometer	
Data collection method	ω rotation with narrow frames	
θ range for data collection	2.20 to 28.25 ^o	
Index ranges	h -9 to 10, k -10 to 10, l -11 to 12	

Completeness to $\theta = 26.00^\circ$	97.9 %
Reflections collected	4875
Independent reflections	2558 ($R_{\text{int}} = 0.0171$)
Reflections with $F^2 > 2\sigma$	2448
Absorption correction	semi-empirical from equivalents
Min. and max. transmission	0.8054 and 0.9600
Structure solution	direct methods
Refinement method	Full-matrix least-squares on F^2
Weighting parameters a, b	0.0449, 0.1962
Data / restraints / parameters	2558 / 0 / 145
Final R indices [$F^2 > 2\sigma$]	$R1 = 0.0285$, $wR2 = 0.0761$
R indices (all data)	$R1 = 0.0296$, $wR2 = 0.0771$
Goodness-of-fit on F^2	1.038
Largest and mean shift/su	0.001 and 0.000
Largest diff. peak and hole	0.352 and $-0.428 \text{ e \AA}^{-3}$

Table 2. Atomic coordinates and equivalent isotropic displacement parameters ($\text{\AA}^2$) for str0434. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Cl(1)	0.69255(4)	0.68486(4)	0.35074(4)	0.03097(11)
S(1)	0.20035(4)	0.66986(4)	0.03427(3)	0.01822(10)
O(1)	0.27770(13)	0.53430(12)	−0.05352(10)	0.0269(2)
O(2)	0.04572(12)	0.78154(12)	−0.01769(10)	0.0270(2)
O(3)	0.30393(12)	1.04315(12)	−0.01700(10)	0.0254(2)
N(1)	0.17071(14)	0.60565(13)	0.20722(11)	0.0200(2)
C(1)	0.30151(16)	0.95296(15)	0.12126(13)	0.0204(2)
C(2)	0.36338(15)	0.76638(14)	0.10404(12)	0.0173(2)
C(3)	0.34361(15)	0.64430(14)	0.24317(12)	0.0176(2)
C(4)	0.33254(16)	0.71912(14)	0.38632(12)	0.0188(2)
C(5)	0.16998(18)	0.76944(16)	0.46372(14)	0.0247(3)
C(6)	0.1632(2)	0.83714(17)	0.59592(14)	0.0299(3)
C(7)	0.3178(2)	0.85735(17)	0.65242(14)	0.0308(3)
C(8)	0.4812(2)	0.81018(17)	0.57626(14)	0.0282(3)
C(9)	0.48637(17)	0.74095(15)	0.44502(13)	0.0217(2)
C(10)	0.1230(2)	0.44079(17)	0.25059(15)	0.0284(3)

Table 3. Bond lengths [$\text{\AA}$] and angles [$^\circ$] for str0434.

Cl(1)–C(9)	1.7410(13)	S(1)–O(2)	1.4368(9)
S(1)–O(1)	1.4384(9)	S(1)–N(1)	1.6558(10)
S(1)–C(2)	1.7910(12)	S(1)–C(3)	2.2925(12)
O(3)–C(1)	1.4196(14)	N(1)–C(10)	1.4617(16)
N(1)–C(3)	1.4900(15)	C(1)–C(2)	1.5156(16)
C(2)–C(3)	1.5679(16)	C(3)–C(4)	1.5082(15)
C(4)–C(9)	1.3895(17)	C(4)–C(5)	1.3935(17)
C(5)–C(6)	1.3903(18)	C(6)–C(7)	1.381(2)
C(7)–C(8)	1.389(2)	C(8)–C(9)	1.3895(17)
O(2)–S(1)–O(1)	116.09(6)	O(2)–S(1)–N(1)	112.20(6)
O(1)–S(1)–N(1)	112.84(6)	O(2)–S(1)–C(2)	116.50(6)
O(1)–S(1)–C(2)	113.16(5)	N(1)–S(1)–C(2)	81.14(5)
O(2)–S(1)–C(3)	133.45(5)	O(1)–S(1)–C(3)	110.21(5)
N(1)–S(1)–C(3)	40.45(4)	C(2)–S(1)–C(3)	43.01(5)
C(10)–N(1)–C(3)	120.94(10)	C(10)–N(1)–S(1)	120.67(8)
C(3)–N(1)–S(1)	93.42(7)	O(3)–C(1)–C(2)	109.13(9)
C(1)–C(2)–C(3)	116.26(9)	C(1)–C(2)–S(1)	113.50(8)
C(3)–C(2)–S(1)	85.81(7)	N(1)–C(3)–C(4)	113.25(9)
N(1)–C(3)–C(2)	94.39(8)	C(4)–C(3)–C(2)	116.76(9)
N(1)–C(3)–S(1)	46.13(5)	C(4)–C(3)–S(1)	141.75(8)
C(2)–C(3)–S(1)	51.18(5)	C(9)–C(4)–C(5)	117.71(11)
C(9)–C(4)–C(3)	120.43(10)	C(5)–C(4)–C(3)	121.85(11)
C(6)–C(5)–C(4)	120.83(12)	C(7)–C(6)–C(5)	120.48(13)
C(6)–C(7)–C(8)	119.70(12)	C(7)–C(8)–C(9)	119.27(13)
C(4)–C(9)–C(8)	122.00(12)	C(4)–C(9)–Cl(1)	119.60(9)
C(8)–C(9)–Cl(1)	118.38(10)		

Table 4. Anisotropic displacement parameters ($\text{\AA}^2$) for str0434. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + \dots + 2hka^*b^*U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	0.02051(16)	0.0416(2)	0.03176(19)	-0.01017(14)	-0.00556(12)	-0.00292(13)
S(1)	0.01692(15)	0.02178(16)	0.01585(15)	-0.00357(10)	-0.00250(10)	-0.00167(11)
O(1)	0.0277(5)	0.0304(5)	0.0232(5)	-0.0114(4)	-0.0026(4)	-0.0019(4)
O(2)	0.0209(4)	0.0315(5)	0.0272(5)	-0.0007(4)	-0.0071(4)	0.0002(4)
O(3)	0.0222(4)	0.0268(4)	0.0236(4)	0.0060(3)	0.0013(3)	-0.0008(3)
N(1)	0.0209(5)	0.0235(5)	0.0170(5)	-0.0014(4)	-0.0026(4)	-0.0076(4)
C(1)	0.0233(6)	0.0199(5)	0.0175(5)	-0.0017(4)	0.0018(4)	-0.0038(4)
C(2)	0.0157(5)	0.0212(5)	0.0151(5)	-0.0027(4)	-0.0008(4)	-0.0030(4)
C(3)	0.0176(5)	0.0196(5)	0.0162(5)	-0.0026(4)	-0.0019(4)	-0.0036(4)
C(4)	0.0235(6)	0.0180(5)	0.0151(5)	-0.0005(4)	-0.0017(4)	-0.0045(4)
C(5)	0.0259(6)	0.0269(6)	0.0216(6)	-0.0034(5)	0.0022(5)	-0.0069(5)
C(6)	0.0386(8)	0.0280(6)	0.0218(6)	-0.0049(5)	0.0087(5)	-0.0065(6)
C(7)	0.0508(9)	0.0262(6)	0.0157(6)	-0.0038(5)	-0.0012(5)	-0.0079(6)
C(8)	0.0376(7)	0.0279(6)	0.0210(6)	-0.0032(5)	-0.0104(5)	-0.0066(5)
C(9)	0.0251(6)	0.0214(6)	0.0186(5)	-0.0006(4)	-0.0039(4)	-0.0035(5)
C(10)	0.0337(7)	0.0276(6)	0.0274(6)	0.0014(5)	-0.0060(5)	-0.0147(5)

Table 5. Hydrogen coordinates and isotropic displacement parameters ($\text{\AA}^2$) for str0434.

	x	y	z	U
H(3B)	0.2070	1.1051	-0.0271	0.038
H(1A)	0.1817	0.9725	0.1674	0.025
H(1B)	0.3797	0.9918	0.1817	0.025
H(2A)	0.4810	0.7412	0.0520	0.021
H(3A)	0.4372	0.5434	0.2404	0.021
H(5A)	0.0647	0.7576	0.4265	0.030
H(6A)	0.0537	0.8690	0.6467	0.036
H(7A)	0.3126	0.9023	0.7411	0.037
H(8A)	0.5859	0.8248	0.6127	0.034
H(10A)	0.1114	0.4244	0.3541	0.043
H(10B)	0.2144	0.3540	0.2135	0.043
H(10C)	0.0117	0.4357	0.2127	0.043

Table 6. Torsion angles [$^\circ$] for str0434.

O(2)-S(1)-N(1)-C(10)	98.86(11)	O(1)-S(1)-N(1)-C(10)	-34.56(12)
C(2)-S(1)-N(1)-C(10)	-146.04(11)	C(3)-S(1)-N(1)-C(10)	-129.37(13)
O(2)-S(1)-N(1)-C(3)	-131.77(7)	O(1)-S(1)-N(1)-C(3)	94.81(7)
C(2)-S(1)-N(1)-C(3)	-16.68(7)	O(3)-C(1)-C(2)-C(3)	-171.59(9)
O(3)-C(1)-C(2)-S(1)	-74.38(11)	O(2)-S(1)-C(2)-C(1)	9.44(10)
O(1)-S(1)-C(2)-C(1)	147.84(8)	N(1)-S(1)-C(2)-C(1)	-101.02(9)
C(3)-S(1)-C(2)-C(1)	-116.86(11)	O(2)-S(1)-C(2)-C(3)	126.30(7)
O(1)-S(1)-C(2)-C(3)	-95.30(7)	N(1)-S(1)-C(2)-C(3)	15.84(7)
C(10)-N(1)-C(3)-C(4)	-90.25(13)	S(1)-N(1)-C(3)-C(4)	140.58(9)
C(10)-N(1)-C(3)-C(2)	148.12(11)	S(1)-N(1)-C(3)-C(2)	18.96(8)
C(10)-N(1)-C(3)-S(1)	129.17(12)	C(1)-C(2)-C(3)-N(1)	96.69(11)
S(1)-C(2)-C(3)-N(1)	-17.49(7)	C(1)-C(2)-C(3)-C(4)	-22.13(15)
S(1)-C(2)-C(3)-C(4)	-136.31(9)	C(1)-C(2)-C(3)-S(1)	114.18(10)

O(2)–S(1)–C(3)–N(1)	72.01(9)	O(1)–S(1)–C(3)–N(1)	–101.87(8)
C(2)–S(1)–C(3)–N(1)	155.44(10)	O(2)–S(1)–C(3)–C(4)	1.57(16)
O(1)–S(1)–C(3)–C(4)	–172.31(12)	N(1)–S(1)–C(3)–C(4)	–70.44(13)
C(2)–S(1)–C(3)–C(4)	85.00(14)	O(2)–S(1)–C(3)–C(2)	–83.43(9)
O(1)–S(1)–C(3)–C(2)	102.69(8)	N(1)–S(1)–C(3)–C(2)	–155.44(10)
N(1)–C(3)–C(4)–C(9)	171.17(10)	C(2)–C(3)–C(4)–C(9)	–80.77(14)
S(1)–C(3)–C(4)–C(9)	–141.15(11)	N(1)–C(3)–C(4)–C(5)	–9.21(16)
C(2)–C(3)–C(4)–C(5)	98.85(13)	S(1)–C(3)–C(4)–C(5)	38.47(19)
C(9)–C(4)–C(5)–C(6)	–0.78(19)	C(3)–C(4)–C(5)–C(6)	179.60(11)
C(4)–C(5)–C(6)–C(7)	0.6(2)	C(5)–C(6)–C(7)–C(8)	0.2(2)
C(6)–C(7)–C(8)–C(9)	–0.9(2)	C(5)–C(4)–C(9)–C(8)	0.06(18)
C(3)–C(4)–C(9)–C(8)	179.69(11)	C(5)–C(4)–C(9)–Cl(1)	–178.48(9)
C(3)–C(4)–C(9)–Cl(1)	1.15(16)	C(7)–C(8)–C(9)–C(4)	0.8(2)
C(7)–C(8)–C(9)–Cl(1)	179.35(10)		

Entry 6

(2-Methyl-1,1-dioxo-3-phenyl-1λ⁶-[1,2]thiazetidin-4-yl)-methanol

(2-Methyl-1,1-dioxo-3-phenyl-1λ⁶-[1,2]thiazetidin-4-yl)-methanol

Yield: 37% isolated, white solid

MPt. 114-116 °C

ν_{max} (thin film, cm^{-1}) 3600.9 w, 3076.2 w, 1317.3 s, 1149.5 s

δ_{H} (CDCl₃, 300MHz) 7.75-7.26 (m, 5H), 4.64 (m, 1H), 4.41 (t, J = 6.8, 6.8 Hz, 1H), 3.85 (t, J = 11.0, 11.0 Hz, 1H), 3.35 (br m, 1H), 2.74 (s, 3H), 2.48 (br s, 1H)

δ_{C} (CDCl₃, 75MHz) 132.8 (Ar), 129.0 (Ar), 128.7 (Ar), 127.0 (Ar), 75.3 (CH), 58.6 (CH₂), 54.8 (CH), 30.4 (NCH₃)

LRMS (EI+) 228 (M⁺, 75%), 118 (M⁺-Ar, CH₂OH, 100%)

HRMS (EI+) Calc'd. for C₁₀H₁₃NO₃S 227.06161 Found: 227.06202

Elemental analysis: Anal.: Calc'd. for C₁₀H₁₃NO₃S C:52.85, H: 5.77 N: 6.17 Found C:53.14, H: 5.93 N: 5.97

Entry 7

[3-(4-Chloro-phenyl)-2-methyl-1,1-dioxo-1λ⁶-[1,2]thiazetidin-4-yl]-methanol

Yield: 37% isolated, white solid.

MPt. 126-127 °C

ν_{max} (thin film, cm^{-1}) 3681.9 w, 3600.9 m, 2885.3 w, 1600.8 w, 1490.9 s, 1319.2 s, 1119.5 s.

δ_{H} (CDCl_3 , 300MHz) 7.39 (d, $J = 8.7\text{Hz}$, 2H), 7.32 (d, $J = 8.5\text{Hz}$, 2H), 4.62 (ddd, $J = 9.4, 8.3, 5.3\text{Hz}$, 1H), 4.37 (d, $J = 8.2\text{Hz}$, 1H), 3.86 (ddd, $J = 12.5, 9.5, 5.7\text{Hz}$, 1H), 3.39 (ddd, $J = 12.4, 6.7, 5.5\text{Hz}$, 1H), 2.74 (s, 3H), 2.24 (t, $J = 6.3\text{Hz}$, 1H)

δ_{C} (CDCl_3 , 75MHz) 135.1 (Ar), 131.3 (Ar), 129.3 (Ar), 128.4 (Ar), 75.2 (CH), 58.5 (CH_2), 54.4 (CH), 30.6 (NCH_3)

LRMS (EI+) 154($\text{M}^+(\text{}^{37}\text{Cl})$ -Ar 42%) 152 ($\text{M}^+(\text{}^{35}\text{Cl})$ -Ar 100%)

HRMS (EI+) Calc'd. for $\text{C}_{10}\text{H}_{12}\text{ClNO}_3\text{S}$ 261.02264 Found: 261.02275

Entry 8

[3-(2-Bromo-phenyl)-2-methyl-1,1-dioxo-1λ⁶-[1,2]thiazetidin-4-yl]-methanol

Yield: 27% isolated, white solid.

MPt. 120-122 °C

ν_{max} (thin film, cm^{-1}) 3595.1 m, 3062.7 w, 2885.3 w, 1467.7 m, 1319.2 s, 1271.0 s, 1151.4 s, 1043.4 m.

δ_{H} (CDCl₃, 300MHz) 7.57 (d, J = 7.9 Hz, 2H), 7.39 (t, J = 7.6 Hz, 1H), 7.24 (dt, J = 7.8, 1.7 Hz, 1H), 4.80 (ddd, J = 9.6, 8.3, 4.8 Hz, 1H), 4.52 (d, J = 8.2 Hz, 1H), 3.71 (ddd, J = 12.5, 9.9, 5.6 Hz, 1H), 3.43 (m, 1H), 2.78 (s, 3H), 2.37 (t, J = 6.4 Hz, 1H)

δ_{C} (CDCl₃, 75MHz) 133.1 (Ar), 132.7 (Ar), 130.4 (Ar), 127.9 (Ar), 122.9 (Ar), 74.4 (CH), 58.7 (CH₂), 55.0 (CH), 31.3 (NCH₃)

LRMS (EI+) 307 (M^{+81}Br , 7%), 305 (M^{+79}Br , 7%), 196 (M^{+79}Br), 198 (M^{+81}Br), Ar, CH₂OH, 100%)

HRMS (EI+) Calc'd. for C₁₀H₁₂BrNO₃S 304.97212 Found: 304.97241

Entry 9

(3-Cyclopropyl-2-methyl-1,1-dioxo-1λ⁶-[1,2]thiazetidin-4-yl)-methanol

(3-Cyclopropyl-2-methyl-1,1-dioxo-1λ⁶-[1,2]thiazetidin-4-yl)-methanol

Yield: 27% isolated, yellow oil.

ν_{max} (thin film, cm^{-1}) 3683.8 w, 3057.0 w, 1604.7 w, 1319.5 m, 1041.5 w.
 δ_{H} (CDCl_3 , 300MHz) 4.41 (tt, $J = 8.7, 4.4$ Hz, 1H), 4.36-4.22 (m, 1H), 4.15 (dt, $J = 10.6, 5.1$ Hz, 1H), 2.82 (dd, $J = 8.2, 4.2$ Hz, 1H), 2.72 (s, 3H), 2.54 (dd, $J = 9.5, 7.8$ Hz, 1H), 1.02 (dtt, $J = 9.6, 6.3, 4.9$ Hz, 1H), 0.69 (m, 1H), 0.57 (m, 1H), 0.31 (dt, $J = 10.0, 4.7$ Hz, 1H), 0.12 (dt, $J = 9.9, 5.0$ Hz, 1H)
 δ_{C} (CDCl_3 , 75MHz) 74.5 (CH), 58.0 (CH_2), 57.7 (CH), 30.3 (CH_3), 8.4 (CH), 4.0 (CH_2), 0.6 (CH_2)
HRMS (EI+) Calc'd. for $\text{C}_7\text{H}_{13}\text{NO}_3\text{S}$ 191.06139 Found: 191.06139

Entry 10

[3-(3-Bromo-phenyl)-2-methyl-1,1-dioxo-1λ⁶-[1,2]thiazetidin-4-yl]-methanol

[3-(3-Bromo-phenyl)-2-methyl-1,1-dioxo-1λ⁶-[1,2]thiazetidin-4-yl]-methanol

Yield: 26% isolated yield, white solid.

MPt. 76-79 °C

ν_{max} (thin film, cm^{-1}) 3676.1 w, 3057.0 w, 1604.7 w, 1319.2 m, 1149.5 m.

δ_{H} (CDCl_3 , 300MHz) 7.54 (d, $J = 1.6$ Hz, 1H), 7.49 (dt, $J = 7.0$, 1.9 Hz, 1H), 7.30 (m, 2H), 4.64 (ddd, $J = 9.5$, 8.3, 5.3 Hz, 1H), 4.34 (d, $J = 8.3$ Hz, 1H), 3.87 (dd, $J = 12.4$, 9.7 Hz, 1H), 3.40 (dd, $J = 12.5$, 5.1 Hz, 1H), 2.75 (s, 3H), 2.28 (br s, 1H)

δ_{C} (CDCl_3 , 75MHz) 135.3 (Ar), 132.2 (Ar), 130.6 (Ar), 130.0 (Ar), 125.8 (Ar), 123.3 (Ar), 75.3 (CH), 58.5 (CH_2), 54.2 (CH), 30.7 (NCH_3)

HRMS (EI^+) Calc'd. for $\text{C}_{10}\text{H}_{12}\text{BrNO}_3\text{S}$ 304.97212 Found: 304.97215