Electronic Supplementary Information

for

The intramolecular amination of allenes

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(1) Details for the single crystal X-ray diffraction experiments

(2) Experimental procedures and characterisation data for experiments in Schemes 1, 3 and 4 and Table 1

(3) ¹H NMR and ¹³C NMR spectra [spectra are the machine-generated original PDFs, except that the spectra for 7, 8, 14, 15, 28, and 29 (obtained at GSK, Harlow, UK) were generated in MestReNova for Mac OS X]

X-ray details and data summary

Single crystal X-ray diffraction data were obtained using a Nonius Kappa-CCD area detector diffractometer, with graphite-monochromated Mo-K α radiation ($\lambda = 0.71073$ Å) at 150 K. Cell parameters and intensity data were processed using the DENZO-SMN package¹ and reflection intensities were corrected for absorption effects by the multi-scan method, based on multiple scans of identical and Laue equivalent reflections. The structures were solved by direct methods using SIR92² and refined by full-matrix least squares on F^2 using the CRYSTALS suite³ as per the details in the CIF.

Crystal data for Compound **5**: (clear colourless, $0.14 \times 0.30 \times 0.34$ mm): $C_{10}H_{17}NO_5S M_r = 263.31$; monoclinic, $P2_1/n$; a = 6.5055(2) Å, b = 22.4224(6) Å, c = 8.9905(3) Å, $\beta = 108.6921(10)^\circ$, V = 1019.55(6) Å³; Z = 4; $\mu = 0.270$ mm⁻¹; $D_{calc} = 1.408$ gcm⁻³; reflections collected = 12353; independent reflections = 2829 ($R_{int} = 0.035$); R values [$I > 2\sigma(I)$, 2141 reflections]: $R_1 = 0.0391$, w $R_2 = 0.0864$; $\rho_{min/max} = -0.47/0.41$ e Å⁻³; CCDC 757478. Crystal data for Compound **12**: (clear colourless, $0.34 \times 0.62 \times 0.64$ mm): $C_{10}H_{17}NO_5S M_r = 263.31$; triclinic, $P\overline{1}$; a = 7.6562(2) Å, b = 8.7683(2) Å, c = 10.0293(2) Å, $a = 77.2922(12)^\circ$, $\beta = 78.9430(12)^\circ$, $\gamma = 69.3572(10)^\circ$, V = 609.79(2) Å³; Z = 2; $\mu = 0.275$ mm⁻¹; $D_{calc} = 1.434$ gcm⁻³; Reflections collected = 8663; independent reflections = 2766 ($R_{int} = 0.021$); R values [$I > 2\sigma(I)$, 2569 reflections]: $R_1 = 0.0330$, w $R_2 = 0.0837$; $\rho_{min/max} = -0.40/0.34$ e Å⁻³; CCDC 757479. Crystal data for Compound **17** were published by Feast *et al.*⁴ but are included in the CIF for completeness (CCDC 763606).

Crystallographic data (excluding structure factors) for the structures of **5**, **12** and **17** have been deposited with the Cambridge Crystallographic Data Centre (CCDC 757478-9 & 763606). Copies of these data can be obtained free of charge via <u>www.ccdc.cam.ac.uk/data_request/cif</u>.

Z. Otwinowski and W. Minor, Processing of X-ray Diffraction Data Collected in Oscillation Mode, *Methods Enzymol.* 1997, 276, eds. C. W. Carter, R. M. Sweet, Academic Press.

² A. Altomare, G. Cascarano, C. Giacovazzo, A. Guagliardi, M. C. Burla, G. Polidori and M. Camalli, *J. Appl. Crystallogr.* 1994, 27, 435.

³ P. W. Betteridge, J. R. Carruthers, R. I. Cooper, K. Prout and D. J. Watkin, *J. Appl. Crystallogr.* 2003, **36**, 1487.

⁴ G. C. Feast, J. Haestier, L. W. Page, J. Robertson, A. L. Thompson and D. J. Watkin, *Acta Cryst.* 2009, C65, o635.

General Procedure A

To a solution of sulfamate (1.0 equiv.) in dichloromethane (20 mL/mmol) was added MgO (2.3 equiv.), rhodium(II) acetate dimer (0.05 equiv.) and iodobenzene diacetate (1.3 equiv.) at RT. After stirring for 18 h, the reaction mixture was filtered through Celite and concentrated *in vacuo* to give the crude product.

4-Oxo-1-(sulfamoyloxy)pentan-3-yl acetate 2

The reaction of penta-3,4-dienyl sulfamate (1) according to General Procedure A and purification by column chromatography (ether) afforded the *title compound* (63 mg, 35%) as a colourless oil. R_f 0.14 (ether); v_{max} (thin film)/cm⁻¹ 3627 br, 1726 br, 1564 s, 1374 s, 1244 s; δ_H (400 MHz, CDCl₃) 2.19 (3 H, s, CH₃COCH), 2.21 (3 H, s, CH₃CO₂), 2.26–2.34 (2 H, m, CH₂CHO), 4.22–4.33 (2 H, m, CH₂O), 5.19 (1 H, dd, *J* 8.3, 4.2, CHOAc); δ_C (100 MHz, CDCl₃) 20.5, 26.1, 29.5, 66.3, 74.8, 170.7, 205.3; HRMS (ESI⁺) found 262.0354, C₇H₁₃NNaO₆S (MNa⁺) requires 262.0356.

(E)-4-(2-Methylpropylidene)-2,2-dioxido-1,2,3-oxathiazepan-5-yl acetate 5

The reaction of 6-methylhepta-3,4-dienyl sulfamate (4) according to General Procedure A and purification by column chromatography (petrol/ether, 2:1) afforded the *title compound* (382 mg, 52%) as a white crystalline solid. R_f 0.52 (ether); m.p. 87 °C; v_{max} (thin film)/cm⁻¹ 3272 br, 2965 s, 2872 w, 1743 s, 1467 s, 1418 s, 1375 s; δ_H (400 MHz, CDCl₃) 1.04 (3 H, d, *J* 6.6) and 1.05 (3 H, d, *J* 6.7, Me_2 CH), 2.10–2.16 (1 H, m, CHH'CH₂O) overlays 2.13 (3 H, s, CH₃CO), 2.20 (1 H, dt, *J* 11.6, 3.3, CHH'CH₂O), 2.59–2.68 (1 H, m, Me₂CH), 4.25 (1 H, dt, *J* 12.3, 3.3) and 4.64 (1 H, dt, *J* 12.3, 1.3, CH₂OS), 5.77 (1 H, d, *J* 10.5, CH=), 5.92 (1 H, t, *J* 3.4, CHOAc), 6.28 (1 H, br s, NH); δ_C (100 MHz, CDCl₃) 21.1, 22.4, 22.6, 27.1, 34.7, 65.1, 66.7, 125.6, 143.0, 169.3; HRMS (ESI⁺) found 286.0719, C₁₀H₁₇NNaO₅S (MNa⁺) requires 286.0720.

4-Methyl-2,2-dioxido-5-phenyl-6,7-dihydro-5H-1,2,3-oxathiazepin-5-yl acetate 7

The reaction of 3-phenylpenta-3,4-dienyl sulfamate (**6**) according to General Procedure A and purification by column chromatography (petrol/ether, 2:1) afforded the *title compound* (135 mg, 24%) as a white crystalline solid. $R_f 0.32$ (ether); m.p. 118 °C; v_{max} (thin film)/cm⁻¹ 3068 s, 1745 s, 1638 s, 1448 m, 1368 s, 1258 w, 1234 s, 1180 s; δ_H (500 MHz, CDCl₃) 2.07 (1 H, ddd, *J* 14.8, 4.1, 1.1, CHH'CH₂O), 2.20 (3 H, s, CH₃CO), 2.24 (3 H, s, CH₃C=N), 3.43–3.51 (1 H, m, CHH'O), 4.06–4.15 (1 H, m, CHH'CH₂O), 4.30 (1 H, dd, *J* 11.0, 5.9, CHH'O), 7.37 (2 H, dd, *J* 7.7, 1.9) and 7.45–7.48 (3 H, m, Ph); δ_C (100 MHz, CDCl₃) 21.3, 25.2, 36.7, 65.2, 88.4, 125.9, 129.3, 129.8, 133.6, 169.8, 181.4; HRMS (ESI⁺) found 320.0558, C₁₃H₁₅NNaO₅S (MNa⁺) requires 320.0563. Also obtained was **(2,2-dioxido-5-phenyl-6,7-dihydro-3H-1,2,3-oxathiazepin-4-yl)methyl acetate** (**8**) (26 mg, 5%) as a yellow solid. $R_f 0.49$ (ether); v_{max} (thin film)/cm⁻¹ 3377 br, 2919s, 2850 m, 1738 s, 1659 w, 1443 m, 1411 s; δ_H (400 MHz, CDCl₃) 2.11 (3 H, s, CH₃), 3.00 (2 H, t, *J* 5.0, CH₂CH₂O), 4.51 (2 H, app. tt, *J* 3.5, 1.5, CH₂O), 4.55 (2 H, s, CH₂OAc), 7.20–7.22 (2 H, m) and 7.34–7.41 (3 H, m, Ph); δ_C (100 MHz, CDCl₃) 20.8, 36.4, 63.1, 70.5, 128.0, 128.3, 128.6, 128.8, 135.9, 139.4, 171.4; HRMS (ESI⁺) found 320.0560, C₁₃H₁₅NNaO₅S (MNa⁺) requires 320.0563.

7-Methyl-3,3-dioxido-4-oxa-3-thia-2-azabicyclo[5.1.0]oct-1-yl acetate 10

The reaction of 3-methylpenta-3,4-dienyl sulfamate (**9**) according to General Procedure A and purification by column chromatography (petrol/ether, 2:1) afforded the *title compound* (236 mg, 49%) as a white crystalline solid. R_f 0.21 (petrol/ether, 2:1); m.p. 91 °C; v_{max} (thin film)/cm⁻¹ 3303 br, 2973 s, 2922 s, 2865 m, 1741 m, 1455 m, 1423 s, 1345 s; δ_H (400 MHz, CDCl₃) 1.03 (1 H, d, *J* 6.6, C*H*H'C(OAc)), 1.35 (3 H, s, CH₃C(CH₂)), 1.38 (1 H, d, *J* 6.6, CHH'C(OAc)), 1.93 (1 H, ddd, *J* 16.0, 12.0, 2.0, C*H*H'CH₂O), 2.10 (3 H, s, CH₃CO), 2.29 (1 H, dd, *J* 16.0, 4.6, CHH'CH₂O), 4.26 (1 H, ddd, *J* 12.0, 4.6, 2.0) and 4.67 (1 H, app. t, *J* 12.0, , CH₂O), 6.75 (1 H, br s, NH); δ_C (100 MHz, CDCl₃) 17.7, 21.2, 27.6, 31.1, 38.7, 69.2, 77.2, 171.6; HRMS (ESI⁺) found 258.0407, C₈H₁₃NNaO₅S (MNa⁺) requires 258.0407.

7-Propyl-3,3-dioxido-4-oxa-3-thia-2-azabicyclo[5.1.0]oct-1-yl acetate 12

The reaction of 3-(vinylidene)hexyl sulfamate (**11**) according to General Procedure A and purification by column chromatography (petrol/ether, 1:1) afforded the *title compound* (167 mg, 40%) as a white crystalline solid. R_f 0.42 (ether); m.p. 79 °C; v_{max} (thin film)/cm⁻¹ 3260 br, 2962 s, 1740 s, 1510 m, 1363 m, 1211 s, 1191 s; δ_H (400 MHz, CDCl₃) 0.95 (3 H, t, *J* 7.3 CH₃CH₂), 1.02 (1 H, d, *J* 6.6) and 1.34 (1 H, d, *J* 6.6, CHH'C(OAc)), 1.30–1.48 (2 H, m, CHH'Et overlays CHH'CH₃), 1.52–1.63 (1 H, m, CHH'CH₃), 1.70 (1 H, ddd, *J* 11.1, 4.3, 3.0, CHH'Et), 1.80 (1 H, dd, *J* 16.0, 11.2, CHH'CH₂O), 2.11 (3 H, s, CH₃CO), 2.42 (1 H, dd, *J* 16.0, 4.8, CHH'CH₂O), 4.24 (1 H, ddd, *J* 12.0, 4.8, 2.0) and 4.64 (1 H, app. t, *J* 12.0, CH₂O), 6.72 (1 H, br s, NH); δ_C (100 MHz, CDCl₃) 14.2, 19.8, 21.2, 30.2, 31.7, 32.4, 35.4, 69.5, 69.6, 171.6; HRMS (FI) found 263.0820, C₁₀H₁₇NO₅S (M⁺) requires 263.0827.

7-(1-Methylethyl)-4-oxa-3-thia-2-azabicyclo[5.1.0]octan-1-ol 3,3-dioxide 14

The reaction of 3-isopropylpenta-3,4-dienyl sulfamate (13) according to General Procedure A and purification by column chromatography (dichloromethane/ethyl acetate, 20:1) afforded the *title compound* (184 mg, 20%) as a white crystalline solid. R_f 0.20 (petrol/ethyl acetate, 2:1); m.p. 167 °C; v_{max} (thin film)/cm⁻¹ 3455 s, 3211 br, 2958 m, 1466 w, 1416 s, 1366 s, 1321 m, 1262 s, 1202 s, 1169 s; δ_H (400 MHz, CDCl₃) 0.88 (1 H, d, *J* 5.7) and 0.92 (1 H, d, *J* 5.7, CHH'C(OAc)), 1.01

(3 H, d, *J* 7.0) and 1.14 (3 H, d, *J* 7.0, *Me*₂CH), 1.68 (1 H, dddd, *J* 16.5, 11.6, 2.0, 0.4, CHH'CH₂O), 1.79 (1 H, dsept, *J* 7.0, 0.4, Me₂CH), 2.48 (1 H, dd, *J* 16.5, 4.6, CHH'CH₂O), 3.62 (1 H, br s, OH), 4.25 (1 H, ddd, *J* 12.3, 4.6, 2.0) and 4.69 (1 H, ddd, *J* 12.3, 11.6, 0.4, CH₂O), 6.05 (1 H, br s, NH); $\delta_{\rm C}$ (100 MHz, CDCl₃) 19.5, 20.5, 30.5, 31.0, 32.0, 37.8, 70.6, 71.6; HRMS (ESI⁺) found 244.0612, C₈H₁₅NNaO₄S (MNa⁺) requires 244.0614. Also obtained was **[5-(1-methyl)-2,2-dioxido-6,7-dihydro-3H-1,2,3-oxathiazepin-4-yl]methyl acetate** (**15**) (137 mg, 12%) as a white crystalline solid. *R*_f 0.24 (petrol/ethyl acetate, 2:1); v_{max} (thin film)/cm⁻¹ 3248 br, 2965 s, 1737 s, 1420 m, 1364 s, 1173 s; $\delta_{\rm H}$ (400 MHz, CDCl₃) 1.02 (6 H, d, *J* 6.8, *Me*₂CH), 2.09 (3 H, s, CH₃CO), 2.56–2.59 (2 H, m, CH₂CH₂O), 2.96 (1 H, sept, *J* 6.8, Me₂CH), 4.33–4.36 (2 H, m, CH₂O), 4.68 (2 H, s, CH₂OAc), 6.39 (1 H, br s, NH); $\delta_{\rm C}$ (100 MHz, CDCl₃) 20.1, 20.8, 27.8, 30.2, 62.2, 70.8, 125.8, 142.6, 171.4; HRMS (ESI⁻) found 262.0748, C₁₀H₁₆NO₅S (M–H)⁻ requires 262.0755.

6-(tert-Butyl)-3-oxa-2-thia-1-azabicyclo[5.1.0]oct-6-ene 2,2-dioxide 17

The reaction of 3-*tert*-butylpenta-3,4-dienyl sulfamate (**16**) according to General Procedure A and purification by column chromatography (petrol/ether, 4:1) afforded the *title compound* (160 mg, 68%) as a white crystalline solid; R_f 0.26 (petrol/ether, 2:1); m.p. 58 °C; v_{max} (thin film)/cm⁻¹ 3075m, 2968s, 1468m, 1359s, 1296w, 1261w, 1183s; δ_H (500 MHz, CD₂Cl₂, 228K) 1.11 (9H, s, (CH₃)₃C), 2.39 (1H, d, *J* 14.3, OCH₂CH*H'*), 2.90 (1H, app. t, 14.3, (OCH₂C*HH'*), 3.45 (1H, br s, NCH*H'*), 3.53 (1H, br s, NC*HH'*), 4.48-4.57 (2H, m, OCH₂); δ_C (100 MHz, CDCl₃) 27.9, 28.0, 35.3, 40.2, 75.3, 114.9, 126.4; HRMS (ESI⁺) found 240.0670, C₉H₁₅NNaO₃S (MNa⁺) requires 240.0665. Also obtained was **6-(***tert***-butyl)-7-methylidene-3-oxa-2-thia-1-azabicyclo[4.1.0]heptane 2,2-dioxide (18**) (31 mg, 13%). R_f 0.26 (petrol/ether, 2:1); v_{max} (thin film)/cm⁻¹ 3298 br, 2972 s, 2875 m, 1754 s, 1626 m, 1468 s, 1371 s, 1187 s; δ_H (500 MHz, CDCl₃) 1.06 (9 H, s, (CH₃)₃C), 2.11 (1 H, dt, *J* 15.2, 3.4) and 2.49 (1 H, ddd, 15.2, 10.8, 5.3, CH₂CH₂O), 4.36 (1 H, ddd, *J* 12.0, 10.8, 3.4) and 4.43 (1 H, ddd, *J* 12.0, 5.3, 3.4, CH₂O), 5.04 and 5.36 (2 × 1 H, 2 × d, *J* 2.8, CH₂=); δ_C (125 MHz, CDCl₃) 21.3, 25.5, 34.9, 66.7, 67.3, 90.0, 133.2; HRMS (ESI⁺) found 240.0661, C₉H₁₅NNaO₃S (MNa⁺) requires 240.0665.

(1S*,7S*,8S*)-7-Ethyl-8-methyl-3,3-dioxido-4-oxa-3-thia-2-azabicyclo[5.1.0]octan-1-yl acetate 20

The reaction of 3-ethylhexa-3,4-dienyl sulfamate (**19**) according to General Procedure A and purification by column chromatography (petrol/ether, 2:1) afforded the *title compound* (170 mg, 26%) as a white crystalline solid. $R_f 0.38$ (ether); m.p. 104 °C; v_{max} (thin film)/cm⁻¹ 3299 br, 2975 s, 1741 s, 1411 s, 1357 s, 1256 m, 1189 s; δ_H (500 MHz, CDCl₃) 0.99 (3 H, d, *J* 6.5, *CH*₃CH), 1.00 (3 H, t, *J* 7.6 *CH*₃CH₂), 1.40 (1 H, q, *J* 6.5, *CHC*(OAc)), 1.50–1.58 (1 H, m, *CH*H'CH₃), 1.65–1.75 (2 H, m, *CH*H'CH₂O overlays CH*H*'CH₃), 2.13 (3 H, s, CH₃CO), 2.50 (1 H, dd, *J* 16.2, 4.6, CH*H*'CH₂O), 4.23 (1 H, ddd, *J* 12.0, 4.6, 2.0) and 4.63 (1 H, app. t, *J* 12.0, CH₂O), 6.64 (1 H, br s, NH); δ_C (125 MHz, CDCl₃) 8.1, 10.2, 18.4, 20.9, 33.2, 33.4, 36.0, 69.2, 71.8, 171.5; HRMS (ESI⁺) found 286.0716, C₁₀H₁₇NNaO₅S (MNa⁺) requires 286.0720. Also obtained was **1-(5-ethyl-2,2-dioxido-6,7-dihydro-3***H***-1,2,3-oxathiazepin-4-yl)ethyl acetate (21)** (180 mg, 28%) as a white crystalline solid. R_f 0.30 (ether); v_{max} (thin film)/cm⁻¹ 3281 br, 2975 s, 2259 s, 1734 s, 1371 s; δ_H (500 MHz, CDCl₃) 1.04 (3 H, t, *J* 7.6, CH₃CH₂), 1.40 (3 H, d, *J* 6.6, CH₃CH), 2.05 (3 H, s, CH₃CO), 2.13–2.19 (1 H, m) and 2.25–2.32 (1 H, m, CH₂CH₃), 2.48 (1 H, ddd, *J* 16.2, 6.8, 1.7) and 2.74 (1 H, ddd, *J* 16.2, 8.4, 2.7, CH₂CH₂O), 4.34–4.42 (2 H, m, CH₂O), 5.74 (1 H, q, *J* 6.7, CHOAc), 6.23 (1 H, br s, NH); δ_C (125 MHz, CDCl₃) 12.3, 18.4, 21.1, 26.9, 33.7, 36.4, 70.5, 128.6, 136.2, 169.9; HRMS (ESI⁺) found 286.0717, C₁₀H₁₇NNaO₅S (MNa⁺) requires 286.0720.

7-(tert-Butyl)-4-oxa-3-thia-2-azabicyclo[5.1.0]octan-1-ol 3,3-dioxide 26

To a stirred solution of methylene aziridine **17** (47 mg, 0.210 mmol) in DMF (1.5 mL) was added NaI (32 mg, 0.210 mmol) at RT. After 20 h the reaction was quenched with sat. aq. NH₄Cl solution (5 mL) and the mixture was extracted with ether (3 × 20 mL). The combined extracts were washed with brine (10 mL), dried over MgSO₄ and concentrated *in vacuo*. Column chromatography (petrol/ether, 2:1) afforded the *title compound* as a white crystalline solid (20 mg, 41%). R_f 0.12 (petrol/ethyl acetate, 2:1); m.p. 132 °C; v_{max} (thin film)/cm⁻¹ 3460 s, 3180 br, 2958 s, 1457 s, 1265 s, 1144 s; δ_H (400 MHz, CDCl₃) 0.75 (1 H, d, *J* 6.2, *CHH*'C(OH)), 1.12 (9 H, s, (CH₃)₃C), 1.47 (1 H, d, *J* 6.2, CH*H*'C(OH)), 1.67 (1 H, ddd, *J* 17.0, 11.2, 1.6) and 2.69 (1 H, dd, *J* 17.0, 5.3, *CH*₂CH₂O), 3.61 (1 H, br s, OH), 4.27 (1 H, ddd, *J* 12.3, 5.3, 1.6) and 4.76 (1 H, app. t, *J* 11.8, CH₂O), 6.05 (1 H, br s, NH); δ_C (100 MHz, CDCl₃) 26.7, 29.8, 34.1, 36.3, 40.1, 71.1, 71.8; HRMS (ESI⁺) found 258.0769, C₉H₁₇NNaO₄S (MNa⁺) requires 258.0770.

7-Methyl-4-oxa-3-thia-2-azabicyclo[5.1.0]octane 3,3-dioxide 27

To a stirred solution of cyclopropane **10** (78 mg, 0.33 mmol) in isopropanol (2 mL) was added NaBH₄ (50 mg, 1.32 mmol) at 0 °C and the reaction mixture was allowed to warm to RT. After 20 h the reaction was quenched with water (5 mL) and extracted with dichloromethane (3 × 20 mL). The combined organic extracts were washed with brine (10 mL), dried over MgSO₄ and concentrated *in vacuo*. Column chromatography (petrol/ethyl acetate, 4:1) afforded the *title compound* as a white solid (36 mg, 62%). R_f 0.28 (petrol/ethyl acetate, 2:1); m.p. 91 °C; v_{max} (thin film)/cm⁻¹ 3300 br, 2961 s, 1338 s, 1163 s; δ_H (500 MHz, CDCl₃) 0.83 (1 H, dd, *J* 5.5, 3.7) and 0.93 (1 H, dd, *J* 7.3, 5.5, CH₂CH), 1.17 (3 H, s, CH₃), 2.00 (1 H, ddd, *J* 16.2, 11.2, 1.4) and 2.21 (1 H, dd, *J* 16.2, 4.7, CH₂CH₂O), 2.45 (1 H, ddd, *J* 7.3, 3.7, 2.0, CHNH), 4.20 (1 H, ddd, *J* 12.3, 4.7, 1.4) and 4.69 (1 H, app. t, *J* 11.7, CH₂O), 5.16 (1 H, br s, NH); δ_C (125 MHz, CDCl₃) 22.2, 24.2, 24.7, 34.9, 37.8, 69.8; *m/z* (CI) 195 (MNH₄⁺, 100%); HRMS (CI) found 195.0795, C₆H₁₅NO₃S (MNH₄⁺) requires 195.0803.

7-Methyl-4-oxa-3-thia-2-azabicyclo[5.1.0]octan-1-ol 3,3-dioxide 28

To a solution of cylopropane **10** (45 mg, 0.191 mmol) in ether (6 mL) at RT was added LiAlH₄ (22 mg, 0.574 mmol) and the mixture was stirred for 1 h. The reaction was quenched by the addition of water (10 mL) and acidified with 1 M hydrochloric acid (5 mL). The mixture was extracted with ether (3 × 20 mL) and the combined organic extracts were washed with brine (10 mL), dried over MgSO₄ and concentrated *in vacuo*. Purification by column chromatography (petrol/ethyl acetate, 3:1) afforded the *title compound* as a white solid (28 mg, 76%). R_f 0.34 (petrol/ethyl acetate, 2:1); m.p. 92 °C; v_{max} (thin film)/cm⁻¹ 3259 br, 2961 s, 1710 w, 1421 s, 1266 s, 1120 s, 1160 s; δ_H (400 MHz, CDCl₃) 0.99 (1 H, d, *J* 6.6) and 1.06 (1 H, d, *J* 6.6, CH₂C(OH)), 1.30 (3 H, s, CH₃), 1.86 (1 H, ddd, *J* 16.2, 11.6, 1.5) and 2.26 (1 H, dd, *J* 16.2, 4.6, CH₂CH₂O), 3.72 (1 H, br s, OH), 4.25 (1 H, ddd, *J* 11.8, 4.6, 1.5) and 4.63 (1 H, app. t, *J* 11.8, CH₂O), 6.14 (1 H, br s, NH); δ_C (100 MHz, CDCl₃) 17.2, 29.2, 29.4, 38.7, 68.5, 69.8; HRMS (ESI⁺) found 216.0300, C₆H₁₁NNaO₄S (MNa⁺) requires 216.0301.

1-(Ethoxy)-7-methyl-4-oxa-3-thia-2-azabicyclo[5.1.0]octane 3,3-dioxide 29

To a stirred solution of cyclopropane **10** (43 mg, 0.183 mmol) in ethanol (15 mL) at RT was added NaOH (146 mg, 3.66 mmol); after 30 min TLC analysis showed the reaction to be complete. The reaction mixture was diluted with water (20 mL), acidified with 1 M hydrochloric acid and extracted with dichloromethane (3 × 30 mL). The combined extracts were dried over Na₂SO₄ and concentrated *in vacuo*. Purification by column chromatography (petrol/ethyl acetate, 2:1) afforded the *title compound* as a white crystalline solid (33 mg, 82%). R_f 0.15 (petrol/ethyl acetate, 2:1); m.p. 126 °C; v_{max} (thin film)/cm⁻¹ 3708 br, 2981 s, 2844 m, 1417 m, 1370 s, 1213 m, 1166 m; δ_H (400 MHz, CDCl₃) 0.89 (1 H, d, *J* 5.7) and 0.92 (1 H, d, *J* 5.7, CH₂C(OEt)), 1.23 (3 H, t, *J* 7.0, CH₃CH₂), 1.32 (3 H, s, CH₃C), 1.87 (1 H, ddd, *J* 16.2, 11.0, 2.0) and 2.29 (1 H, ddd, *J* 16.2, 5.0, 1.1, CH₂CH₂O), 3.41 (1 H, dq, *J* 9.0, 7.0) and 3.84 (1 H, dq, *J* 9.0, 7.0, CH₂CH₃), 4.21 (1 H, ddd, *J* 12.3, 5.0, 2.0) and 4.64 (1 H, ddd, *J* 12.0, 11.0, 1.1, CH₂O), 5.93 (1 H, br s, NH); δ_C (100 MHz, CDCl₃) 15.1, 18.0, 29.2, 30.9, 39.1, 61.9, 69.2, 72.8; HRMS (ESI⁺) found 244.0615, C₈H₁₅NNaO₄S (MNa⁺) requires 244.0614.

1-Ethyl-7-methyl-4-oxa-3-thia-2-azabicyclo[5.1.0]octane 3,3-dioxide 30

To a stirred solution of cyclopropane **10** (70 mg, 0.297 mmol) in THF (10 mL) at 0 °C was added ethylmagnesium bromide (0.89 mL of a 1.0 M solution in THF, 0.89 mmol). The reaction mixture was allowed to warm to RT and stirring was continued for 20 h. The reaction was quenched with sat. aq. NH₄Cl solution (10 mL) and extracted with ether (3 × 20 mL). The combined organic extracts were washed with brine (10 mL), dried over MgSO₄ and concentrated *in vacuo*. Purification by column chromatography (petrol/ethyl acetate, 5:1) afforded the *title compound* as a white crystalline solid (42 mg, 68%). R_f 0.48 (petrol/ethyl acetate, 2:1); m.p. 88 °C; v_{max} (thin film)/cm⁻¹ 3299 br, 2962 s, 1421 s, 1332 s, 1164 s, 1105 m; δ_H (500 MHz, CDCl₃) 0.57 (1 H, d, *J* 5.5) and 0.80 (1 H, dd, *J* 5.5, 1.9, CH₂C(Et)), 0.94–1.01 (1 H, m, CHH'CH₃), 1.04 (3 H, t, *J* 6.9, CH₃CH₂), 1.24 (3 H, s, CH₃C), 2.03 (1 H, ddd, *J* 16.1, 11.0, 1.6) and 2.27 (1 H, dd, *J* 16.1, 4.7, CH₂CH₂O), 2.34–2.42 (1 H, m, CHH'CH₃), 4.19 (1 H, ddd, *J* 12.0, 4.7, 1.6) and 4.57 (1 H, dd, *J* 12.0, 11.0, CH₂O), 5.13 (1 H, br s, NH); δ_C (125 MHz, CDCl₃) 10.0, 19.3, 25.7, 29.3, 29.8, 40.5, 44.0, 69.0; HRMS (ESI⁺) found 228.0667, C₈H₁₅NNaO₃S (MNa⁺) requires 228.0665.

Ethyl 2-(7-methyl-3,3-dioxido-4-oxa-3-thia-2-azabicyclo[5.1.0]octan-1-yl)-3-oxobutanoate 31

To a stirred solution of ethyl acetoacetate (28 mg, 0.212 mmol) in THF (2 mL) at 0 °C was added NaH (9 mg of a 60% suspension in mineral oil, 0.212 mmol). After 15 min cyclopropane **10** (30 mg, 0.106 mmol) was added and the solution was allowed to warm to RT and stirred for 18 h. The reaction was quenched with water (2 mL), acidified with 1 M hydrochloric acid (0.5 mL) and extracted with ether (3 × 5 mL). The combined organic extracts were dried over MgSO₄ and concentrated *in vacuo*. Purification by column chromatography (petrol/ether, 1:1) afforded the *title compound* as a mixture of inseparable diastereomers A and B (26 mg, 77%, A:B = 2.4:1). R_f 0.16 (petrol/ether, 1:1); v_{max} (thin film)/cm⁻¹ 3301 br, 2983 s, 1716 s, 1619 m, 1414 s, 1178 s; δ_H (400 MHz, CDCl₃) 0.69 (1 H, d, *J* 6.9, C*H*H'C(EAA), A), 0.88 (1 H, d, *J* 6.6, C*H*H'C(EAA), B), 1.29 (3 H, s, CH₃C, B), 1.29 (3 H, s, CH₃C, A), 1.30 (3 H, t, *J* 7.1, CH₃CH₂, B), 1.31 (3 H, t, *J* 7.1, CH₃CH₂, A), 1.47 (1 H, d, *J* 6.6, CHH'C(EAA), B), 1.74 (1 H, d, *J* 6.9, CHH'C(EAA), A), 2.06–2.13 (1 H, m, CHH'CH₂O, A & B), 3.26 (1 H, d, *J* 1.0, CHCOCH₃, A), 3.64 (1 H, br s, CHCOCH₃, B), 4.07–4.15 (1 H, m, CHH'O, A & B), 4.19–4.28 (2 H, m, CH₂CH₃, A & B), 4.33–4.38 (1 H, m, CHH'O, A & B), 6.08 (1 H, br s, NH, B), 6.12 (1 H, br s, NH, A); δ_C (100 MHz, CDCl₃) data for A: 13.9, 22.0, 24.9, 28.7, 29.4, 37.1, 41.9, 62.1, 63.5, 67.2, 168.6, 202.4; HRMS (ESI⁺) found 328.0822, C₁₂H₁₉NNaO₆S (MNa⁺) requires 328.0825.

Diethyl 2-(7-methyl-3,3-dioxido-4-oxa-3-thia-2-azabicyclo[5.1.0]octan-1-yl)malonate 32

To a stirred solution of diethyl malonate (37 mg, 0.230 mmol) in THF (2 mL) at 0 °C was added NaH (9 mg of a 60% suspension in mineral oil, 0.230 mmol). After 15 min cyclopropane **10** (27 mg, 0.114 mmol) was added and the solution was allowed to warm to RT and stirred for 18 h. The reaction was quenched with water (2 mL), acidified with 1M hydrochloric acid (0.5 mL) and extracted with ether (3 × 5 mL). The combined extracts were dried over MgSO₄ and concentrated *in vacuo*. Purification by column chromatography (petrol/ether, 2:1) afforded the *title compound* as a

colourless oil (25 mg, 65%). R_f 0.20 (petrol/ether, 1:1); v_{max} (thin film)/cm⁻¹ 3313 br, 2984 s, 1729 s, 1391 s, 1369 s, 1313 s, 1238 s, 1175 s; δ_H (500 MHz, CDCl₃) 0.71 (1 H, d, *J* 6.3, *CH*H'C(DEM)), 1.30 (6 H, 2 × d, *J* 7.1, 2 × *CH*₃CH₂), 1.36 (1 H, app. s, CHH'C(DEM)), 1.37 (3 H, s, CH₃C), 2.11 (1 H, ddd, *J* 16.2, 8.8, 1.1) and 2.43 (1 H, ddd, *J* 16.2, 7.6, 1.0 *CH*₂CH₂O), 3.24 (1 H, s, *CH*(CO₂Et)₂), 4.12 (1 H, ddd, *J* 12.3, 7.6, 1.3, *CH*H'O), 4.19–4.42 (2 H, m) and 4.26–4.31 (2 H, m, 2 × *CH*₂CH₃), 4.43 (1 H, ddd, *J* 12.3, 8.8, 1.1, *CHH'O*), 6.24 (1 H, br s, NH); δ_C (100 MHz, CDCl₃) 13.8, 21.5, 27.2, 29.4, 38.5, 41.9, 56.1, 61.8, 67.7, 167.1, 168.7; HRMS (ESI⁺) found 358.0925, C₁₃H₂₁NNaO₇S (MNa⁺) requires 358.0931.



H1-(2)

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C13-(20)



WROCHEM.OX	NAME gf63292110 EXPNO PROCNO 20091022 Date_ 4.13 Time avc500 PROBHD 5 mm CPDUL 13C PULPROG 299930 PULPROG 29930 FULPROG 25536 SOLVENT 256 DS 31250.000 Hz SWH 0.476837 Hz FIDRES 1.0486259 sec 3G 1.0486259 sec 3G 16.000 usec	DE 20.00 usec TE 298.0 K D11 2.0000000 sec D11 0.03000000 sec TD0 1 ===== CHANNEL f1 ======= NUC1 8.00 usec P1 28.15752029 W PLIW 28.15752029 W SFO1 125.8131151 MHz	====== CHANNEL f2 ========= CPDPRG2 waltz16 NUC2 80.00 usec PL2 -6.00 dB PL13 15.1999991 W PL13 15.19999981 W PL12W 0.21970686 W PL13W 0.21970686 W PL13W 0.21970686 W SFO2 32768 NHz ST 125.8005438 MHz SFO2 32768 EM SFO2 32768 L SSB 1.00 Hz EM	GB PC 1.40
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C13-(21)



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VIIRe	ACCOLOR EXPNO FXOCNO Date INSTRUM PULPROBHD PULPROG PULPROG FULPROG SOLVENT SOLVENT SOLVENT AQ FIDRES AQ FULRES AQ FULRES AQ FULRES DI TE D1 D1 D1 D1 D1 D1 D1 D1 D1 D1 D1 D1 D1	TD0 TD0 NUC1 PL1 PL1 PL1 PL1W SF01 SF01 SF01 PL12W PL12W PL13 PL13W PL13W PL13W PL13W PL13W PL13W PL12W PL13W PL13W PL12W PL13W PL13W PL13W PL13W PL13W PL12W PL13W PL12W PL13W PL12W PL13W PL13W PL12W PL13W PL12W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13W PL13	E
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;500 east 20/8/09	84.581		180
Instrument AVC 5629 George Fé			500

C13-(26)



H1-(27)

HEM.OX	gf54770608 4 1 20090807 0.36 avc500 avc500 65536 65536 65536 65536 65536 65536 65536 31250.000 Hz	0.476837 Hz 1.0486259 sec 16.000 usec 20.00 usec 298.0 K 2.0000000 sec 0.0300000 sec	CHANNEL f1 ======== 13C 8.00 usec -4.40 dB 28.15752029 W 125.8131151 MHz	CHANNEL f2 ======= waltz16 1H 80.00 usec -6.00 dB 12.40 dB 12.19999981 W 0.21970686 W 0.21970686 W	500.3020012 MHz 32768 125.8005438 MHz EM 0 1.00 Hz 0 1.40	
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C13-(27)



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H1-(30)

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						PROBHD PULPROG TD SOLVENT NS DSS	5 mm CPDUL 13C zgpg30 65536 CDC13 2048 2048
						SWH FIDRES AQ DG DE	0.476837 Hz 0.476837 Hz 1.0486259 sec 16.000 usec 20.00 usec
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						======================================	CHANNEL f1 ======== 13C 8.00 usec -4.40 dB 28.15752029 W 125.8131151 MHz
						EEEEEEEC CPDFRG2 NUC2 PL2 PL2 PL112	CHANNEL f2 ======== waltz16 1H 80.00 usec -6.00 dB
						PL12 PL13 PL12W PL12W SF02 ST02	15.1999981 W 0.21970686 W 0.21970686 W 0.07618046 W 500.3020012 MHz 32768
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H1-(31)

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C13-(31)



H1-(32)

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