Electronic Supplementary Information

Diastereoselective synthesis of 3-acetoxy-4-(3-aryloxiran-2-yl)azetidin-2-ones and their transformation into 3,4-oxolane-fused bicyclic β-lactams

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Contents

1H and 13C NMR spectra of all new compounds:

- Compound 3
- Compound 5a
- Compounds 9a,c-e
- Compounds 4a-e
- Compounds 11a-e
- Compound 12a
- Compounds 13a-b

Single crystal X-ray diffraction:

- Compounds 11b, 12a
Compound 3: $^1$H NMR
Compound 3: $^{13}$C NMR
Compound 5a: $^1$H NMR
Compound 5a: $^{13}$C NMR

[Chemical structure and spectrum image]
Compound 9a: $^1$H NMR
Compound 9a: $^{13}$C NMR
Compound 9c: $^1$H NMR
Compound 9c: $^{13}$C NMR
Compound 9d: $^1$H NMR
Compound 9d: $^{13}$C NMR
Compound 9e: $^1$H NMR
Compound 9e: $^{13}$C NMR
Compound 4a: $^1$H NMR
Compound 4a: $^{13}$C NMR
Compound 4b: $^1$H NMR
Compound 4b: $^{13}$C NMR
Compound 4c: $^1$H NMR
Compound 4c. $^{13}$C NMR
Compound 4d: $^1$H NMR
Compound 4d: $^{13}$C NMR
Compound 4e: $^1$H NMR
Compound 4e: $^{13}$C NMR

[Image of a chemical structure and NMR spectrum]

[Detailed chemical structure and peak assignments]
Compound 11a: $^1$H NMR
Compound 11a: $^{13}$C NMR
Compound 11b: $^1$H NMR
Compound 11b: $^{13}$C NMR
Compound 11c: $^1$H NMR
Compound 11c: $^{13}$C NMR
Compound 11d: $^1$H NMR

[Diagram of the NMR spectrum with peaks and assignments]
Compound 11d: $^{13}$C NMR
Compound 11e: $^1$H NMR
Compound 11e: $^{13}$C NMR
Compound 12a: $^1$H NMR
Compound 12a: $^{13}$C NMR
Compound 13a: $^1$H NMR
Compound 13a: $^{13}$C NMR

[Chemical structure diagram]

[Graph of NMR spectrum with chemical shifts]
Compound 13b: $^1$H NMR
Compound 13b: $^{13}$C NMR
Single crystal X-ray diffraction

For the structures of compounds 11b and 12a, X-ray intensity data were collected on an Agilent Supernova Dual Source (Cu at zero) diffractometer equipped with an Atlas CCD detector using CuKα radiation (λ = 1.54178 Å) and ω scans. The images were interpreted and integrated with the program CrysAlisPro (Agilent Technologies) [1]. Using Olex2 [2], the structure was solved by direct methods using the ShelXS structure solution program and refined by full-matrix least-squares on F² using the ShelXL program package [3]. Non-hydrogen atoms were anisotropically refined and the hydrogen atoms in the riding mode and isotropic temperature factors fixed at 1.2 times U(eq) of the parent atoms (1.5 times for methyl and hydroxyl groups).

CCDC 1400949-1400950 contain the supplementary crystallographic data for this paper and can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44-1223-336033; or deposit@ccdc.cam.ac.uk).

Crystal data for compound 11b. C₁₈H₁₇NO₄, M = 311.33, triclinic, space group P-1 (No. 2), a = 6.7690(4) Å, b = 17.4058(7) Å, c = 26.8238(9) Å, α = 94.767(3)°, β = 94.714(4)°, γ = 99.371(4)°, V = 3092.7(2) Å³, Z = 8, T = 100 K, ρcalc = 1.337 g cm⁻³, μ(Cu-Kα) = 0.781 mm⁻¹, F(000) = 1312, 28410 reflections measured, 12222 unique (Rint = 0.0660) which were used in all calculations. The final R1 was 0.0617 (I >2σ(I)) and wR2 was 0.1754 (all data). The asymmetric unit contains four crystallographic independent molecules.

Crystal data for compound 12a. C₁₄H₁₇NO₃, M = 247.29, orthorhombic, space group Pna₂₁ (No. 33), a = 16.3439(5) Å, b = 5.9484(2) Å, c = 12.9762(5) Å, V = 1261.55(8) Å³, Z = 4, T = 100 K, ρcalc = 1.302 g cm⁻³, μ(Cu-Kα) = 0.747 mm⁻¹, F(000) = 528, 6703 reflections measured, 2069 unique (Rint = 0.0391) which were used in all calculations. The final R1 was 0.0326 (I >2σ(I)) and wR2 was 0.0820 (all data). The crystal appeared to be racemically twinned, hence the structure shows a refined Flack parameter of 0.49(18).