Crystal Structure Analysis and Sublimation Thermodynamics of Bicyclo Derivatives of a Neuroprotector Family

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Supplementary Material

Synthesis Details and Spectroscopic Characterization

[3-Thia-1-aza-bicyclo[3.3.1]nonylidene]-(4-trifluoromethyl-phenyl)-amine (1)

Yield 73 %; mp 91-92 °C.


1H NMR (200MHz, CDCl3, d): 1.54 (1?, m, ?(6)? γ), 1.93 (3?, m, ?(6)? γ, ?(7)? γ), 2.51 (1?, dd, J = 1.9, 10.2 Hz, ?(5)? γ), 2.84 (1?, dd, J = 2.1, 12.6 Hz, ?(4)? γ), 3.23 (3?, m, ?(4)? γ, ?(8)? γ, ?(9)? γ), 3.66 (1?, dd, J = 2.3, 13.7 Hz, ?(8)? γ), 4.22 (1?, dd, J = 2.3, 12.8 Hz, ?(9)? γ), 6.96 (2H, d, J = 8.4, ? γ), 7.57 (2H, d, J = 8.4, ? γ).
[3-Thia-1-aza-bicyclo[3.3.1]nonylidene]- (4-fluoro-phenyl)-amine (2)

Yield 68%; mp 89-90 °C.


^1H NMR (200MHz, CDCl₃, d): 1.52 (1?, m, ?(6)?), 1.83 (3?, m, ?(6)? γ, ?(7)? δ), 2.47 (1?, dd, J = 1.9, 11.4 Hz, ?(5)?), 2.83 (1?, dd, J = 2.3, 12.8 Hz, ?(4)? γ), 3.22 (3?, m, ?(4)? γ, ?(8)? γ, ?(9)? γ), 3.65 (1?, dd, J = 2.0, 13.7 Hz, ?(8)? γ), 4.20 (1?, dd, J = 2.3, 13.0 Hz, ?(9)? γ), 6.83 (2H, dd, J = 5.0, 8.6, ? γ), 7.03 (2H, t, J = 8.6, ? γ).

[3-Thia-1-aza-bicyclo[3.3.1]nonylidene]-p-tolyl-amine (3)

Yield 70%; mp 74-75 °C.

MS (ESI), m/z: 247 (MH+). Anal. Calcd. for C_{14}H_{18}N_{2}S: ? 68.25%, H 7.36%, N 11.37%. Found: ? 68.18%, H 7.12%, N 11.22%.

^1H NMR (200MHz, CDCl₃, d): 1.51 (1?, m, ?(6)? γ), 1.83 (3?, m, ?(6)? γ, ?(7)? δ), 2.36 (3H,c,CH₃), 2.42 (1?, dd, J = 2.0, 11.4 Hz, ?(5)?), 2.82 (1?, dd, J = 2.1, 12.7 Hz, ?(4)? γ), 3.21 (3?, m, ?(4)? γ, ?(8)? γ, ?(9)? γ), 3.66 (1?, dd, J = 2.1, 13.7 Hz, ?(8)? γ), 4.21 (1?, dd, J = 2.3, 13.3 Hz, ?(9)? γ), 6.79 (2H, d, J = 8.0, ? γ), 7.03 (2H, d, J = 8.0, ? γ).