

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

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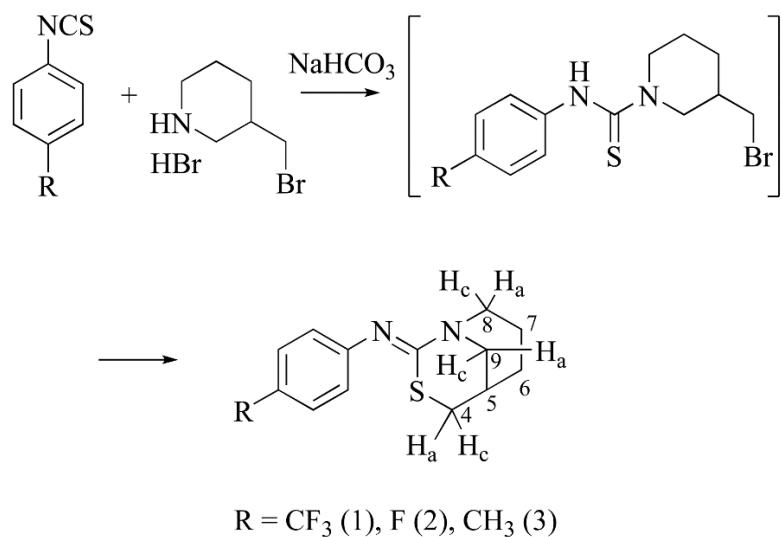
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Supplementary Material

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.

MS (ESI), m/z: 301 (MH⁺). Anal. Calcd. for C₁₄H₁₅F₃N₂S: ? 55.99%, H 5.03%, N 9.33%. Found: ? 56.08%, H 4.94%, N 9.24%.

¹H NMR (200MHz, CDCl₃, d): 1.54 (1?, m, ?(6)??), 1.93 (3?, m, ?(6)??, ?(7)?2), 2.51 (1?, dd, J = 1.9, 10.2 Hz, ?(5)?), 2.84 (1?, dd, J = 2.1, 12.6 Hz, ?(4)??), 3.23 (3?, m, ?(4)??, ?(8)??, ?(9)??), 3.66 (1?, dd, J = 2.3, 13.7 Hz, ?(8)??), 4.22 (1?, dd, J = 2.3, 12.8 Hz, ?(9)??), 6.96 (2H, d, J = 8.4, ??), 7.57 (2H, d, J = 8.4, ??).

[3-Thia-1-aza-bicyclo[3.3.1]nonylidene]- (4-fluoro-phenyl)-amine (2)

Yield 68 %; mp 89-90 °C.

MS (ESI), m/z: 251 (MH⁺). Anal. Calcd. for C₁₃H₁₅FN₂S: ? 62.37%, H 6.04%, N 11.19%. Found: ? 62.18%, H 5.94%, N 11.26%.

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

[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 C₁₄H₁₈N₂S: ? 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)? ?), 1.83 (3? , m, ?(6)? ?, ?(7)? ?), 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)? ?), 3.21 (3? , m, ?(4)? ?, ?(8)? ?, ?(9)? ?), 3.66 (1? , dd, J = 2.1, 13.7 Hz, ?(8)? ?), 4.21 (1? , dd, J = 2.3, 13.3 Hz, ?(9)? ?), 6.79 (2H, d, J = 8.0, ? ?), 7.03 (2H, d, J = 8.0, ? ?).