

Regiodirected synthesis of hydroazolic compounds with the use of thiosemicarbazide

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2c: yield 68%; mp 190–191 °C (PrⁱOH). IR (n/cm⁻¹): 3390, 3360 (NH₂), 3200 (NH), 3120–3000 (=CH), 3165–3140 (CH_{Fur}), 2945–2864 (CH₂), 1665 (C=C), 1193 (C=S). ¹H NMR, d: 1.85 (s, 1H, NH), 2.10–2.31 (m, 6H, CH₂), 6.33 (s, 1H, C=C), 6.78–6.86 (m, 2H, Fur), 7.35 (d, 1H, Fur), 9.67 (s, 2H, NH₂). ¹³C NMR, d: 24.6, 25.1, 46.8 (3CH₂), 129.7 (=CH–Fur), 118.4 (C⁴_{Fur}), 115.1 (C³_{Fur}), 141.6 (C²_{Fur}), 149.1 (C⁵_{Fur}), 151.3 (=C–), 149.5 (C=N), 181.7 (C=S). Found, (%): C, 56.31; H, 5.53; N, 17.97. Calc. for C₁₁H₁₃N₃OS (%): C, 56.15; H, 5.57; N, 17.86.

2d: yield 55%, mp 175–177 °C (PrⁱOH). IR (n/cm⁻¹): 3350, 3280 (NH₂), 3150 (NH), 3100–3012 (=CH), 3195–3130 (CH_{Fur}), 2935–2856 (CH₂), 1671 (C=C), 1195 (C=S). ¹H NMR, d: 1.75 (s, 1H, NH), 2.05–2.41 (m, 10H, CH₂), 6.31 (s, 1H, C=C), 6.79–6.87 (m, 2H, Fur), 7.41 (d, 1H, Fur), 10.17 (s, 2H, NH₂). ¹³C NMR, d: 23.2, 24.9, 25.9, 26.4, 49.1 (5CH₂), 129.9 (=CH–Fur), 128.3 (C⁴_{Fur}), 116.2 (C³_{Fur}), 141.1 (C²_{Fur}), 148.2 (C⁵_{Fur}), 156.4 (=C–), 150.9 (C=N), 184.3 (C=S). Found (%): C, 58.97; H, 6.74; N, 15.95. Calc. for C₁₃H₁₇N₃OS (%): C, 59.29; H, 6.51; N, 15.96.

3d: yield 35% (procedure I), 55% (procedure II); mp 195–197 °C (PrⁱOH). IR (n/cm⁻¹): 3291, 3299 (NH₂), 3028–3019 (=CH), 2879 (CH₂, CH), 1605 (C=N), 1200 (C=S). ¹H NMR, d: 2.16–2.46 (m, 10H, CH₂), 2.41 (s, 1H, CH), 6.35 (s, 1H, CH–N), 6.69–6.82 (m, 2H, Fur), 7.45 (d, 1H, Fur), 10.15 (s, 2H, NH₂). ¹³C NMR, d: 24.1, 25.2, 26.1, 26.4, 43.1 (5 CH₂), 46.7 (CH), 54.7 (C–N), 107.4 (C⁴_{Fur}), 107.2 (C³_{Fur}), 140.8 (C²_{Fur}), 147.2 (C⁵_{Fur}), 156.4 (=C–), 150.9 (C=N), 184.3 (C=S). Found (%): C, 58.97; H, 6.74; N, 15.95. Calc. for C₁₃H₁₇N₃OS (%): C, 59.29; H, 6.51; N, 15.96.

4d: yield 56%, mp 152–155 °C (EtOH). IR (n/cm⁻¹): 3480 (NH), 1685 (Amide I), 1600 (C=N), 1490 (Amide II), 1626 (C=C). ¹H NMR, d: 1.32–2.22 (m, 10H, CH₂), 1.75

(s, 3H, COMe), 2.25 (s, 3H, COMe), 5.9 (s, 1H, C=C), 6.77–6.85 (m, 2H, -Fur), 7.3 (d, 1H, -Fur), 8.9 (s, 1H, NH). ¹³C NMR, δ: 26.8, 28.7, 28.6, 31.4, 56.2 (5CH₂), 84.7 (C_{spiro}), 38.1 (Me), 45.4 (Me), 127.2 (=CH-Fur), 128.6 (C⁴_{Fur}), 113.9 (C³_{Fur}), 112.1 (C²_{Fur}), 148.9 (C⁵_{Fur}), 139.9 (C=N), 145.9 (=C-), 175.9 (C=O), 177.1 (C=O). Found (%): C, 58.34; H, 6.01; N, 12.01. Calc. for C₁₈H₂₁N₃O₃S (%): C, 58.77; H, 6.09; N, 12.09.