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Non-catalytic thermal multicomponent assembling of isatin, cyclic CH-acids and malononitrile: an efficient approach to spirooxindole scaffold

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*7'-Amino-1',3'-dimethyl-2,2',4'-trioxo-1,1',2,2',3',4'-hexahydrospiro[indole-3,5'-pyrano[2,3-*d*]pyrimidine]-6'-carbonitrile* **3a**.

White solid; yield 1.67 g (95%); mp 229–230 ºC (lit.[1] mp 230 ºC).

^{1}H NMR (300 MHz, [D_6]DMSO): δ = 3.00 (s, 3H, CH_3), 3.36 (s, 3H, CH_3), 6.78 (d, J = 7.3 Hz, 1H, Ar), 6.88 (t, J = 7.3 Hz, 1H, Ar), 7.07–7.17 (m, 2H, Ar), 7.55 (s, 2H, NH_2), 10.48 (s, 1H, NH) ppm.

2-Amino-2',5-dioxo-1',2',5,6,7,8-hexahydrospiro[chromene-4,3'-indole]-3-carbonitrile **3b**.

White solid; yield 1.44 g (94%); mp 311–312 ºC (lit.[2] mp 312–313 ºC).

^{1}H NMR (300 MHz, [D_6]DMSO): δ = 1.91–2.00 (m, 2H, CH_2), 2.10–2.30 (m, 2H, CH_2), 2.62–2.73 (m, 2H, CH_2), 6.78 (d, J = 7.3 Hz, 1H, Ar), 6.88 (t, J = 7.3 Hz, 1H, Ar), 7.00 (d, J = 7.3 Hz, 1H, Ar), 7.14 (t, J = 7.3 Hz, 1H, Ar), 7.23 (s, 2H, NH_2), 10.40 (s, 1H, NH) ppm.

2-Amino-7,7-dimethyl-2',5-dioxo-1',2',5,6,7,8-hexahydrospiro[chromene-4,3'-indole]-3-carbonitrile **3c.**

White solid; yield 1.63 g (97%); mp 305–306 ºC (lit.[2] mp 305−307 ºC).

^{1}H NMR (300 MHz, [D_6]DMSO): δ = 1.00 (s, 3H, CH_3), 1.03 (s, 3H, CH_3), 2.08–2.19 (m, 2H, CH_2), 2.56 (s, 2H, CH_2), 6.78 (d, J = 7.3 Hz, 1H, Ar), 6.89 (t, J = 7.3 Hz, 1H, Ar), 6.97 (d, J = 7.3 Hz, 1H, Ar), 7.14 (t, J = 7.3 Hz, 1H, Ar), 7.23 (s, 2H, NH_2), 10.40 (s, 1H, NH) ppm.

*6'-Amino-3'-methyl-2-oxo-1,2-dihydro-1'*H*-spiro[indole-3,4'-pyrano[2,3-*c*]pyrazole]-5'-carbonitrile* **3d**.

White solid; yield 1.33 g (91**%**); mp 277–278 ºC (lit. [3] mp 278–280 ºC).

^{1}H NMR (300 MHz, [D_6]DMSO): δ = 1.52 (s, 3H, CH_3), 6.90 (d, J = 7.3 Hz, 1H, Ar), 6.95–7.05 (m, 2H, Ar), 7.17–7.27 (m, 3H, Ar, NH_2), 10.58 (s, 1H, NH), 12.28 (s, 1H, NH) ppm.

*6′-Amino-3′-methyl-2-oxo-1′-phenyl-1,2-dihydro-1′*H*-spiro[indole-3,4′-pyrano[2,3-*c*]pyrazole]-5′-carbonitrile* **3e**.

White solid; yield 1.70 g (92%); mp 235–237 ºC (lit.[3] mp 236–237 ºC).

^{1}H NMR (300 MHz, [D_6]DMSO): δ = 1.54 (s, 3H, CH_3), 6.94 (d, J = 7.3 Hz, 1H, Ar), 7.03 (t, J = 7.3 Hz, 1H, Ar), 7.18 (d, J = 7.3 Hz, 1H, Ar), 7.28 (t, J = 7.3 Hz, 1H, Ar), 7.35 (d, J = 7.7 Hz, 1H, Ar), 7.52 (t, J = 7.7 Hz, 2H, Ar), 7.57 (s, 2H, NH_2), 7.78 (d, J = 7.7 Hz, 2H, Ar), 10.74 (s, 1H, NH) ppm.

*2′-Amino-7′-methyl-2,5′-dioxo-1,2-dihydro-5′*H*-spiro[indole-3,4′-pyrano[4,3-*b*]pyran]-3′-carbonitrile* **3f**.

White solid; yield 1.44 g (90%); mp 297–299 ºC (lit.[4] mp 262–263 ºC).

^{1}H NMR (300 MHz, [D_6]DMSO): δ = 2.24 (s, 3H, CH_3), 6.34 (s, 1H, CH), 6.83 (d, J = 8.1 Hz, 1H, Ar), 6.93 (t, J = 7.7 Hz, 1H, Ar), 7.10 (d, J = 7.3 Hz, 1H, Ar), 7.19 (t, J = 7.3 Hz, 1H, Ar), 7.42 (s, 2H, NH_2), 10.57 (s, 1H, NH) ppm.

*2′-Amino-2,5′-dioxo-1,2-dihydro-5′*H*-spiro[indole-3,4′-pyrano[3,2-*c*]chromene]-3′-carbonitrile* **3g**.

White solid; yield 1.62 g (91%); mp 307–308 ºC (lit.[4] mp 283–285 ºC).

^{1}H NMR (300 MHz, [D_6]DMSO): δ = 6.86 (d, J = 7.7 Hz, 1H, Ar), 6.94 (t, J = 7.3 Hz, 1H, Ar), 7.21 (d, J = 7.7 Hz, 1H, Ar), 7.22 (t, J = 7.3 Hz, 1H, Ar), 7.48 (d, J = 8.4 Hz, 1H, Ar), 7.53 (t, J = 7.7 Hz, 1H, Ar), 7.66 (s, 2H, NH_2), 7.76 (t, J = 7.7 Hz, 1H, Ar), 7.95 (d, J = 7.7 Hz, 1H, Ar), 10.68 (s, 1H, NH) ppm.

References

1. M. N. Elinson, A. I. Ilovaisky, V. M. Merkulova, D. V. Demchuk, P. A. Belyakov, Y. N. Ogibin and G. I. Nikishin, *Electrochim. Acta,* 2008, **53**, 8346.
2. M. N. Elinson, A. I. Ilovaisky, A. S. Dorofeev, V. M. Merkulova, N. O. Stepanov, F. M. Miloserdov, Yu. N. Ogibin and G. I. Nikishin, *Tetrahedron,* 2007, **63**, 10543.
3. M. N. Elinson, A. S. Dorofeev, F. M. Miloserdov and G. I. Nikishin, *Mol. Diversity,* 2009, **13**, 47.
4. L. M. Wang, N. Jiao, J. Qiu, J. J. Yu, J. Q. Liu, F. L. Guo and Y. Liu, *Tetrahedron*, 2010, **66**, 339.