New approach to the synthesis of benzo[c][1,7]naphthyridin-4(3H)-ones

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Experimental

General Information. The \textsuperscript{1}H and \textsuperscript{13}C NMR spectra of the investigated compounds were recorded on a JEOL JNM-ECA-400 or a Bruker DRX-400 instruments (400 and 100 MHz, respectively) with TMS as internal standard. \textsuperscript{13}C NMR signals were assigned by using the APT (Attached Proton Test). The IR spectra were recorded on an INFRAUM FT-801 spectrometer. The reaction course and purity of the products were checked by thin-layer chromatography on Sorbfil UV-254 plates. Melting points were determined on a Kofler bench. Elemental analysis was performed with a Carlo Erba 1106 CHN analyzer. Compounds 5,6 were prepared as previously reported\textsuperscript{1b}. Aldehydes 11a-j were commercially available and used as received. PPA was prepared directly before the reactions from 6 g P2O5 and 4 ml 80% PA.

General Procedure for the Synthesis of Compounds 7, 8. A solution of benzyol chloride (170 mg; 1.2 mmol) in 1 ml of anhydrous chloroform was added dropwise to a solution of 3-aminopyridine 5, 6 (1.0 mmol) and anhydrous pyridine (120 mg, 1.5 mmol) in absolute chloroform (5 ml) with stirring and ice cooling. The mixture was stirred for 0.5 h with cooling in ice and for 2 h at room temperature. The solvent was evaporated, the residue triturated with H2O, and recrystallized from a mixture of solvents: ethanol-water.

The Synthesis of 6-Aryl-2-methylbenzo[c][1,7]-naphthyridin-4(3H)-one 12 a-d. (General Procedure A). 3-Aminopyridin-2(1H)-one 6 (260 mg, 1 mmol) and aldehyde 11 a,b,c,d (1.5 mmol) in 1.0 ml 80% trifluoroacetic acid was refluxed for 4 h. Upon cooling reaction mixture was poured on a crushed ice and neutralized by solution of 1N NaOH. The solid was filtered off and recrystallized from the mixture of solvents: 2-propanol-1,4-dioxane = 2:1.

The Synthesis of 6-Aryl-2-methylbenzo[c][1,7]-naphthyridin-4(3H)-one 12 a-h. (General Procedure B). 3-Aminopyridin-2(1H)-one 5, 6 (1 mmol) and aldehyde 11 a-h (1.5 mmol) in 1 ml PPA was heated at 110-130°C for 4-11 h. Upon cooling the mixture was poured on a crushed ice and neutralized by 1N NaOH. The solid was filtered off and recrystallized from the mixture of solvents: 2-propanol-1,4-dioxane = 2:1.

The Synthesis of 6-Aryl-2-methylbenzo[c][1,7]-naphthyridin-4(3H)-one 12 a-h. (General Procedure C). 3-Aminopyridin-2(1H)-one 5, 6 (1 mmol) and aldehyde 11 a-i (1.5 mmol) in 1.0 ml 80% phosphoric acid was heated at 110-130°C for 10-12 h. Upon cooling the mixture was poured on a crushed ice and neutralized by 1N NaOH. The solid was filtered off and recrystallized from the mixture of solvents: 2-propanol-1,4-dioxane, 2:1.

Spectroscopic and physical data
N-(6-methyl-2-oxo-4-phenyl-1,2-dihydropyridin-3-yl)benzamide (7)
Yield: 246 mg (81%); white crystals, m.p. 276-278°C (ethanol-water, 2:1).
IR (KBr), ν, cm⁻¹: 3320 (N-H), 1643, 1511 (C(O)NH).

1H NMR spectrum, δ, ppm (400 MHz, DMSO-d6): 2.23 (s, 3H, CH₃); 6.06 (s, 1H, H-5); 7.29-7.38 (m, 3H, H-3', 4', 5' Ph); 7.39-7.50 (m, 5H, H2,3,4,5,6 Ph); 7.79 (d, 2H, J = 5.8 Hz, H-2', 6' Ph); 9.38 (s, 1H, N-H Bz); 11.92 (bs, 1H, N-H).

13C NMR (DMSO-d6): δ: 18.4, 105.6, 121.6, 127.5, 127.8, 128.1, 128.2, 131.3, 134.3, 137.7, 143.3, 149.4, 161.1, 166.0.

N-[4-(3,4-dimethoxyphenyl)-6-methyl-2-oxo-1,2-dihydropyridin-3-yl]benzamide (8)
Yield: 247 mg (68%); white crystals, m.p. 234-236°C (ethanol-water, 2:1).
IR (KBr), ν, cm⁻¹: 1614, 3295.

1H NMR spectrum, δ, ppm (400 MHz, DMSO-d6): 2.23 (s, 3H, CH₃); 3.63 (s, 3H, OCH₃); 3.72 (s, 3H, OCH₃); 6.10 (s, 1H, H-5); 6.94 (d, 1H, J = 8.4 Hz, H-5' Ar); 7.03 (d, 1H, J = 8.4 Hz, H-6' Ar); 7.10 (s, 1H, H-2' Ar); 7.45 (t, 2H, J = 7.4 Hz, H-3', 5' Ph); 7.53 (t, 1H, J = 7.1 Hz, H-4' Ph); 7.86 (d, 2H); 3.97 (s, 1H, N-H Bz); 11.84 (bs, 1H, N-H).

13C NMR spectrum, δ, ppm (100 MHz): δ: 18.4, 55.3, 55.4, 105.6, 111.3, 111.6, 120.4, 121.5, 127.5, 128.3, 129.9, 131.4, 134.2, 143.0, 148.0, 148.9, 149.2, 161.2, 166.0.

5-methyl-2,7-diphenyloxazolo[5,4-b]pyridine (9) and 7-(3,4-dimethoxyphenyl)-5-methyl-2-phenyl[1,3]oxazolo[5,4-b]pyridine (10);

5-methyl-2,7-diphenyloxazolo[5,4-b]pyridine (9)
Yield: 115 mg (80%); white crystals, m.p. 114-115°C (ethanol). IR (KBr), ν, cm⁻¹: 1613, 3385.

1H NMR spectrum, δ, ppm (400 MHz, (CD₃)₂CO): 2.65 (s, 3H, CH₃); 7.48 - 7.59 (m, 4H, H-3', 4', 5', 4' Ph); 7.60 (s, 1H, H-6); 7.61-7.63 (m, 2H, H-2', 6' Ph); 8.25-8.29 (m, 4H, H-2'', 3'', 5'', 6'' Ph). 13C NMR spectrum, δ, ppm (100 MHz): 24.5, 119.2, 127.9, 128.5, 129.8, 129.9, 130.0, 130.2, 130.6, 133.1, 133.9, 141.1, 155.8, 161.4, 162.7.


7-(3,4-dimethoxyphenyl)-5-methyl-2-phenyl[1,3]oxazolo[5,4-b]pyridine (10)
Yield: 125 mg (72%); white crystals, m.p. 168-170°C (ethanol).
IR (KBr), ν, cm⁻¹: 1660, 3281.

1H NMR spectrum, δ, ppm (400 MHz, CDCl₃): 2.71 (s, 3H, CH₃); 3.97 (s, 3H, OCH₃); 4.05 (s, 3H, OCH₃); 7.03 (d, 1H, J = 8.4 Hz, H-5' Ar); 7.38 (s, 1H, H-6); 7.48 - 7.60 (m, 3H, H-3'', 4'', 5'' Ph); 7.78 (d, 1H, J = 8.4, 2.0 Hz, H-6' Ar); 7.95 (d, 1H, J = 2.0 Hz, H-2' Ar); 8.28 (d, 2H, J = 7.5, 2.0 Hz, H-2'', 6'' Ph).

13C NMR spectrum, δ, ppm (100 MHz): 24.4, 56.0, 111.2, 112.3, 117.3, 121.6, 126.9, 127.4, 127.6, 128.7, 128.9, 131.7, 140.0, 149.0, 150.3, 154.3, 160.4, 161.5.
Anal. Calcd for C₂₁H₁₆N₂O₃: C, 72.82; H, 5.24; N, 8.09. Found: C, 73.09; H, 5.03; N, 8.21.
**8,9-Dimethoxy-2-methyl-6-phenylbenzo[c][1,7]-naphthyridin-4(3H)-one (12a)**

Yield by method A 163 mg (47%), yield by method B 204 mg (59%), yield by method C 207 mg (60%); Pale yellow powder; mp 326-329 °C (ethanol-dioxane, 2:1). IR (KBr), ν, cm^{-1}: 1640, 3392;

^1^H NMR spectrum, δ, ppm (400 MHz, DMSO-d6): 2.37 (s, 3H, CH₃); 3.78 (s, 3H, OCH₃); 4.06 (s, 3H, OCH₃); 7.12 (s, 1H, H-1); 7.37 (s, 1H, H-10); 7.52-7.58 (m, 3H, H-3', 4', 5' Ph); 7.70 (dd, 2H, ^3^J = 7.8, ^4^J = 1.5 Hz, H-2', 6' Ph); 7.90 (s, 1H, H-7); 11.43 (bs, 1H, N-H).

^1^C NMR spectrum, δ, ppm (100 MHz, DMSO-d6): 18.6, 55.2, 55.9, 97.1, 103.7, 106.6, 122.1, 127.3, 127.8, 128.5, 129.3, 129.7, 132.8, 139.2, 140.0, 150.7, 152.3, 155.2, 161.0.

Anal. Calcd for C_{21}H_{18}N_{2}O_{3}: C, 72.82; H, 5.24; N, 8.09. Found: C, 72.67; H, 5.09; N, 8.21

**8,9-dimethoxy-6-(4-methoxyphenyl)-2-methylbenzo[c][1,7]-naphthyridin-4(3H)-one (12b)**

Yield by method C 181 mg (48%), pale yellow powder, mp 315-320 °C (2-propanol-1,4-dioxane, 2:1); IR (KBr). ν cm^{-1}: 1656, 3409;

^1^H NMR spectrum, δ, ppm (400 MHz, DMSO-d6): 2.36 (s, 3H, CH₃); 3.80 (s, 3H, OCH₃); 3.86 (s, 3H, OCH₃); 4.05 (s, 3H, OCH₃); 7.09-7.11 (m, 3H, H-1, H-3', 5' Ar); 7.43 (s, 1H, H-7); 7.66 (d, 2H, ^3^J = 8.6, H-2', 6' Ar); 7.88 (s, 1H, H-10); 11.43 (bs, 1H, N-H).

^1^C NMR spectrum, δ, ppm (100 MHz, DMSO-d6): 18.7, 55.0, 55.2, 56.0, 97.3, 103.7, 106.7, 113.5, 122.2, 127.3, 129.5, 130.8, 131.8, 139.7, 150.7, 152.2, 155.0, 159.3, 161.3.

Anal. Calcd for C_{22}H_{20}N_{2}O_{4}: C, 70.20; H, 5.36; N, 7.44. Found: C, 70.09; H, 5.12; N, 7.61

**8,9-dimethoxy-2-methyl-(pyridin-2-yl)benzo[c][1,7]naphthyridin-4(3H)-one (12c)**

Yield by method C 139 mg (40%), yellow powder, mp 340-342 °C (2-propanol-1,4-dioxane, 2:1). IR (KBr): 1640, 3415 cm^{-1}.

^1^H NMR spectrum, δ, ppm (400 MHz, DMSO-d6): 2.37 (s, 3H, CH₃); 3.85 (s, 3H, OCH₃); 4.06 (s, 3H, OCH₃); 7.15 (s, 1H, H-1); 7.52 (dd, 1H, ^3^J = 6.7 Hz, ^4^J = 4.5 Hz, H-5' Py); 7.89 (s, 1H, H-10); 8.02 (t, 1H, ^3^J = 8.1 Hz, H-4' Py); 8.14 (d, 1H, ^3^J = 8.1 Hz, H-3' Py); 8.39 (s, 1H, H-7); 8.76 (d, ^3^J = 4.1 Hz, H-6' Py); 11.51 (bs, 1H, N-H).

^1^C NMR spectrum, δ, ppm (100 MHz, DMSO-d6): 18.7, 55.3, 55.9, 97.4, 103.4, 107.5, 122.3, 123.1, 124.8, 127.7, 130.6, 132.7, 136.7, 140.6, 147.7, 150.7, 151.6, 152.1, 158.0, 161.1.

Anal. Calcd for C_{20}H_{17}N_{3}O_{3}: C, 69.15; H, 4.93; N, 12.10. Found: C, 69.39; H, 4.82; N, 12.21
8,9-dimethoxy-2-methyl-6-(pyridin-4-yl)benzo[c][1,7]naphthyridin-4(3H)-one (12d)

Yield by method C 115 mg (33%), yellow powder, mp. 322-324 °C (2-propanol-1,4-dioxane, 2:1). IR (KBr): 1641, 3484 cm⁻¹.

1H NMR spectrum, δ, ppm (400 MHz, DMSO-d6): 2.35 (s, 3H, CH₃); 3.84 (s, 3H, OCH₃); 4.05 (s, 3H, OCH₃); 7.20 (s, 1H, H-1); 7.30 (s, 1H, H-7); 7.62 (d, 2H, H = 3.7 Hz, H-3', 5' Py); 7.93 (s, 1H, H-10); 8.76 (d, H = 3.7 Hz, H-2', 6' Py); 11.65 (bs, 1H, N-H).

13C NMR spectrum, δ, ppm (100 MHz, DMSO-d6): 19.0, 56.1, 56.2, 97.7, 104.2, 107.3, 122.5, 124.4, 128.0, 130.9, 132.5, 140.8, 147.0, 149.6, 149.9, 152.3, 152.8, 161.5.

Anal. Calcd for C₂₀H₁₇N₃O₃: C, 69.15; H, 4.93; N, 12.10. Found: C, 69.51; H, 4.70; N, 12.35

6-(4-fluorophenyl)-8,9-dimethoxy-2-methylbenzo[c][1,7]naphthyridin-4(3H)-one (12e)

Yield by method C 168 mg (46%); pale yellow powder, mp. 336-338 °C (2-propanol-1,4-dioxane, 2:1). IR (KBr): 1632, 1658, 3404 cm⁻¹.

1H NMR spectrum, δ, ppm (400 MHz, DMSO-d6): 2.34 (s, 3H, CH₃); 3.80 (s, 3H, OCH₃); 4.06 (s, 3H, OCH₃); 7.18 (s, 1H, H-1); 7.23-7.28 (m, 2H, H-3', 5' Ar); 7.65 (s, 1H, H-7); 7.60 (dd, 2H, H = 8.2, 6' Ar); 7.79 (s, 1H, H-10).

13C NMR spectrum, δ, ppm (100 MHz, DMSO-d6): 18.7, 55.3, 55.7, 97.3, 103.4, 106.4, 114.5 (d, J₁₃C-F = 21.3 Hz), 122.2, 127.3, 129.9, 130.2, 131.3 (d, J₁₃C-F = 8.07 Hz), 133.0, 139.5, 150.9, 152.3, 154.3, 161.4, 161.7 (d, J₁₃C-F = 244 Hz).


6-(4-(dimethylamino)phenyl)-8,9-dimethoxy-2-methylbenzo[c][1,7]-naphthyridin-4(3H)-one (12f)

Yield by method C 323 mg (83%), green powder, mp. 338-342 °C (2-propanol-1,4-dioxane, 2:1). IR (KBr): 1643, 1665, 3402 cm⁻¹.

1H NMR spectrum, δ, ppm (400 MHz, DMSO-d6): 2.35 (s, 3H, CH₃); 3.80 (s, 3H, OCH₃); 3.82 (s, 3H, OCH₃); 4.05 (s, 3H, OCH₃); 6.87 (d, 2H, H = 8.0 Hz, H-3', 5' Ph); 7.10 (s, 1H, H-1); 7.56 (s, 1H, H-7); 7.61 (d, 2H, H = 8.0 Hz, H-2', 6' Ph); 7.88 (s, 1H, H-10); 11.39 (bs, 1H, N-H).

13C NMR spectrum, δ, ppm (100 MHz, DMSO-d6): 18.6, 39.8, 55.3, 55.9, 97.3, 103.7, 107.0, 111.3, 122.2, 127.4, 129.0, 130.4, 131.6, 132.1, 139.2, 150.2, 150.5, 152.0, 155.6, 161.3.

Anal. Calcd for C₂₃H₂₃N₃O₃: C, 70.93; H, 5.95; N, 10.79. Found: C, 70.59; H, 5.78; N, 10.50.

6-(2-hydroxyphenyl)-8,9-dimethoxy-2-methylbenzo[c][1,7]-naphthyridin-4(3H)-one (12g)

Yield 145 mg (40%), pale yellow powder, mp. 338-340 °C (2-propanol-1,4-dioxane, 2:1). IR (KBr): 1650, 3163 cm⁻¹.

1H NMR spectrum, δ, ppm (400 MHz, DMSO-d6): 2.38 (s, 3H, CH₃); 3.80 (s, 3H, OCH₃); 4.06 (s, 3H, OCH₃); 6.95 (t, 1H, H = 6.9 Hz, H-5' Ar); 7.05 (d, 1H, H = 8.0 Hz, H-3' Ar); 7.17 (s, 1H, H-1); 7.34 (d, 1H, H = 7.0 Hz, H-4' Ar); 7.38 (s, 1H, H-7); 7.55 (d, 1H, H = 7.0, H-6' Ar); 7.90 (s, 1H, H-10); 9.02 (bs, 1H, OH); 11.53 (bs, 1H, N-H).

13C NMR spectrum, δ, ppm (100 MHz, DMSO-d6): 18.7, 55.2, 56.0, 97.4,
103.6, 107.3, 116.4, 118.6, 122.6, 124.2, 127.3, 129.8, 129.8, 130.6, 131.8, 140.2, 150.7, 152.4, 153.8, 155.9, 160.9.

Anal. Calcd for C_{21}H_{18}N_{2}O_{4}: C, 69.60; H, 5.36; N, 7.44. Found: C, 69.84; H, 5.12; N, 7.69

8,9-dimethoxy-2-methyl-6-(thiophen-2-yl)benzo[c][1,7]naphthyridin-4(3H)-one (12h)
Yield 187 mg (53%), pale yellow powder, m.p. 338-344 °C (2-propanol-1,4-dioxane, 2:1). IR (KBr): 1649, 3052 cm⁻¹.

\(^1\)H NMR spectrum, δ, ppm (400 MHz, DMSO-d₆): 2.36 (s, 3H, CH₃); 3.94 (s, 3H, OCH₃); 4.07 (s, 3H, OCH₃); 7.13 (s, 1H, H-1); 7.26 (dd, 1H, ^3\)J = 5.1 Hz, ^3\)J = 3.5 Hz, H-4' Th); 7.75 (dd, 1H, ^3\)J = 5.1 Hz, ^4\)J = 1.2 Hz, H-3' Th); 7.78 (dd, 1H, ^3\)J = 3.6 Hz, ^4\)J = 1.1 Hz, H-5' Th); 7.87 (s, 1H, H-7); 7.90 (s, 1H, H-10); 11.48 (bs, 1H, N-H).

\(^1\)C NMR spectrum, δ, ppm (100 MHz, DMSO-d₆): 18.6, 35.4, 55.4, 56.0, 97.3, 103.9, 106.0, 112.5, 127.4, 127.5, 127.8, 129.7, 132.9, 140.2, 142.9, 148.1, 151.3, 152.3, 160.9.

Anal. Calcd for C_{21}H_{18}N_{2}O_{3}: C, 64.76; H, 4.58; N, 7.95. Found: C, 64.98; H, 4.81; N, 7.73

3-(benzylideneamino)-6-methyl-4-phenylpyridin-2(1H)-one (13a)
Yield: 236 mg (82%); yellow crystals, m.p. 214-215°C (2-propanol-chloroform). IR (KBr): v, cm⁻¹: 1636, 1525.

\(^1\)H NMR spectrum, δ, ppm (400 MHz, CDCl₃): 2.41 (s, 3H, CH₃); 6.25 (s, 1H, H-5); 7.35-7.40 (m, 5H, H-2',3',4',5',6' Ph); 7.48-7.53 (m, 3H, H-3",4",5" Ph); 7.75 (dd, 2H, ^3\)J = 7.1 Hz, ^4\)J = 2.4 Hz, H-2", 6" Ph); 9.35 (s, 1H, =C-H); 13.18 (bs, 1H, N-H).

\(^1\)C NMR spectrum, δ, ppm (100 MHz, CDCl₃): 18.7, 108.7, 121.6, 127.5, 128.0, 128.4, 128.6, 130.0, 130.6, 131.0, 131.7, 137.7, 140.8, 146.3, 161.2, 162.6.


2-Methyl-6-phenylbenzo[c][1,7]-naphthyridin-4(3H)-one (14a)
Yield by method B: 200 mg (70%); yield by method C: 143 mg (50%); pale yellow powder, m.p. 315-317 °C (2-propanol-1,4-dioxane, 2:1). IR (KBr): 1650, 3406 cm⁻¹.

\(^1\)H NMR spectrum, δ, ppm (400 MHz, DMSO-d₆): 2.39 (s, 3H, CH₃); 7.13 (s, 1H, H-1); 7.57-7.59 (m, 3H, H-3', 4', 5' Ph); 7.65-7.68 (m, 2H, H-2',6' Ph); 7.79 (td, 1H, ^3\)J = 7.7 Hz, ^4\)J = 1.2 Hz, H-8 ); 7.92 (td, 1H, ^3\)J = 7.6 Hz, ^4\)J = 1.2 Hz, H-9); 8.05 (d, ^3\)J = 8.2 Hz, H-7); 8.64 (d, ^3\)J = 8.2 Hz, H-10); 11.52 (bs, 1H, N-H).

\(^1\)C NMR spectrum, δ, ppm (100 MHz, DMSO-d₆): 18.6, 96.7, 123.6, 126.3, 127.1, 127.7, 128.0, 129.0, 129.3, 130.0, 130.4, 131.1, 133.4, 138.9, 141.0, 157.1, 160.9.

Anal. Calcd for C_{19}H_{14}N_{2}O: C, 79.70; H, 4.93; N, 9.78. Found: C, 79.48; H, 4.79; N, 9.97.
6-(4-methoxyphenyl)-2-methylbenzo[c][1,7]naphthyridin-4(3H)-one (14b)

Yield by method C 161 mg (51%), pale yellow powder, mp. 338-340 °C (2-propanol-1,4-dioxane, 2:1). IR (KBr): 1635, 1668, 3409 cm⁻¹.

¹H NMR spectrum, δ, ppm (400 MHz, DMSO-d6): 2.37 (s, 3H, CH₃); 3.86 (s, 1H, OCH₃); 7.11 - 7.13 (m, 3H, H-1, H-3, 5' Ph); 7.62 (d, 2H, J = 8.6 Hz, H-2', 6' Ph); 7.78 (t, 1H, J = 4.1 Hz, H-4'); 7.90 (t, 1H, J = 8.2 Hz, H-9); 8.10 (d, J = 8.2 Hz, H-7); 8.61 (d, J = 8.2 Hz, H-10), 11.62 (bs, 1H, N-H).

¹³C NMR spectrum, δ, ppm (100 MHz, DMSO-d6): 18.7, 55.1, 97.0, 113.5, 123.8, 126.4, 127.4, 129.1, 130.2, 130.2, 131.0, 131.3, 131.4, 133.5, 140.9, 156.9, 159.5, 161.1.

Anal. Calcd for C₂₉H₁₆N₃O₂: C, 75.93; H, 5.10; N, 8.86. Found: C, 75.65; H, 5.33; N, 8.98

2-methyl-6-(pyridin-2-yl)benzo[c][1,7]naphthyridin-4(3H)-one (14c)

Yield by method B 184 mg (64%), yellow powder, mp. 315-317 °C (2-propanol-1,4-dioxane, 2:1). IR (KBr): 1645, 3406 cm⁻¹.

¹H NMR spectrum, δ, ppm (400 MHz, DMSO-d6): 2.34 (s, 3H, CH₃); 7.02 (s, 1H, H-1); 7.45 (t, 1H, J = 6.1 Hz, H-5' Py); 7.68 (t, 1H, J = 7.6 Hz, H-4' Py); 7.81 (t, 1H, J = 7.6 Hz, H-8 ); 7.92 (t, 1H, J = 7.5 Hz, H-9); 8.01 (dd, J = 7.8 Hz, J = 0.9 Hz, H-3' Py); 8.49 (d, J = 8.4 Hz, H-7); 8.55 (d, J = 8.2 Hz, H-10); 8.71 (d, J = 4.1 Hz, H-6' Py).

¹³C NMR spectrum, δ, ppm (100 MHz, DMSO-d6): 18.7, 97.0, 123.1, 123.5, 124.8, 126.3, 128.0, 129.0, 130.2, 131.3, 131.4, 133.1, 136.7, 141.8, 147.8, 154.1, 157.5, 160.1.


2-methyl-6-(pyridin-4-yl)benzo[c][1,7]naphthyridin-4(3H)-one (14d)

Yield by method B 172 mg (60%), yellow powder, mp. 300-302 °C (2-propanol-1,4-dioxane, 2:1). IR (KBr): 1655, 3404 cm⁻¹.

¹H NMR spectrum, δ, ppm (400 MHz, DMSO-d6): 2.38 (s, 3H, CH₃); 7.26 (s, 1H, H-1); 7.68 (d, 2H, J = 5.0 Hz, H-3', 5' Py); 7.85 (t, 1H, J = 7.3 Hz, H-8 ); 7.98 (t, 1H, J = 7.8 Hz, H-9); 8.03 (d, J = 8.2 Hz, H-7); 8.73 (d, J = 8.2 Hz, H-10); 8.79 (d, J = 5.0 Hz, H-2', 6' Py); 11.88 (bs, 1H, N-H).

¹³C NMR spectrum, δ, ppm (100 MHz, DMSO-d6): 19.6, 97.8, 125.0, 126.4, 127.1, 127.5, 130.7, 131.8, 132.0, 132.7, 134.0, 143.7, 144.4, 153.3, 153.4, 161.5.


6-(4-fluorophenyl)-2-methylbenzo[c][1,7]naphthyridin-4(3H)-one (14e)

Yield by method C 174 mg (57%), pale yellow powder, mp. 339-341 °C (2-propanol-1,4-dioxane, 2:1). IR (KBr): 1639, 1673, 3411 cm⁻¹.

¹H NMR spectrum, δ, ppm (400 MHz, DMSO-d6): 2.37 (s, 3H, CH₃); 7.23 (s, 1H, H-1); 7.40 (t, 2H, J = 8.7 Hz, H-3', 5' Ph); 7.72 (dd, 2H, J = 8.2 Hz, J = 5.5 Hz, H-2', 6' Ph); 7.82 (t, 1H, J = 7.6 Hz, H-8 ), 7.95 (t, 1H, J = 7.6 Hz, H-9); 8.03 (d, J = 8.2 Hz, H-7); 8.69 (d, J = 8.2 Hz, H-10), 11.86 (bs, 1H, N-H).

¹³C NMR spectrum, δ, ppm (100 MHz, DMSO-d6): 18.7, 96.7, 114.7 (d, J = 22.01 Hz), 114.8, 123.8, 126.3, 127.1, 129.2, 130.3, 130.5, 131.2, 131.5 (d, J = 8.07 Hz), 133.4, 135.4, 141.2, 156.1, 161.0, 162.1 (d, J = 246 Hz).
6-(4-(dimethylamino)phenyl)-2-methylbenzo[c][1,7]naphthyridin-4(3H)-one (14f)
Yield by method C 178 mg (54%), pale green powder, mp. 361-362 °C (2-propanol-1,4-dioxane, 2:1). IR (KBr): 1610, 3428 cm⁻¹.
¹H NMR spectrum, δ, ppm (400 MHz, DMSO-d6): 2.34 (s, 3H, CH₃); 3.01 (s, 6H, N(CH₃)₂); 6.89 (d, 2H, ³J = 8.8 Hz, H-3', 5' Ph); 7.04 (s, 1H, H-1); 7.56 (2H, ³J = 8.8 Hz, H-2', 6' Ph); 7.73 (t, 1H, ³J = 7.6 Hz, H-8); 7.85 (t, 1H, ³J = 7.7 Hz, H-9); 8.16 (d, ³J = 8.4 Hz, H-7); 8.57 (d, ³J = 7.8 Hz, H-10).
¹³C NMR spectrum, δ, ppm (100 MHz, DMSO-d6): 20.2, 21.2, 111.5, 126.9, 127.5, 128.6, 129.4, 130.5, 131.3, 133.2, 133.6, 134.6, 145.9, 150.9, 156.7, 159.0, 159.1, 160.0, 160.8.
Anal. Calcd for C₁₉H₁₃FN₂O: C, 74.99; H, 4.31; N, 9.21. Found: C, 75.31; H, 4.16; N, 9.44.

6-(2-hydroxyphenyl)-2-methylbenzo[c][1,7]naphthyridin-4(3H)-one (14g)
Yield by method C 160 mg (53%), pale yellow powder, mp. 340-345 °C (2-propanol-1,4-dioxane, 2:1). IR (KBr): 1649, 3486 cm⁻¹.
¹H NMR spectrum, δ, ppm (400 MHz, DMSO-d6): 2.39 (s, 3H, CH₃); 6.99 (td, 1H, ³J = 7.5 Hz, ³²J = 1.0 Hz, H-5' Ar); 7.05 (dd, 1H, ³J = 8.1 Hz, ³²J = 0.7 Hz, H-3' Ar); 7.15 (s, 1H, H-1); 7.36 (td, 1H, ³J = 7.7 Hz, ³²J = 1.5 Hz, H-4' Ar); 7.44 (dd, 1H, ³J = 7.4 Hz, ³²J = 1.6 Hz, H-6' Ar); 7.77 (td, 1H, ³J = 7.5 Hz, ³²J = 0.9 Hz, H-8); 7.90 (td, 1H, ³J = 7.6 Hz, ³²J = 1.2 Hz, H-9); 7.97 (d, ³J = 8.2 Hz, H-7); 8.61 (d, ³J = 8.2 Hz, H-10); 10.30 (bs, 1H, O-H); 11.60 (bs, 1H, N-H).
¹³C NMR spectrum, δ, ppm (100 MHz, DMSO-d6): 18.7, 96.9, 116.1, 118.5, 123.5, 124.7, 126.8, 127.8, 129.7, 132.7, 132.7, 141.1, 155.6, 156.0, 160.8.
Anal. Calcd for C₁₀H₁₉N₂O: C, 75.48; H, 4.67; N, 9.27. Found: C, 75.67; H, 4.88; N, 9.42.

2-methyl-6-(thiophen-2-yl)benzo[c][1,7]naphthyridin-4(3H)-one (14h)
Yield 131 mg (45%), pale yellow powder, mp. 289-292 °C (2-propanol-1,4-dioxane, 2:1). IR (KBr), ν, cm⁻¹: 1639, 3453.
¹H NMR spectrum, δ, ppm (400 MHz, DMSO-d6): 2.37 (s, 3H, CH₃); 7.09 (s, 1H, H-1); 7.26 (dd, 1H, ³J = 5.1, ³²J = 3.7, H-4 Th); 7.68 (dd, 1H, ³J = 3.6, ³²J = 0.9, H-3 Th); 7.76 (dd, 1H, ³J = 5.1, ³²J = 0.9, H-5 Th); 7.85 (t, 1H, ³J = 7.1 Hz, H-8); 7.91 (t, 1H, ³J = 7.6 Hz, H-9); 8.52 (d, ³J = 8.4 Hz, H-7); 8.59 (d, ³J = 8.1 Hz, H-10); 11.56 (bs, 1H, N-H).
¹³C NMR spectrum, δ, ppm (100 MHz, DMSO-d6): 18.7, 96.9, 123.9, 125.7, 126.6, 127.2, 128.1, 128.6, 129.5, 130.3, 130.4, 131.3, 133.2, 141.3, 142.1, 149.9, 160.7.
Anal. Calcd for C₁₇H₁₂N₂OS: C, 69.84; H, 4.14; N, 9.58. Found: C, 70.15; H, 4.37; N, 9.84.
2-methyl-6-(4-morpholinophenyl)benzo[c][1,7]naphthyridin-4(3H)-one (14i)

Yield 237 mg (64%), pale yellow powder, mp. 332-334 °C (2-propanol-dioxane, 2:1). IR (KBr): v cm⁻¹: 1517, 1662, 1642, 3143.

¹H NMR spectrum, δ, ppm (400 MHz, DMSO-d6): 2.36 (s, 3H, CH₃); 3.24 (t, 4H, J=4.2 Hz, H-5", H-3" N(CH₂)₂); 3.78 (t, 4H, J=4.2 Hz, H-6", H-2" O(CH₂)₂); 7.12 (d, 2H, J=8.2 Hz, H-3', 5' Ar); 7.20 (s, 1H, H-1); 7.59 (d, 2H, J=8.8 Hz, H-2', 6' Ar); 7.81 (t, 1H, J=8.0 Hz, H-8); 7.93 (t, 1H, J=7.1 Hz, H-9); 8.17 (d, 1H, J=7.8 Hz, H-7); 8.66 (d, 1H, J=8.4 Hz, H-10); 11.78 (bs, 1H, N-H).

¹³C NMR spectrum, δ, ppm (100 MHz, DMSO-d6): 19.0, 48.0, 66.0, 97.4, 114.2, 124.2, 126.6, 127.8, 129.4, 129.5, 130.3, 130.5, 130.9, 131.6, 133.7, 140.9, 151.2, 157.3, 161.5.

Anal. Calcd for C₂₃H₂₁N₂O₂: 74.37; H, 5.70; N, 11.31. Found: C, 74.73; H, 5.27; N, 11.02.

2-methyl-6-(4-morpholinophenyl)-5,6-dihydrobenzo[c][1,7]naphthyridin-4(3H)-one (17i)

Yield 134 mg (36%), pale yellow powder, mp. 313-315 °C (2-propanol-dioxane, 2:1). IR (KBr): 1608, 1643, 3480 cm⁻¹.

¹H NMR spectrum, δ, ppm (400 MHz, DMSO-d6): 2.35 (s, 3H, CH₃); 3.23 (t, 4H, J=4.1 Hz, H-5", H-3" N(CH₂)₂); 3.55 (s, 1H, H-6); 3.78 (t, 4H, J=4.1 Hz, H-6", H-2" O(CH₂)₂); 7.11 (d, 2H, J=8.2 Hz, H-3', 5' Ar); 7.18 (s, 1H, H-1); 7.58 (d, 2H, J=8.7 Hz, H-2', 6' Ar); 7.80 (t, 1H, J=7.6 Hz, H-8); 7.91 (t, 1H, J=7.6 Hz, H-9); 8.16 (d, 1H, J=8.2 Hz, H-7); 8.64 (d, 1H, J=8.2 Hz, H-10); 11.78 (bs, 1H, N-H).

¹³C NMR spectrum, δ, ppm (100 MHz, DMSO-d6): 19.0, 48.0, 66.3, 66.6, 97.4, 114.2, 124.2, 126.5, 127.7, 129.4, 129.5, 130.2, 130.5, 130.9, 131.6, 133.7, 140.9, 151.2, 161.5.

Anal. Calcd for C₂₃H₂₃N₂O₂: C, 73.97; H, 6.21; N, 11.25. Found: C, 74.33; H, 6.57; N, 11.02.

2-methylbenzo[c][1,7]naphthyridin-4(3H)-one (18)

Yield 86 mg (41%), pale yellow powder, mp. 220-225 °C (2-propanol-dioxane, 2:1). IR (KBr): 1642, 3168 cm⁻¹.

¹H NMR spectrum, δ, ppm (400 MHz, DMSO-d6): 2.35 (s, 3H, CH₃); 7.13 (s, 1H, H-1); 7.86 (td, 1H, J = 7.4 Hz, J = 4.1 Hz, H-8); 7.92 (td, 1H, J = 7.7 Hz, J = 1.6 Hz, H-9); 8.20 (d, J = 7.8 Hz, H-7); 8.58 (d, J = 8.2 Hz, H-10); 9.21 (s, 1H, H-6); 11.66 (bs, 1H, N-H).

¹³C NMR spectrum, δ, ppm (100 MHz, DMSO-d6): 18.9, 97.4, 123.5, 128.2, 128.4, 129.5, 130.3, 131.0, 131.4, 134.4, 141.4, 150.2, 161.4.

NMR Spectral Data

N-(6-methyl-2-oxo-4-phenyl-1,2-dihydropyridin-3-yl)benzamide (7)

$^1$H NMR (400 MHz, DMSO-d6)

$^{13}$C NMR (100 MHz)
N-[4-(3,4-dimethoxyphenyl)-6-methyl-2-oxo-1,2-dihydropyridin-3-yl]benzamide (8)

$^1$H NMR (400 MHz, DMSO-d6)

$^{13}$C NMR (100 MHz)
5-methyl-2,7-diphenyloxazolo[5,4-b]pyridine (9)

$^1$H NMR (400 MHz, Acetone-d$_6$)

$^{13}$C NMR (100 MHz)
7-(3,4-dimethoxyphenyl)-5-methyl-2-phenyl[1,3]oxazolo[5,4-b]pyridine (10)

$^1$H NMR (400 MHz, DMSO-d6)

$^{13}$C NMR (100 MHz)
8,9-dimethoxy-2-methyl-6-phenylbenzo[c][1,7]-naphthyridin-4(3H)-one (12a)

^1^H NMR (400 MHz, DMSO-d6)

^13^C NMR (100 MHz)
8,9-dimethoxy-6-(4-methoxyphenyl)-2-methylbenzo[c][1,7]-naphthyridin-4(3H)-one (12b)

$^1$H NMR (400 MHz, DMSO-d6)

$^{13}$C NMR (100 MHz)
8,9-dimethoxy-2-methyl-6-(pyridin-2-yl)benzo[c][1,7]naphthyridin-4(3H)-one (12c)

$^1$H NMR (400 MHz, DMSO-d6)

$^{13}$C NMR (100 MHz)
8,9-dimethoxy-2-methyl-6-(pyridin-4-yl)benzo[c][1,7]naphthyridin-4(3H)-one (12d)

\(^1\)H NMR (400 MHz, DMSO-d\(_6\))

\(13\)C NMR (100 MHz)
6-(4-fluorophenyl)-8,9-dimethoxy-2-methylbenzo[c][1,7]naphthyridin-4(3H)-one (12e)

$^1$H NMR (400 MHz, DMSO-d6)

$^{13}$C NMR (100 MHz)
6-(4-(dimethylamino)phenyl)-8,9-dimethoxy-2-methylbenzo[c][1,7]naphthyridin-4(3H)-one (12f)

$^1$H NMR (400 MHz, DMSO-d6)

$^{13}$C NMR (100 MHz)
6-(2-hydroxyphenyl)-8,9-dimethoxy-2-methylbenzo[c][1,7]naphthyridin-4(3H)-one (12g)

$^1$H NMR (400 MHz, DMSO-d6)

$^{13}$C NMR (100 MHz)
8,9-dimethoxy-2-methyl-6-(thiophen-2-yl)benzo[c][1,7]naphthyridin-4(3H)-one (12h)

$^1$H NMR (400 MHz, DMSO-d$_6$)

$^{13}$C NMR (100 MHz)
3-(benzylideneamino)-6-methyl-4-phenylpyridin-2(1H)-one (13a)

$^1$H NMR (400 MHz, DMSO-d6)

$^{13}$C NMR (100 MHz)
2-methyl-6-phenylbenzo[c][1,7]naphthyridin-4(3H)-one (14a)

$^1$H NMR (400 MHz, DMSO-d$_6$)

$^{13}$C NMR (100MHz)
6-(4-methoxyphenyl)-2-methylbenzo[c][1,7]naphthydin-4(3H)-one (14b)

$^1\text{H} \text{NMR (400 MHz, DMSO-d}6\text{)}$

$^{13}\text{C} \text{NMR (100MHz)}$
2-methyl-6-(pyridin-2-yl)benzo[c][1,7]naphthyridin-4(3H)-one (14c)

$^1$H NMR (400 MHz, DMSO-d$_6$)

$^{13}$C NMR (100 MHz)
2-methyl-6-(pyridin-4-yl)benzo[c][1,7]naphthyridin-4(3H)-one (14d)

$^1$H NMR (400 MHz, DMSO-d6)

$^{13}$C NMR (100 MHz)
6-(4-fluorophenyl)-2-methylbenzo[c][1,7]naphthyridin-4(3H)-one (14e)

$^1$H NMR (400 MHz, DMSO-d$_6$)

$^{13}$C NMR (100 MHz)
6-(4-(dimethylamino)phenyl)-2-methylbenzo[c][1,7]naphthyridin-4(3H)-one (14f)

$^1$H NMR (400 MHz, DMSO-d$_6$)

$^{13}$C NMR (100 MHz)
6-(2-hydroxyphenyl)-2-methylbenzo[c][1,7]naphthyridin-4(3H)-one (14g)

$^1$H NMR (400 MHz, DMSO-d6)

$^{13}$C NMR (100 MHz)
2-methyl-6-(thiophen-2-yl)benzo[c][1,7]naphthyridin-4(3H)-one (14h)

$^1$H NMR (400 MHz, DMSO-d$_6$)

$^{13}$C NMR (100 MHz)
2-methyl-6-(4-morpholinophenyl)benzo[c][1,7]naphthyridin-4(3H)-one (14i)

$^1$H NMR (400 MHz, DMSO-d$_6$)

$^{13}$C NMR (100 MHz)
2-methyl-6-(4-morpholinophenyl)-5,6-dihydrobenzo[c][1,7]naphthyridin-4(3H)-one (17i)

\(^1\)H NMR (400 MHz, DMSO-d\textsubscript{6})

\[^{13}\text{C} \text{ NMR (100 MHz)}\]
2-methylbenzo[c][1,7]naphthyridin-4(3H)-one (18)

$^1$H NMR (400 MHz, DMSO-d6)

$^{13}$C NMR (100 MHz)
Figure 2. X-ray structure of 14a. Thermal ellipsoids are shown at 50% probability. (Chloroform molecule is disordered over two positions with occupancies 0.866(7):0.134(7). Cl1A, Cl2A and Cl3A atoms are the minor part)

Analysis of Potential Hydrogen Bonds

N(3)-H(3)...O(1) [3665.01]  0.86  2.02  2.843(4)  160
C(7)-H(7)...Cl(1) [1455.02]  0.93  2.82  3.619(4)  144
C(12)-H(12)...O(1) [ ]  0.98  2.50  3.283(4)  137
C(12)-H(12)...N(5) [ ]  0.98  2.38  3.286(4)  154

Translation of ARU-code to Equivalent Position Code

[ 3665. ] = 1-x, 1-y, -z
[ 1455. ] = -1+x, y, z

Pi-stacking interactions: interplane distance 3.412(2) (between benzo[c][1,7]naphthyridin frames), intercentroid Cg-Cg range 3.571(2) – 3.892(2) Å.
Data were collected on a Bruker Kappa Apex II CCD diffractometer using graphite monochromated MoKα radiation. All usual procedures were performed with SHELX-97 program set. CCDC 1518432 contains the supplementary crystallographic data for this paper.

**Table 2. Crystal data and structure refinement for 14a**

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<td><strong>Temperature</strong></td>
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<td><strong>Wavelength</strong></td>
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<td></td>
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<td></td>
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<td><strong>Largest diff. peak and hole</strong></td>
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X-ray data of 14a (CIF)

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_symmetry_space_group_name_H-M  'P2(1)/n'

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                       '-x, y, z'
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_cell_length_c                 26.9825(15)
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_cell_angle_beta               91.560(2)
_cell_angle_gamma              90.00
_cell_volume                   1904.57(18)
_cell_formula_units_Z          4
_cell_measurement_temperature  296(2)
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_diffrn_reflns_av_sigmaI/netI     0.0276
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_diffrn_reflns_limit_k_min       -9
_diffrn_reflns_limit_k_max       9
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_computing_structure_solution    'SHELXS-97 (Sheldrick, 2008)'
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_computing_molecular_graphics    'Bruker SHELXTL'
_computing_publication_material  'Bruker SHELXTL'

_refine_special_details
;
Refinement of F^2^ against ALL reflections. The weighted R-factor wR and
goodness of fit S are based on F^2^, conventional R-factors R are based
on F, with F set to zero for negative F^2^*. The threshold expression of
F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
not relevant to the choice of reflections for refinement. R-factors based
on F^2^ are statistically about twice as large as those based on F, and R-
factors based on ALL data will be even larger.
;
_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
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_refine_ls_weighting_details      "calc w=1/[\s^2^>(Fo^2^)+(0.1759P)^2^+3.0519P] where P=(Fo^2^+2Fc^2^)/3"  
_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     constr
_refine_ls_extinction_method      none
_refine_ls_extinction_coef        ?
_refine_ls_number_reflns          4212
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_refine_ls_number_restraints      6
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_refine_ls_wR_factor_gt 0.2118
_refine_ls_goodness_of_fit_ref 0.925
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_atom_site_disorder_group

Cl1 Cl 0.51797(19) 0.8708(3) 0.14330(10) 0.0806(8) Uani 0.866(7) 1 d P A 1
Cl2 Cl 0.25664(19) 0.9508(5) 0.18028(9) 0.0878(8) Uani 0.866(7) 1 d P A 1
Cl3 Cl 0.3801(3) 0.5935(4) 0.20038(8) 0.0970(8) Uani 0.866(7) 1 d P A 1
C12 C 0.3612(4) 0.7768(6) 0.15807(13) 0.0579(10) Uani 1 1 d . A 1
H12 H 0.3187 0.7272 0.1276 0.070 Uiso 1 1 calc R .
C12A C 0.3612(4) 0.7768(6) 0.15807(13) 0.0579(10) Uani 1 1 d . A 1
Cl1A Cl 0.5470(9) 0.8137(14) 0.1626(4) 0.057(3) Uiso 0.134(7) 1 d PD A 2
Cl2A Cl 0.2900(19) 1.0058(19) 0.1735(7) 0.090(5) Uiso 0.134(7) 1 d PD A 2
Cl3A Cl 0.3344(14) 0.6535(17) 0.2115(4) 0.075(3) Uiso 0.134(7) 1 d PD A 2
O1 O 0.3969(2) 0.5755(4) 0.04997(8) 0.0454(6) Uani 1 1 d . . .
C1 C 0.1459(3) 0.7172(4) -0.06672(10) 0.0353(6) Uani 1 1 d . . .
H1 H 0.0912 0.7498 -0.0940 0.042 Uiso 1 1 calc R .
C2 C 0.2759(3) 0.6632(4) -0.07319(10) 0.0371(6) Uani 1 1 d . . .
N3 N 0.3569(3) 0.6184(4) -0.03236(9) 0.0371(6) Uani 1 1 d . . .
H3 H 0.4396 0.5865 -0.0376 0.044 Uiso 1 1 calc R .
C4 C 0.3164(3) 0.6206(4) -0.01584(10) 0.0579(10) Uani 1 1 d . . .
C4A C 0.1750(3) 0.6774(3) -0.02247(9) 0.0289(5) Uani 1 1 d . . .
N5 N 0.1331(2) 0.6777(3) 0.07073(8) 0.0311(5) Uani 1 1 d . . .
C6 C 0.0063(3) 0.7221(4) 0.07956(10) 0.0313(6) Uani 1 1 d . . .
C6A C -0.0917(3) 0.7690(3) 0.04068(10) 0.0305(6) Uani 1 1 d . . .
C7 C -0.2299(3) 0.8036(4) 0.04964(12) 0.0395(6) Uani 1 1 d . . .
H7 H -0.2612 0.7936 0.0818 0.047 Uiso 1 1 calc R .
C8 C -0.3194(3) 0.8520(5) 0.01182(14) 0.0469(8) Uani 1 1 d . . .
H8 H -0.4100 0.8764 0.0186 0.056 Uiso 1 1 calc R .
C9 C -0.2747(4) 0.8645(5) -0.03675(13) 0.0469(8) Uani 1 1 d . . .
H9 H -0.3351 0.8998 -0.0622 0.056 Uiso 1 1 calc R .
C10 C -0.1417(3) 0.8249(4) -0.04702(11) 0.0385(6) Uani 1 1 d . . .
H10 H -0.1132 0.8312 -0.0796 0.046 Uiso 1 1 calc R .
C10A C -0.0478(3) 0.7748(3) -0.00883(10) 0.0305(6) Uani 1 1 d . . .
C10B C 0.0917(3) 0.7245(3) -0.01806(9) 0.0293(5) Uani 1 1 d . . .
C11 C 0.3405(4) 0.6435(6) -0.12252(13) 0.0552(9) Uani 1 1 d . . .
H11A H 0.2751 0.6737 -0.1484 0.066 Uiso 1 1 calc R .
H11B H 0.3709 0.5176 -0.1266 0.066 Uiso 1 1 calc R .
H11C H 0.4168 0.7264 -0.1242 0.066 Uiso 1 1 calc R .
C1' C -0.0298(3) 0.7162(4) 0.13291(10) 0.0368(6) Uani 1 1 d . . .
C2' C 0.0134(4) 0.5652(5) 0.16151(12) 0.0479(8) Uani 1 1 d . . .
H2' H 0.0615 0.4691 0.1469 0.058 Uiso 1 1 calc R .
C3' C -0.0144(5) 0.5566(8) 0.21140(15) 0.0704(12) Uani 1 1 d . . .
H3' H 0.0135 0.4541 0.2301 0.084 Uiso 1 1 calc R .
C4' C -0.0831(5) 0.6990(9) 0.23341(14) 0.0782(15) Uani 1 1 d . . .
H4' H -0.1017 0.6926 0.2670 0.094 Uiso 1 1 calc R .
C5' C -0.1245(4) 0.8504(8) 0.20631(15) 0.0703(12) Uani 1 1 d . . .
H5' H -0.1694 0.9479 0.2215 0.084 Uiso 1 1 calc R .
C6' C -0.0991(4) 0.8584(6) 0.15580(13) 0.0524(8) Uani 1 1 d . . .
H6' H -0.1291 0.9604 0.1373 0.063 Uiso 1 1 calc R .
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C11 0.0557(8) 0.0921(12) 0.0952(15) -0.0151(11) 0.0245(9) -0.0125(8)
C12 0.0534(8) 0.1184(17) 0.0915(12) -0.0424(12) 0.0007(8) 0.0071(10)
C13 0.0989(13) 0.1193(16) 0.0732(11) 0.0294(10) 0.0092(9) 0.0118(13)
C12 0.0469(18) 0.087(3) 0.0403(17) -0.0106(17) 0.0006(14) -0.0092(18)
C12A 0.0469(18) 0.087(3) 0.0403(17) -0.0106(17) 0.0006(14) -0.0092(18)
Cl1 0.0557(8) 0.0921(12) 0.0952(15) -0.0151(11) 0.0245(9) -0.0125(8)
Cl2 0.0534(8) 0.1184(17) 0.0915(12) -0.0424(12) 0.0007(8) 0.0071(10)
Cl3 0.0989(13) 0.1193(16) 0.0732(11) 0.0294(10) 0.0092(9) 0.0118(13)
C12 0.0469(18) 0.087(3) 0.0403(17) -0.0106(17) 0.0006(14) -0.0092(18)
C12A 0.0469(18) 0.087(3) 0.0403(17) -0.0106(17) 0.0006(14) -0.0092(18)
Cl1 0.0557(8) 0.0921(12) 0.0952(15) -0.0151(11) 0.0245(9) -0.0125(8)
Cl2 0.0534(8) 0.1184(17) 0.0915(12) -0.0424(12) 0.0007(8) 0.0071(10)
Cl3 0.0989(13) 0.1193(16) 0.0732(11) 0.0294(10) 0.0092(9) 0.0118(13)
C12 0.0469(18) 0.087(3) 0.0403(17) -0.0106(17) 0.0006(14) -0.0092(18)
C12A 0.0469(18) 0.087(3) 0.0403(17) -0.0106(17) 0.0006(14) -0.0092(18)
Cl1 0.0557(8) 0.0921(12) 0.0952(15) -0.0151(11) 0.0245(9) -0.0125(8)
Cl2 0.0534(8) 0.1184(17) 0.0915(12) -0.0424(12) 0.0007(8) 0.0071(10)
Cl3 0.0989(13) 0.1193(16) 0.0732(11) 0.0294(10) 0.0092(9) 0.0118(13)
C12 0.0469(18) 0.087(3) 0.0403(17) -0.0106(17) 0.0006(14) -0.0092(18)
C12A 0.0469(18) 0.087(3) 0.0403(17) -0.0106(17) 0.0006(14) -0.0092(18)
All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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C11 C12 1.738(4) . ?
C12 C12 1.736(5) . ?
C13 C12 1.750(5) . ?
C12 H12 0.980 . ?
O1 C4 1.240(4) . ?
C1 C2 1.351(4) . ?
C1 C10B 1.431(4) . ?
C1 H1 0.930 . ?
C2 N3 1.379(4) . ?
C2 C11 1.497(4) . ?
N3 C4 1.370(4) . ?
N3 H3 0.860 . ?
C4 C4A 1.464(4) . ?
C4A N5 1.376(3) . ?
C4A C10B 1.390(4) . ?

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N5 C6 1.314(4) .
C6 C6A 1.444(4) .
C6 C1' 1.492(4) .
C6A C7 1.408(4) .
C6A C10A 1.416(4) .
C7 C8 1.373(5) .
C7 H7 0.9300 .
C8 C9 1.396(5) .
C8 H8 0.9300 .
C9 C10 1.372(5) .
C9 H9 0.9300 .
C10 C10A 1.412(4) .
C10 H10 0.9300 .
C11 H11A 0.9600 .
C11 H11B 0.9600 .
C11 H11C 0.9600 .
C1' C6' 1.383(5) .
C1' C2' 1.391(5) .
C2' C3' 1.382(5) .
C2' H2' 0.9300 .
C3' C4' 1.370(8) .
C3' H3' 0.9300 .
C4' C5' 1.367(8) .
C4' H4' 0.9300 .
C5' C6' 1.393(5) .
C5' H5' 0.9300 .
C6' H6' 0.9300 .

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C11 C12 C12 109.6(3) .
C13 C12 C12 111.8(2) .
C11 C12 H12 108.0 .
C13 C12 H12 108.0 .
C12 C12 H12 108.0 .
C2 C1 C10B 120.4(3) .
C2 C1 H1 119.8 .
C10B C1 H1 119.8 .
C1 C2 N3 119.4(2) .
C1 C2 C11 124.6(3) .
N3 C2 C11 116.0(3) .
C4 N3 C2 125.4(3) .
C4 N3 H3 117.3 .
C2 N3 H3 117.3 .
O1 C4 N3 120.4(3) .
O1 C4 C4A 124.7(3) .
N3 C4 C4A 114.8(2) .
N5 C4A C10B 124.0(2) .
N5 C4A C4 115.2(2) .
C10B C4A C4 120.8(2) .
C6 N5 C4A 118.8(2) .
N5 C6 C6A 122.8(2) .
N5 C6 C1' 114.8(2) .
C6A C6 C1' 122.5(2) .
C7 C6A C10A 118.5(3) .
C7 C6A C6 122.9(3) .
C10A C6A C6 118.6(2) .
C8 C7 C6A 121.2(3) .
C8 C7 H7 119.4 .
C6A C7 H7 119.4 . . ?
C7 C8 C9 120.2(3) . . ?
C7 C8 H8 119.9 . . ?
C9 C8 H8 119.9 . . ?
C10 C9 C8 120.1(3) . . ?
C10 C9 H9 119.9 . . ?
C8 C9 H9 119.9 . . ?
C9 C10 C10A 120.8(3) . . ?
C9 C10 H10 119.6 . . ?
C10A C10 H10 119.6 . . ?
C10 C10A C6A 119.1(3) . . ?
C10 C10A C10B 122.8(3) . . ?
C6A C10A C10B 118.1(2) . . ?
C4A C10B C1 119.2(2) . . ?
C4A C10B C10A 117.8(2) . . ?
C1 C10B C10A 123.1(2) . . ?
C2 C11 H11A 109.5 . . ?
C2 C11 H11B 109.5 . . ?
H11A C11 H11B 109.5 . . ?
C2 C11 H11C 109.5 . . ?
H11A C11 H11C 109.5 . . ?
C6' C1' C2' 118.4(3) . . ?
C6' C1' C6 122.9(3) . . ?
C2' C1' C6 118.7(3) . . ?
C3' C2' C1' 120.6(4) . . ?
C3' C2' H2' 119.7 . . ?
C1' C2' H2' 119.7 . . ?
C4' C3' C2' 120.1(4) . . ?
C4' C3' H3' 119.9 . . ?
C2' C3' H3' 119.9 . . ?
C5' C4' C3' 120.4(4) . . ?
C5' C4' H4' 119.8 . . ?
C3' C4' H4' 119.8 . . ?
C4' C5' C6' 119.8(4) . . ?
C4' C5' H5' 120.1 . . ?
C6' C5' H5' 120.1 . . ?
C1' C6' C5' 120.6(4) . . ?
C1' C6' H6' 119.7 . . ?
C5' C6' H6' 119.7 . . ?

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