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Synthesis, and spectroscopic and structural characterization of three new styrylquinoline–benzimidazole hybrids

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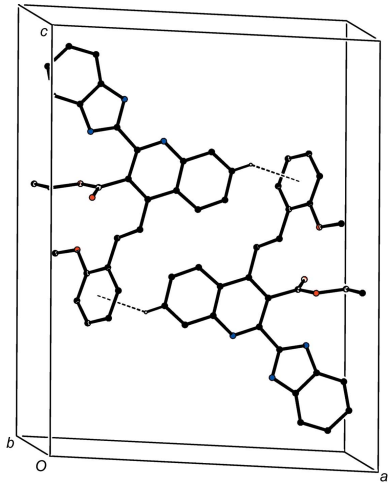
Three new 4-styrylquinoline–benzimidazole hybrids have been synthesized using a reaction sequence in which 2-methylquinoline precursors first undergo selective oxidation by selenium dioxide to form the corresponding 2-formylquinoline intermediates, followed by oxidative cyclocondensation reactions with benzene-1,2-diamine to yield the hybrid products. The formyl intermediates and the hybrid products have all been fully characterized using a combination of IR, ^{1}H and ^{13}C NMR spectroscopy, and high-resolution mass spectrometry, and the structures of the three hybrid products have been determined using single-crystal X-ray diffraction. Ethyl (*E*)-2-(1*H*-benzo[*d*]imidazol-2-yl)-4-(4-chlorostyryl)quinoline-3-carboxylate, $C_{27}H_{20}ClN_3O_2$, (IIIa), and ethyl (*E*)-2-(1*H*-benzo[*d*]imidazol-2-yl)-4-(2-methoxystyryl)quinoline-3-carboxylate, $C_{28}H_{23}N_3O_3$, (IIIb), both crystallize in the solvent-free form with Z' = 1, but ethyl (*E*)-2-(1*H*-benzo[*d*]imidazol-2-yl)-4-(4-methylstyryl)quinoline-3-carboxylate, $C_{28}H_{23}N_3O_2$, (IIIc), crystallizes as a partial hexane solvate with Z' = 3, and the ester group in one of the independent molecules is disordered over two sets of atomic sites having occupancies of 0.765 (7) and 0.235 (7). The molecules of (IIIc) enclose continuous channels which are occupied by disordered solvent molecules having partial occupancy. In all of the molecules of (IIIa)–(IIIc), the styrylquinoline fragment is markedly nonplanar. Different combinations of N—H···O and C—H···π hydrogen bonds generate supramolecular assemblies which are two-dimensional in (IIIb) and (IIIc), but three-dimensional in (IIIa). Comparisons are made with the structures of some related compounds.



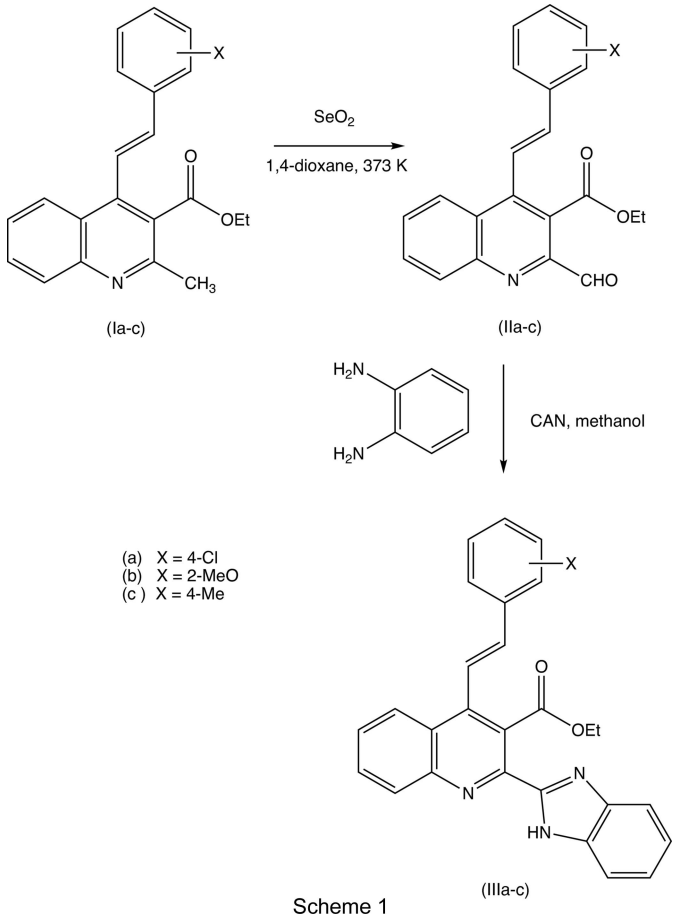
1. Introduction

Among different privileged scaffolds, quinolines can be considered as one of the most versatile pharmacophores due to their presence in a wide variety of natural and synthetic molecules. Quinoline derivatives exhibit a broad range of biological activity, such as antimalarial (*e.g.* quinine and mefloquine) (Hu *et al.*, 2017; Kaur *et al.*, 2010; Orozco *et al.*, 2020), antiviral (*e.g.* saquinavir) (Matada *et al.*, 2021), anticancer (*e.g.* camptothecin and topotecan) (Afzal *et al.*, 2015; Lauria *et al.*, 2021; Musiol, 2017; Yadav & Shah, 2021) and anti-asthmatic (*e.g.* montelukast) (Matada *et al.*, 2021; Nayak, 2004). Quinoline derivatives are also frequently used as building blocks in the design and synthesis of new biologically active molecular hybrids with the aim of developing new chemical entities for further clinical assays (Jagdale & Patil, 2019; Yadav & Shah, 2021).

The benzimidazole nucleus also constitutes a privileged scaffold which has been extensively studied as a potential building block for the development of biologically active

molecules with diverse applications as therapeutic agents, including anticancer agents (*e.g.* dovitinib and selumetinib) (Hernández-Romero *et al.*, 2021), anthelmintics (*e.g.* albendazole, mebendazole and thabendazole) (Salahuddin *et al.*, 2017) or antacids and anti-ulcer agents (*e.g.* omeprazole, lansoprazole and pantoprazole) (Gurvinder *et al.*, 2013).

Most of the synthetic methods for building the benzimidazole nucleus reported hitherto are based on cyclocondensation reactions of benzene-1,2-diamine (*o*-phenylenediamine) either with carboxaldehydes in the presence of Lewis acids or oxidizing agents (Agrawal *et al.*, 2012; Bellam *et al.*, 2017; Kidwai *et al.*, 2010; Lin & Yang, 2005; Singh *et al.*, 2000), or with carboxylic acids in strongly acidic conditions at high temperatures (Cosimelli *et al.*, 2011; Singhal *et al.*, 2019). Because of the medicinal importance of quinoline and benzimidazole derivatives, considerable efforts have been made in the development of novel quinoline–benzimidazole hybrids (Cosimelli *et al.*, 2011; Hranjec *et al.*, 2010; Mantu *et al.*, 2016; Perin *et al.*, 2016; Renhowe *et al.*, 2009; Yaragorla & Vijaya Babu, 2017).



Scheme 1

With these considerations in mind, and as a continuation of our earlier work on the synthesis of polysusbstituted 4-styrylquinolines from 2′-aminophenylchalcones and 1,3-dicarbonyl compounds (Meléndez *et al.*, 2020), we report here the synthesis and spectroscopic characterization of three representative examples of a novel class of molecular hybrids of the type benzimidazole-4-styrylquinoline, namely, ethyl (*E*)-2-(1*H*-benzo[*d*]imidazol-2-yl)-4-(4-chlorostyryl)quinoline-3-carboxylate, (IIIa), ethyl (*E*)-2-(1*H*-benzo[*d*]imidazol-2-yl)-4-(2-methoxystyryl)quinoline-3-carboxylate, (IIIb), and ethyl (*E*)-2-(1*H*-benzo[*d*]imidazol-2-yl)-4-(4-methylstyryl)quinoline-3-carboxylate, (IIIc), which differ only in the nature of the substituents in the benzene ring of the styryl fragment (see Scheme 1), along with the molecular and supramolecular structures of the hybrid products (IIIa)–(IIIc) (Figs. 1–3).

2. Experimental

2.1. Synthesis and crystallization

The 4-styrylquinoline precursors of type (I) (see Scheme 1) were prepared using a previously reported method (Meléndez *et al.*, 2020; Rodríguez *et al.*, 2020). In the NMR data listed below, for compounds (III), unprimed ring atoms form part of the quinoline unit, ring atoms carrying a single prime form part of the benzimidazole unit and ring atoms carrying a double prime form part of the styryl unit (see Figs. 1 and 2).

For the synthesis of the formyl intermediates of type (II), a suspension of the appropriate 4-styrylquinoline-3-carboxylate (I) (Meléndez *et al.*, 2020; see Scheme 1) (1.0 mmol) and selenium dioxide (2.0 mmol) in 1,4-dioxane (5 ml) was stirred and heated at 373 K for the time required to complete the reaction. After the complete consumption of (I) [as monitored by thin-layer chromatography (TLC)], dichloromethane (15 ml) was added and the resulting suspension was filtered. The solvent was removed under reduced pressure and the resulting crude products were purified by flash column chromatography on silica gel using hexane–ethyl acetate (10:1 *v*/*v*) as eluent to give the required formyl intermediates (IIa)–(IIc) as solid compounds.

Compound (IIa), ethyl (*E*)-4-(4-chlorostyryl)-2-formylquinoline-3-carboxylate; yield 0.15 g (90%); m.p. 387–389 K; R_F = 0.28 (12.5% ethyl acetate–hexane). FT–IR (ATR, cm^{-1}): 1727 (C═O$_{formyl}$), 1706 (C═O$_{ester}$), 1638 (C═N), 1612 (C═C$_{vinyl}$), 1558 (C═C$_{arom}$), 1488 (C═C$_{arom}$), 971 (═C—H$_{\textit{trans}}$). NMR (CDCl$_3$): δ(^{1}H) 10.19 (*s*, 1H, –COH), 8.28 (*dd*, *J* = 8.4, 1.3 Hz, 1H, H8), 8.20 (*dd*, *J* = 8.4, 1.5 Hz, 1H, H5), 7.88 (*ddd*, *J* = 8.4, 6.9, 1.5 Hz, 1H, H7), 7.74 (*ddd*, *J* = 8.3, 6.8, 1.3 Hz, 1H, H6), 7.51–7.48 (*m*, 2H, H2′, H6′), 7.45 (*d*, *J* = 16.5 Hz, 1H, H$_A$C═), 7.41–7.38 (*m*, 2H, H3′, H5′), 7.03 (*d*, *J* = 16.5 Hz, 1H, ═CH$_B$), 4.45 (*q*, *J* = 7.2 Hz, 2H, –OCH$_2$–), 1.33 (*t*, *J* = 7.2 Hz, 3H, –CH$_3$); δ(^{13}C) 192.5 (C═O$_{formyl}$), 167.4 (C═O$_{ester}$), 148.7 (C2), 147.5 (C8a), 143.1 (C4), 138.0 (═CH$_B$), 135.0 (C4′), 134.5 (C1′), 131.1 (C7), 131.0 (C8), 130.0 (C6), 129.2 (C3′, C5′), 128.2 (C2′, C6′), 127.5 (C4a), 125.4 (C5), 124.2 (C3), 121.3 (H$_A$C═), 62.2 (–OCH$_2$–), 14.1 (–CH$_3$). HRMS (ESI$^+$) *m*/*z* found for [*M* + H]$^+$ 366.0892, C$_{21}$H$_{16}$ClNO$_3$ requires 366.0891.

Compound (IIb), ethyl (*E*)-2-formyl-4-(2-methoxystyryl)-quinoline-3-carboxylate; yield 0.16 g (97%); m.p. 372–373 K; R_F = 0.31 (12.5% ethyl acetate–hexane). FT–IR (ATR, cm^{-1}): 1726 (C═O), 1704 (C═O), 1597 (C═N), 1563 (C═C$_{vinyl}$), 1484 (C═C$_{arom}$), 1462 (C═C$_{arom}$), 970 (═C—H$_{\textit{trans}}$). NMR (CDCl$_3$): δ(^{1}H) 10.19 (*s*, 1H, –COH), 8.28 (*ddd*, *J* = 8.5, 1.4, 0.7 Hz, 1H, H5), 8.27 (*ddd*, *J* = 8.6, 1.4, 0.7 Hz, 1H, H8), 7.86

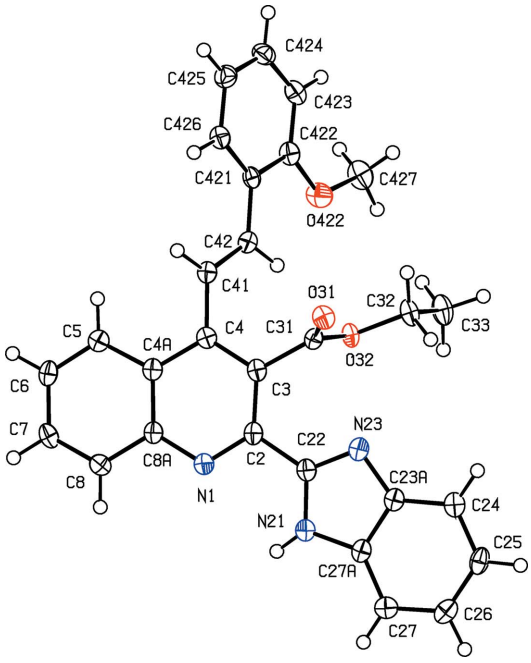


Figure 2
The molecular structure of compound (IIIb), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

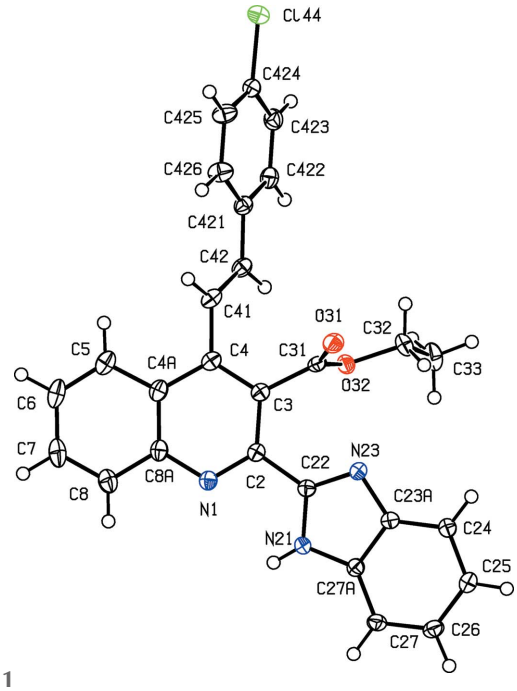


Figure 1
The molecular structure of compound (IIIa), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

(*ddd*, J = 8.4, 6.9, 1.4 Hz, 1H, H7), 7.72 (*ddd*, J = 8.3, 6.9, 1.3 Hz, 1H, H6), 7.62 (*dd*, J = 7.6, 1.7 Hz, 1H, H6′), 7.54 (*d*, J = 16.7 Hz, 1H, H_AC═), 7.42 (*d*, J = 16.7 Hz, 1H, ═CH_B), 7.34 (*ddd*, J = 8.3, 7.4, 1.7 Hz, 1H, H4′), 7.02 (*td*, J = 7.4, 1.1 Hz, 1H, H5′), 6.95 (*dd*, J = 8.3, 1.1 Hz, 1H, H3′), 4.47 (*q*, J = 7.2 Hz, 2H, –OCH_2–), 3.88 (*s*, 3H, 2′-OCH_3), 1.36 (*t*, J = 7.2 Hz, 3H, –CH_3); δ(^{13}C) 192.6 (C═O_{formyl}), 167.3 (C═O_{ester}), 157.5 (C2′), 148.6 (C2), 147.5 (C8a), 144.2 (C4), 134.7 (═CHB), 130.9 (C8), 130.8 (C7), 130.2 (C4′), 129.8 (C6), 127.7 (C4a), 127.5 (C6′), 125.7 (C5), 125.1 (C1′), 124.1 (C3), 121.2 (H_AC═), 120.8 (C5′), 111.1 (C3′), 62.0 (–OCH_2–), 55.5 (2′-OCH_3), 14.0 (–CH_3). HRMS (ESI$^+$) m/z found for $[M + H]^+$ 362.1388, $C_{22}H_{19}NO_4$ requires 362.13868.

Compound (IIc), ethyl (*E*)-2-formyl-4-(4-methylstyryl)-quinoline-3-carboxylate; yield 0.145 g (92%); m.p. 364–365 K; R_F = 0.32 (12.5% ethyl acetate–hexane). FT–IR (ATR, cm^{-1}):

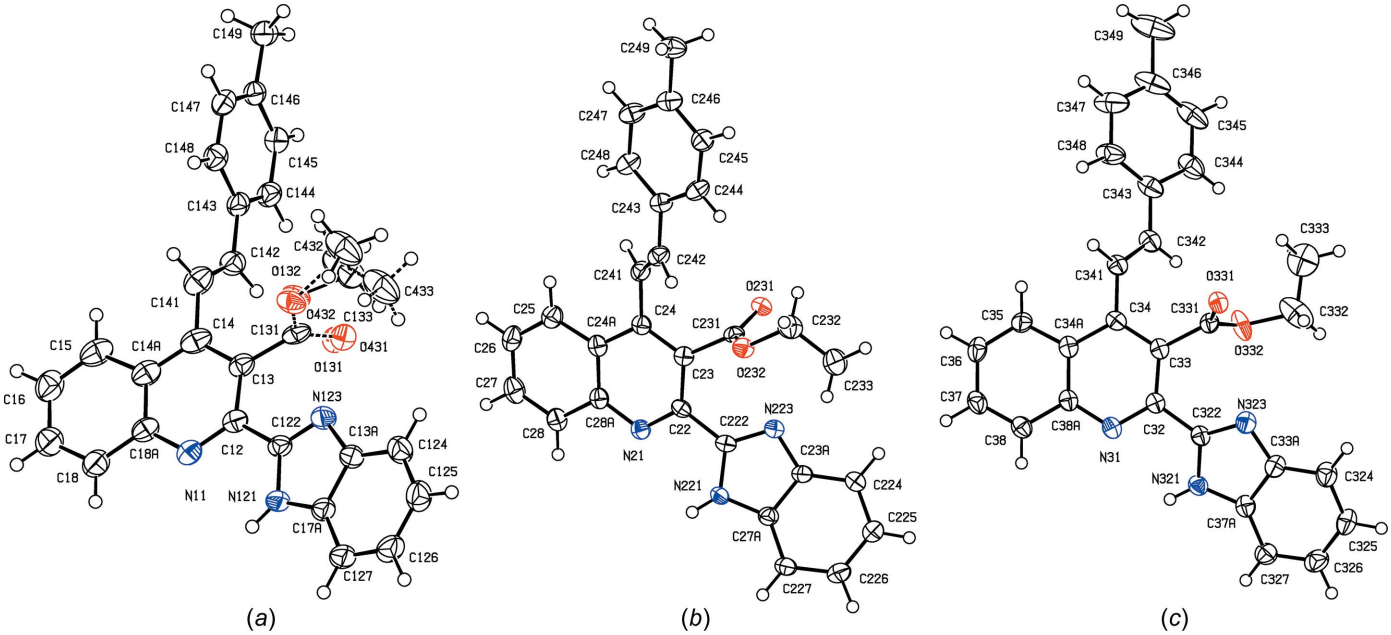


Figure 3
The three independent molecules of compound (IIIc), showing the atom-labelling schemes for (*a*) molecule 1, where the minor disorder component has been drawn using broken lines, (*b*) molecule 2 and (*c*) molecule 3. Displacement ellipsoids are drawn at the 50% probability level.

Table 1
Experimental details.

Experiments were carried out at 100 K with Mo $K\alpha$ radiation using a Bruker D8 Venture diffractometer. Absorption was corrected for by multi-scan methods (*SADABS*; Bruker, 2016). H atoms were treated by a mixture of independent and constrained refinement.

	(IIIa)	(IIIb)	(IIIc)
Crystal data			
Chemical formula	$C_{27}H_{20}ClN_3O_2$	$C_{28}H_{23}N_3O_3$	$C_{28}H_{23}N_3O_2$(+solvent)
M_r	453.91	449.49	433.49
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/n$
a, b, c (Å)	12.1791 (5), 18.8348 (7), 10.7533 (4)	15.8086 (14), 6.9536 (6), 20.4101 (19)	20.2611 (7), 9.8675 (4), 36.8434 (14)
β (°)	114.242 (1)	94.330 (4)	104.332 (1)
V (Å^3)	2249.19 (15)	2237.2 (3)	7136.7 (5)
Z	4	4	12
μ (mm^{-1})	0.20	0.09	0.08
Crystal size (mm)	0.18 × 0.16 × 0.12	0.16 × 0.12 × 0.08	0.18 × 0.12 × 0.06
Data collection			
T_{min}, T_{max}	0.928, 0.976	0.817, 0.993	0.917, 0.995
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	56016, 4969, 4417	41777, 5131, 3453	151790, 17718, 10436
R_{int}	0.051	0.132	0.188
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.642	0.650	0.667
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.037, 0.096, 1.05	0.059, 0.133, 1.06	0.096, 0.216, 1.08
No. of reflections	4969	5131	17718
No. of parameters	302	312	921
No. of restraints	0	0	7
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.61, −0.32	0.25, −0.26	0.71, −0.41

Computer programs: *APEX3* (Bruker, 2018), *SAINT* (Bruker, 2017), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *PLATON* (Spek, 2020).

1707 (C═$O_{formyl/ester}$), 1628 (C═N), 1560 (C═C_{vinyl}), 1513 (C═C_{arom}), 1463 (C═C_{arom}), 987 (═C—H_{trans}). NMR ($CDCl_3$): δ(^{1}H) 10.19 (*s*, 1H, –COH), 8.27 (*dd*, *J* = 8.3, 1.3 Hz, 1H, H8), 8.23 (*dd*, *J* = 8.4, 1.3 Hz, 1H, H5), 7.86 (*ddd*, *J* = 8.4, 6.9, 1.4 Hz, 1H, H7), 7.72 (*ddd*, *J* = 8.3, 6.9, 1.3 Hz, 1H, H6), 7.46 (*d*, *J* = 7.8 Hz, 2H, H2′, H6′), 7.43 (*d*, *J* = 16.6 Hz, 1H, H_AC═), 7.23 (*d*, *J* = 7.8 Hz, 2H, H3′, H5′), 7.06 (*d*, *J* = 16.6 Hz, 1H, ═CH_B), 4.46 (*q*, *J* = 7.2 Hz, 2H, –OCH_2–), 2.40 (*s*, 3H, 4′-CH_3), 1.34 (*t*, *J* = 7.2 Hz, 3H, –CH_3); δ(^{13}C) 192.6 (C═O_{formyl}), 167.6 (C═O_{ester}), 148.6 (C2), 147.5 (C8a), 143.7 (C4), 139.3 (═CH_B, C4′), 133.3 (C1′), 131.0 (C7), 130.9 (C8), 129.8 (C6), 129.7 (C3′, C5′), 127.7 (C4a), 127.0 (C2′, C6′), 125.6 (C5), 124.2 (C3), 119.6 (H_AC═), 62.1 (–OCH_2–), 21.4 (4′-CH_3), 14.3 (–CH_3). HRMS (ESI$^+$) *m*/*z* found for $[M + H]^+$ 346.1452, $C_{22}H_{19}NO_3$ requires 346.1438.

For the synthesis of the benzimidazole products of type (III), a suspension of the appropriate formyl derivatives (II) (1.0 mmol), *o*-phenylenediamine (1.0 mmol) and ceric ammonium nitrate (CAN) (10 mol%) in methanol (2 ml) was magnetically stirred at ambient temperature for the time required to complete the reaction. After the complete consumption of (II) (as monitored by TLC), methanol was removed under reduced pressure and the crude products were purified by flash column chromatography on silica gel using hexane–ethyl acetate (8:1 *v*/*v*) as eluent to yield the target hybrid products (IIIa)–(IIIc), which were then recrystallized from hexane–ethyl acetate (7:1 *v*/*v*), at ambient temperature and in the presence of air, to give yellow crystals suitable for single-crystal X-ray diffraction.

Compound (IIIa), ethyl (*E*)-2-(1*H*-benzo[*d*]imidazol-2-yl)-4-(4-chlorostyryl)quinoline-3-carboxylate; yield 0.145 g (65%); m.p. 447–448 K; R_F = 0.31 (15% ethyl acetate–hexane). FT–IR (ATR, cm^{-1}): 3377 (N—H), 1716 (C═O), 1583 (C═N), 1488 (C═C_{vinyl}), 1455 (C═C_{arom}), 1434 (C═C_{arom}), 964 (═C—H_{trans}). NMR ($CDCl_3$): δ(^{1}H) 10.69 (*s*, 1H, N—H), 8.15 (*ddd*, *J* = 8.4, 1.5, 0.7 Hz, 1H, H5), 8.13 (*ddd*, *J* = 8.4, 1.4, 0.7 Hz, 1H, H8), 7.82–7.79 (*m*, 1H, H4′), 7.78 (*ddd*, *J* = 8.4, 6.8, 1.4 Hz, 1H, H′), 7.61 (*ddd*, *J* = 8.3, 6.9, 1.3 Hz, 1H, H6), 7.53–7.51 (*m*, 1H, H″), 7.51–7.49 (*m*, 2H, H6″, H2″), 7.49 (*d*, *J* = 16.6 Hz, 1H, H_AC═), 7.41–7.38 (*m*, 2H, H3″, H5″), 7.33–7.29 (*m*, 1H, H6′), 7.27 (*ddd*, *J* = 8.5, 7.2, 1.0 Hz, 1H, H5′), 7.09 (*d*, *J* = 16.6 Hz, 1H, ═CH_B), 4.59 (*q*, *J* = 7.2 Hz, 2H, –OCH_2–), 1.39 (*t*, *J* = 7.1 Hz, 3H, -CH_3); δ(^{13}C) 168.0 (C═O), 149.3 (C2), 147.2 (C8a), 144.6 (C3′a), 143.9 (C2′), 142.9 (C4), 137.7 (═CH_B), 134.7 (C4″), 133.6 (C1″, C7′a), 130.8 (C7), 129.7 (C8), 129.1 (C3″, C5″), 128.2 (C2″, C6″), 127.9 (C6), 126.1 (C4a), 125.5 (C5), 125.4 (C3), 124.4 (C6′), 122.5 (C5′), 121.9 (H_AC═), 121.0 (C4′), 111.1 (C7′), 62.2 (–OCH_2–), 14.1 (–CH_3). HRMS (ESI$^+$) *m*/*z* found for $[M + H]^+$ 454.1317, $C_{27}H_{20}ClN_3O_2$ requires 454.1317.

Compound (IIIb), ethyl (*E*)-2-(1*H*-benzo[*d*]imidazol-2-yl)-4-(2-methoxystyryl)quinoline-3-carboxylate; yield: 0.12 g (65%); m.p. 452–453 K, R_F = 0.30 (15% ethyl acetate–hexane). FT–IR (ATR, cm^{-1}): 3432 (N—H), 1713 (C═O), 1568 (C═N), 1487 (C═C_{vinyl}), 1467 (C═C_{arom}), 1439 (C═C_{arom}), 984 (═C—H_{trans}). NMR ($CDCl_3$): δ(^{1}H) 10.72 (*s*, 1H, N—H), 8.25 (*ddd*, *J* = 8.5, 1.4, 0.6 Hz, 1H, H5), 8.12 (*ddd*, *J* = 8.6, 1.4, 0.6 Hz, 1H, H8), 7.82–7.80 (*m*, 1H, H4′), 7.77 (*ddd*, *J* = 8.3, 6.8, 1.4 Hz, 1H, H7), 7.66 (*dd*, *J* = 7.7, 1.7 Hz, 1H, H6″), 7.60 (*ddd*, *J* = 8.3, 6.8,

1.3 Hz, 1H, H6), 7.58 (*d*, *J* = 16.7 Hz, 1H, H_AC═), 7.52–7.50 (*m*, 1H, H7′), 7.50 (*d*, *J* = 16.7 Hz, 1H, ═CH_B), 7.35 (*ddd*, *J* = 8.3, 7.4, 1.7 Hz, 1H, H4″), 7.32–7.24 (*m*, 2H, H5′, H6′), 7.03 (*td*, *J* = 7.5, 1.1 Hz, 1H, H5″), 6.95 (*dd*, *J* = 8.3, 1.1 Hz, 1H, H3″), 4.62 (*q*, *J* = 7.2 Hz, 2H, –OCH_2–), 3.89 (*s*, 3H, 2″-OCH_3), 1.41 (*t*, *J* = 7.2 Hz, 3H, –CH_3); δ(^{13}C) 168.2 (C═O_{ester}), 157.7 (C2′), 149.5 (C2), 147.2 (C8a), 144.7 (C3′a), 144.1 (C2″), 143.9 (C4), 134.3 (═CH_B), 133.5 (C7′a), 130.6 (C7), 130.0 (C4″), 129.6 (C8), 127.7 (C6), 127.4 (C6″), 126.4 (C4a), 125.8 (C5), 125.4 (C1″), 125.2 (C3), 124.2 (C6′), 122.3 (C5′), 121.8 (H_AC═), 121.0 (C4′), 120.9 (C5″), 111.1 (C7′), 62.0 (–OCH_2–), 55.5 (2″-OCH_3), 14.0 (–CH_3). HRMS (ESI$^+$) *m*/*z* found for $[M + H]^+$ 450.1815, $C_{28}H_{23}N_3O_3$ requires 450.1812.

Compound (IIIc), ethyl (*E*)-2-(1*H*-benzo[*d*]imidazol-2-yl)-4-(4-methylstyryl)quinoline-3-carboxylate; yield 0.135 g (65%); m.p. 418–419 K; R_F = 0.30 (15% ethyl acetate–hexane). FT–IR (ATR, cm^{-1}): 3439 (N—H), 1705 (C═O), 1564 (C═N), 1510 (C═C_{vinyl}), 1494 (C═C_{arom}), 1434 (C═C_{arom}), 976 (═C—H_{trans}). NMR ($CDCl_3$): δ(^{1}H) 13.05 (*s*, 1H, N—H), 8.31 (*d*, *J* = 8.4 Hz, 1H, H5), 8.22 (*d*, *J* = 8.4 Hz, 1H, H8), 7.94 (*t*, *J* = 7.6 Hz, 1H, H7), 7.75 (*t*, *J* = 7.6 Hz, 1H, H6), 7.68–7.64 (*m*, 2H, H4′, H7′), 7.64 (*d*, *J* = 16.5 Hz, 1H, H_AC═), 7.61–7.59 (*m*, 2H, H2″, H6″), 7.31–7.24 (*m*, 4H, H5′, H6′, H3″, H5″), 7.08 (*d*, *J* = 16.5 Hz, 1H, ═CH_B), 4.42 (*q*, *J* = 7.2 Hz, 2H, –OCH_2–), 2.36 (*s*, 3H, 4″-CH_3), 1.27 (*t*, *J* = 7.1 Hz, 3H, –CH_3); δ(^{13}C) 167.6 (C═O), 149.7 (C2), 147.1 (C8a), 144.8 (C2′), 144.1 (C3′a), 143.5 (C4), 139.1 (C4″), 138.5 (═CH_B), 135.1 (C1″), 133.7 (C7′a), 131.8 (C7), 129.9 (C3″, C5″), 129.7 (C8), 128.7 (C6), 127.6 (C2″, C6″), 126.3 (C5), 125.9 (C4a), 125.4 (C3), 124.3 (C6′), 122.6 (C5′), 120.9 (H_AC═), 120.2 (C4′), 112.7 (C7′), 61.8 (–OCH_2–), 21.4 (4″-CH_3), 14.4 (–CH_3). HRMS (ESI$^+$) *m*/*z* found for $[M + H]^+$ 434.1860, $C_{28}H_{23}N_3O_2$ requires 434.1863.

2.2. Refinement

Crystal data, data collection and refinement details for compounds (IIIa)–(IIIc) are summarized in Table 1. For each of these compounds, one bad outlier reflection, *i.e.* $\bar{3}96$ for (IIIa), 303 for (IIIb) and $\bar{1}05$ for (IIIc), was omitted from the data set. All H atoms were located in difference maps. H atoms bonded to C atoms were then treated as riding atoms in geometrically idealized positions, with C—H distances of 0.95 (alkenic and aromatic), 0.98 (CH_3) or 0.99 Å (CH_2) and with U_{iso}(H) = kU_{eq}(C), where *k* = 1.5 for the methyl groups, which were permitted to rotate but not to tilt, and 1.2 for all other H atoms. For the H atoms bonded to N atoms, the atomic coordinates were refined with U_{iso}(H) = 1.2U_{eq}(N), giving the N—H distances shown in Table 3.

For compound (IIIa), the final difference map contained one fairly large maximum, 0.61 e Å^{-3}, at 0.5175, 0.8375, 0.7907. An attempt to treat this as the O atom of a partial-occupancy water molecule gave a refined occupancy of 0.057 (3), but the angles subtended at this site by every pair of potential donors and/or acceptors which were within plausible hydrogen-bonding range were all less than 60°, some of them barely half the idealized tetrahedral value. Accordingly, this possibility was discounted.

For compound (IIIc), the crystals were consistently of poor quality; this compound crystallizes in the space group $P2_1/n$ with Z' = 3 and, for the best crystal examined, the R_{int} value was 0.176. In molecule 1 of (IIIc), containing atom N11, the ester group is disordered over two sets of atomic sites having unequal occupancy. For the minor disorder component, the bonded distances and the [1,3] nonbonded distances were restrained to have the same values as the corresponding distances in the major component, subject to s.u. values of 0.01 and 0.02 Å, respectively. In addition, the anisotropic displacement parameters for pairs of partial-occupancy atoms within essentially the same physical space were constrained to be equal. Conventional refinement then converged only to R_1 = 0.132 and wR_2 = 0.391, and examination of the structure of (IIIc) at this point using *PLATON* (Spek, 2020) confirmed that no additional crystallographic symmetry was present and that twinning was also absent. However, *PLATON* showed that the structure formed by the molecules of (IIIc) enclosed two voids, centred at (0,0,0) and ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$) and each of volume *ca* 314 Å^3, and that corresponding voids in unit cells related by translation along [010] are connected, thus forming continuous channels along (0, *y*, 0) and ($\frac{1}{2}$, *y*, $\frac{1}{2}$). Further examination of this structure using the SQUEEZE procedure (Spek, 2015) indicated that each void contained around 55 electrons not hitherto accounted for, equivalent to just over one molecule of hexane per void. The largest peaks in the difference map for (IIIc) lie within the channels, in the form of a zigzag chain, but no convincing solvent model could be developed from these peaks. It seems possible that the channels contain partial-occupancy disordered and possibly mobile hexane molecules. Accordingly, the reflection data were subjected to the SQUEEZE procedure (Spek, 2015), and the resultant modified reflection file was used for the refinement reported here; the final refined values of the site-occupancy factors for the disordered ester group were 0.765 (7) and 0.235 (7).

3. Results and discussion

The synthesis of the hybrid products (IIIa)–(IIIc) (see Scheme 1) starts from the precursor ethyl (*E*)-2-methyl-4-styrylquinoline-3-carboxylates (Ia)–(Ic), using methods recently reported by us (Meléndez *et al.*, 2020). The conversion of precursors (Ia)–(Ic) to the formyl intermediates (IIa)–(IIc) was effected by selective oxidation of the 2-methyl group using selenium dioxide in refluxing 1,4-dioxane as the oxidant (Yaragorla & Vijaya Babu, 2017). The intermediates were isolated in yields of over 90%, and the conversion of the 2-methyl group to a 2-formyl group was confirmed by both the ^{1}H and ^{13}C NMR spectra (see Section 2.1) Finally, the formyl intermediates (IIa)–(IIc) were successfully converted into the target hybrid products (IIIa)–(IIIc) in yields of 65% by means of an oxidative cyclocondensation reaction with *o*-phenylenediamine (1,2-diaminobenzene), promoted by cerium(IV) ammonium nitrate (CAN) (see Scheme 1).

Compounds (IIa)–(IIc) and (IIIa)–(IIIc) were all fully characterized using IR, ^{1}H and ^{13}C NMR spectroscopy, and high-resolution mass spectrometry (see Section 2.1). The

Table 2
Selected torsion and dihedral angles (°) for compounds (IIIa)–(IIIc).

The term dihedral here refers to the dihedral angle between the pyridine and the imidazole rings. In order to specify an asymmetric unit in which the three independent molecules of (IIIc) were linked by hydrogen bonds, it was necessary to select molecule 2 (x = 2) to be the conformational enantiomer opposite from those selected for molecules 1 and 3 (x = 1 and 3) (see text). For ease of comparison, the values of the torsion angles cited for x = 2 refer to the inverted molecule at ($-x$, $-y$, $-z$) so that the values refer to corresponding conformational enantiomers for all three molecules, with positive values for the torsion angles Cx3—Cx4—Cx41—Cx41.

Compounds (IIIa) and (IIIb)		
Parameter	(IIIa)	(IIIb)
N1—C2—C22—N21	10.95 (18)	8.2 (2)
C2—C3—C31—O31	−99.75 (18)	−98.1 (3)
C2—C3—C31—O32	81.29 (15)	85.2 (2)
C3—C4—C41—C42	53.3 (2)	49.8 (3)
C41—C42—C421—C422	169.18 (14)	−167.1 (2)
Dihedral	11.62 (1)	8.52 (3)

Compound (IIIc)			
Parameter	x = 1	x = 2	x = 3
Nx1—Cx2—Cx22—Nx21	9.0 (5)	31.1 (4)	22.1 (4)
Cx2—Cx3—Cx31—Ox31	84.4 (5)	−103.2 (4)	−102.2 (4)
Cx2—Cx3—Cx31—Ox32	−97.2 (4)	78.9 (4)	78.3 (4)
Cx3—Cx4—Cx41—Cx42	65.6 (6)	42.9 (5)	47.0 (5)
Cx41—Cx42—Cx43—Cx44	−168.5 (4)	155.1 (4)	171.9 (4)
Dihedral	12.4 (3)	32.26 (11)	23.24 (18)

formation of the required benzimidazole–quinoline molecular hybrid products (IIIa)–(IIIc) was confirmed by the disappearance of the formyl signals from both the ^{1}H and ^{13}C NMR spectra, and their replacement by new sets of signals corresponding to the five H atoms and seven C atoms of the newly formed benzimidazole ring, and by the appearance of new signals in the IR spectra corresponding to the N—H unit of the newly-formed benzimidazole ring.

The precursors of type (I) were prepared (Meléndez *et al.*, 2020; Rodríguez *et al.*, 2020) using a two-step reaction sequence starting from 2-aminoacetophenone, a substituted benzaldehyde and a 1,3-dicarbonyl compound. With such simple starting materials, a wide range of substituted derivatives is readily available, opening the way to the formation of a rich and diverse library of substituted styrylquinoline–benzimidazole products and their analogues.

The constitutions of compounds (IIIa)–(IIIc), which were deduced from the spectroscopic data, were fully confirmed by the results of single-crystal X-ray diffraction (Figs. 1–3), which additionally provided information on the molecular conformations and the intermolecular interactions in the solid state. Compound (IIIc) crystallizes with Z' = 3, but a search for possible additional crystallographic symmetry revealed none; it will be convenient to refer to the molecules of (IIIc) containing atoms N11, N21 and N31 (Fig. 3) as molecules 1–3, respectively.

The molecules of compounds (IIIa)–(IIIc) exhibit no internal symmetry, as indicated by the key torsion angles (Table 2). They are thus not superimposable upon their mirror images and hence they are conformationally chiral (Moss, 1996; Flack & Bernardinelli, 1999). In each compound, the styrylquinoline fragment is nonplanar, as indicated by the values of the C3—C4—C41—C42 torsion angle (Table 2). We have noted previously (Vera *et al.*, 2022) that 4-styrylquinoline derivatives typically have nonplanar skeletons, whereas 2-styrylquinolines and 8-styrylquinolines typically have planar skeletons.

In all the molecules of (IIIa)–(IIIc), the benzimidazole fragments have the N—H unit directed away from the ester group, so precluding the possibility intramolecular N—H···O

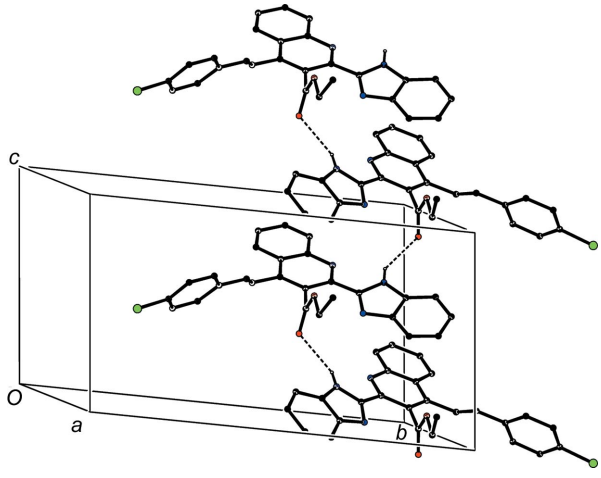


Figure 4
Part of the crystal structure of compound (IIIa), showing the formation of a $C(7)$ chain built from N—H···O hydrogen bonds and running parallel to the [001] direction. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms not involved in the motif shown have been omitted.

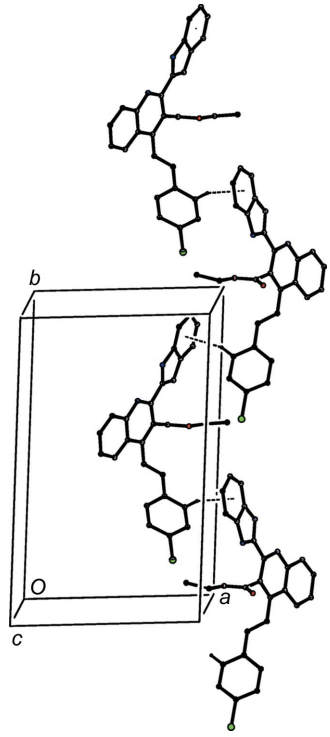


Figure 5
Part of the crystal structure of compound (IIIa), showing the formation of a chain built from C—H···π hydrogen bonds and running parallel to the [010] direction. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms not involved in the motif shown have been omitted.

Table 3
Hydrogen bonds (Å, °) for compounds (IIIa)–(IIIc).

*Cg*1–*Cg*5 represent the centroids of the rings C23*A*/C24–C27/C27*A*, N1/C2–C4/C4*A*/C8*A*, C421–C426, C24*A*/C25–C28/C28*A* and C23*A*/C27*A*/C227/C226/C225/C224, respectively.

	D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
(IIIa)	N21—H21···O31^{i}	0.832 (19)	2.379 (18)	3.0764 (16)	141.8 (16)
	C422—H422···*Cg*1^{ii}	0.95	2.53	3.4420 (17)	168
	C426—H426···*Cg*2^{iii}	0.95	2.68	3.5161 (18)	148
(IIIb)	N21—H21···O31^{iv}	0.86 (3)	2.45 (2)	3.081 (3)	130.4 (19)
	C7—H7···*Cg*3^{iii}	0.95	2.95	3.704 (2)	138
	C33—H33*B*···*Cg*3^{v}	0.98	2.98	3.768 (3)	139
(IIIc)	N121—H121···O231	0.78 (4)	2.17 (4)	2.876 (4)	150 (4)
	N221—H221···O331	0.83 (4)	2.13 (4)	2.882 (4)	151 (4)
	N321—H321···O131^{vi}	0.84 (4)	2.20 (4)	2.910 (4)	141 (3)
	N321—H321···O431^{vi}	0.84 (4)	2.35 (5)	4.03 (3)	137 (3)
	C127—H127···N223	0.95	2.58	3.432 (5)	149
	C227—H227···N323	0.95	2.62	3.522 (5)	159
	C332—H32*B*···*Cg*4^{iv}	0.99	2.80	3.702 (5)	152
	C347—H347···*Cg*5^{vii}	0.95	2.70	3.548 (5)	149

Symmetry codes: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$; (iii) $-x+1, -y+1, -z+1$; (iv) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (v) $-x, -y+1, -z+1$; (vi) $x-1, y, z$; (vii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$.

hydrogen bonding; the pyridine and imidazole rings are not coplanar, as shown by the dihedral angles between their planes (Table 2). In one of the molecules of (IIIc), the ester group is disordered over two sets of atomic sites, having occupancies 0.765 (7) and 0.235 (7) [see Fig. 3(*a*)]. The only 2-benzimidazolylquinoline derivatives recorded in the Cambridge Structural Database (CSD; Groom *et al.*, 2016) are titanium complexes in which the quinolone N atom and one of the imidazole N atoms are both coordinated to Ti, forming a five-membered ring, and hence the conformations of the organic ligands in these compounds are not usefully comparable with those in metal-free systems. The orientations of the ester groups relative to the pyridine ring may be a consequence of the N—H···O hydrogen bond (see below), as in every molecule in (IIIa)–(IIIc), the carbonyl O atom acts as an acceptor in such an interaction.

While compounds (IIIa) and (IIIb) crystallize in the solvent-free form, compound (IIIc) contains disordered solvent within continuous channels; hence, it is to be expected that the supramolecular assembly for (IIIc) will differ from those of (IIIa) and (IIIb), as the Z' value immediately indicates.

For compound (IIIa), the supramolecular assembly is based upon three hydrogen bonds, one of the N—H···O type and two of the C—H···π type (Table 2), and the combination of these three interactions links the molecules of (IIIa) into a three-dimensional framework structure. However, the formation of the framework is readily analysed in terms of three simple substructures (Ferguson *et al.*, 1998*a*,*b*; Gregson *et al.*, 2000), each involving just one type of hydrogen bond.

In the first substructure, molecules of (IIIa) which are related by the *c*-glide plane at $y=\frac{3}{4}$ are linked by N—H···O to form a *C*(7) (Etter, 1990; Etter *et al.*, 1990; Bernstein *et al.*, 1995) chain running parallel to the [001] direction (Fig. 4). A second substructure involves the C—H···π hydrogen bond having atom C422 as the donor (Table 2); this interaction links molecules of (IIIa) which are related by the 2_1 screw axis along

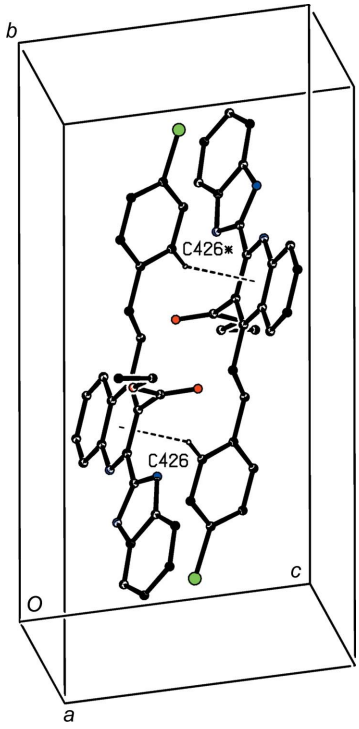


Figure 6
Part of the crystal structure of compound (IIIa), showing the formation of a cyclic centrosymmetric motif built from C—H···π hydrogen bonds and linking adjacent (100) sheets. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms not involved in the motif shown have been omitted. The atom marked with an asterisk (*) is at the symmetry position $(-x+1, -y+1, -z+1)$.

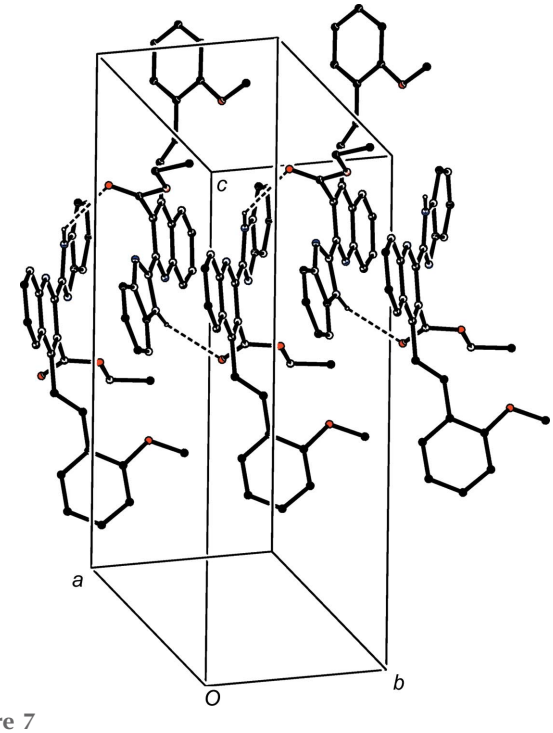


Figure 7
Part of the crystal structure of compound (IIIb), showing the formation of a *C*(7) chain built from N—H···O hydrogen bonds and running parallel to the [010] direction. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms not involved in the motif shown have been omitted.

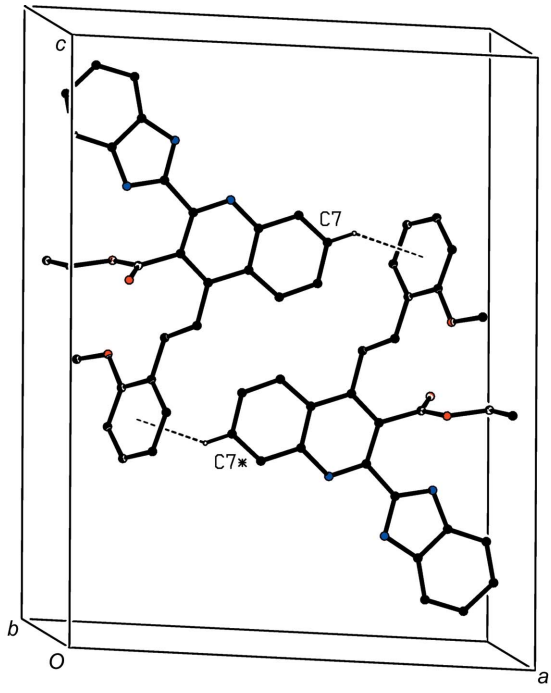


Figure 8
Part of the crystal structure of compound (IIIb), showing the formation of a cyclic centrosymmetric motif built from C—H···π hydrogen bonds and linking adjacent [010] chains. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms not involved in the motif shown have been omitted. The atom marked with an asterisk (*) is at the symmetry position ($-x+1, -y+1, -z+1$).

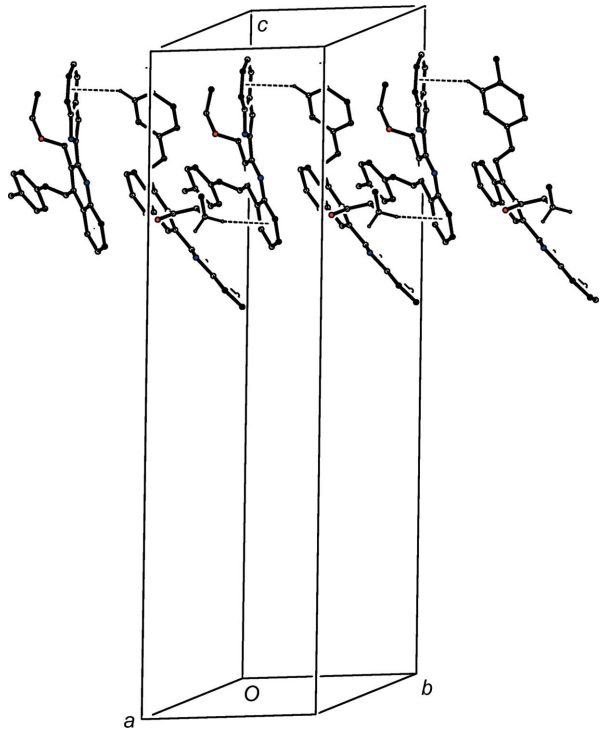


Figure 10
Part of the crystal structure of compound (IIIc), showing the formation of a chain parallel to [010] built from C—H···π hydrogen bonds (drawn as dashed lines). For the sake of clarity, the minor disorder component and the H atoms bonded to those atoms not involved in the motif shown have been omitted.

(1, y, $\frac{3}{4}$) to form a chain running parallel to the [010] direction (Fig. 5). The combination of the chains along [010] and [001] gives rise to a sheet lying parallel to (100). Adjacent sheets are then linked by the third substructure, which is built from C—H···π hydrogen bonds having atom C426 as the donor, which links inversion-related molecules from adjacent sheets (Fig. 6), so completing the three-dimensional assembly.

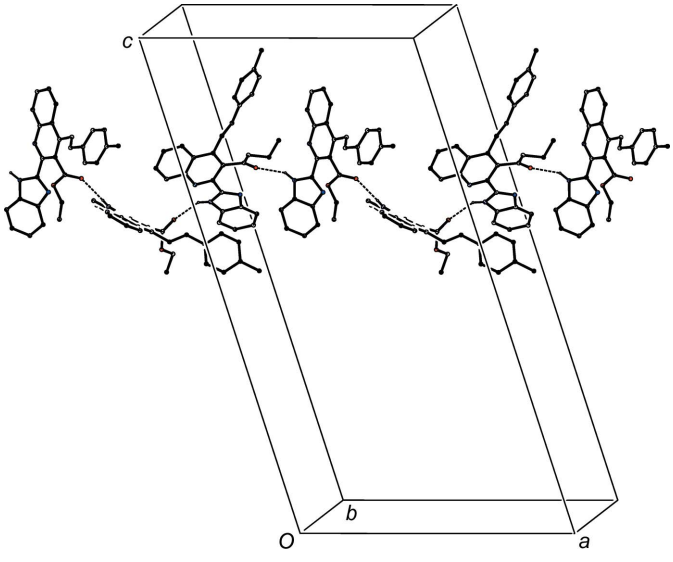


Figure 9
Part of the crystal structure of compound (IIIc), showing the formation of a $C_3^3(21)$ chain built from N—H···O hydrogen bonds and running parallel to the [100] direction. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, the minor disorder component and the H atoms not involved in the motif shown have been omitted.

An N—H···O hydrogen bond is also present in the structure of compound (IIIb) (Table 3), and this links molecules which are related by the 2_1 screw axis along ($\frac{1}{4}$, y, $\frac{3}{4}$) to form a $C(7)$ chain running parallel to the [010] direction (Fig. 7). The C—H···π hydrogen bond (Table 3) links inversion-related molecules in adjacent chains into a cyclic centrosymmetric motif (Fig. 8), which links the [010] chains into a sheet lying parallel to (101). There are no direction-specific interactions between adjacent sheets in (IIIb); the only other short intermolecular contact in the structure involves a C—H bond in a methyl group, which is almost certainly undergoing rapid rotation about the adjacent C—C bond (Riddell & Rogerson, 1996, 1997).

The hydrogen-bonded supramolecular assembly in compound (IIIc), where Z' = 3, is also two-dimensional and can readily be analysed in terms of two simple substructures. In the first of these, the three independent N—H···O hydrogen bonds of compound (IIIc) are linked by two N—H···O hydrogen bonds (Table 3) to form a linear three-molecule aggregate, and aggregates of this type which are related by translation are linked by a third N—H···O hydrogen bond to form a $C_3^3(21)$ chain running parallel to the [100] direction (Fig. 9). The formation of this chain is thus analogous to those formed in compounds (IIIa) and (IIIb) (Figs. 4 and 7), but it is interesting to note that the components of the chains formed

by N—H···O hydrogen bonds are related by a *c*-glide plane in (IIIa), by a 2_1 screw axis in (IIIb) and by translation in (IIIc).

The second substructure in (IIIc) is built from two C—H···π hydrogen bonds in which molecule 3 acts as a twofold donor and molecule 2 acts as a twofold acceptor. These two interactions generate a chain running parallel to the [010] direction (Fig. 10). The combination of the chains running parallel to [100] and [010] generates a sheet lying parallel to (001) and occupying the domain $\frac{1}{2} < z < 1.0$; a second sheet, related to the first by inversion, occupies the domain $0 < z < \frac{1}{2}$, but there are no direction-specific interactions between adjacent sheets.

In summary, therefore, we have developed an efficient and versatile synthetic route to novel hybrid (*E*)-2-(1*H*-benzo[*d*]imidazol-2-yl)-4-styrylquinolines from very simple starting materials; we have fully characterized by spectroscopic means (IR, ^{1}H and ^{13}C NMR spectroscopy, and HR-MS) three representative examples, together with one intermediate on the pathway to each product, and we have determined the molecular and supramolecular structures of the three products thus formed.

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supporting information

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Synthesis, and spectroscopic and structural characterization of three new styrylquinoline–benzimidazole hybrids

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Computing details

For all structures, data collection: *APEX3* (Bruker, 2018); cell refinement: *SAINT* (Bruker, 2017); data reduction: *SAINT* (Bruker, 2017); program(s) used to solve structure: SHELXT2014 (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *PLATON* (Spek, 2020); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015b) and *PLATON* (Spek, 2020).

Ethyl (*E*)-2-(1*H*-benzo[*d*]imidazol-2-yl)-4-(4-chlorostyryl)quinoline-3-carboxylate (IIIa)

Crystal data

$C_{27}H_{20}ClN_3O_2$
$M_r = 453.91$
Monoclinic, $P2_1/c$
$a = 12.1791$ (5) Å
$b = 18.8348$ (7) Å
$c = 10.7533$ (4) Å
$\beta = 114.242$ (1)°
$V = 2249.19$ (15) Å^3
$Z = 4$
$F(000) = 944$
$D_x = 1.340$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4970 reflections
$\theta = 2.1$–27.1°
$\mu = 0.20$ mm^{-1}
$T = 100$ K
Block, yellow
0.18 × 0.16 × 0.12 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
$T_{min} = 0.928$, $T_{max} = 0.976$
56016 measured reflections
4969 independent reflections
4417 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$
$\theta_{max} = 27.1°$, $\theta_{min} = 2.1°$
$h = -15 \rightarrow 15$
$k = -24 \rightarrow 24$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.096$
$S = 1.05$
4969 reflections
302 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0407P)^2 + 1.3439P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.61$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ e Å^{-3}

Special details

Geometry. 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.61736 (10)	0.70058 (6)	0.74826 (11)	0.0183 (2)
C2	0.68196 (12)	0.68530 (7)	0.67887 (13)	0.0167 (3)
C3	0.69721 (12)	0.61526 (7)	0.63775 (13)	0.0175 (3)
C4	0.64259 (12)	0.55932 (7)	0.67335 (14)	0.0187 (3)
C4A	0.57161 (12)	0.57459 (8)	0.74822 (14)	0.0208 (3)
C5	0.50806 (14)	0.52179 (9)	0.78603 (17)	0.0284 (3)
H5	0.5130	0.4735	0.7633	0.034*
C6	0.43975 (15)	0.54013 (10)	0.85510 (18)	0.0342 (4)
H6	0.3965	0.5044	0.8785	0.041*
C7	0.43243 (15)	0.61107 (10)	0.89209 (18)	0.0337 (4)
H7	0.3852	0.6227	0.9410	0.040*
C8	0.49288 (14)	0.66333 (9)	0.85805 (16)	0.0264 (3)
H8	0.4881	0.7111	0.8836	0.032*
C8A	0.56266 (12)	0.64610 (8)	0.78455 (14)	0.0195 (3)
N21	0.73895 (11)	0.81164 (6)	0.70447 (12)	0.0181 (2)
H21	0.7141 (16)	0.8189 (9)	0.7648 (18)	0.022*
C22	0.73830 (12)	0.74719 (7)	0.64549 (13)	0.0168 (3)
N23	0.78911 (10)	0.74835 (6)	0.55841 (12)	0.0171 (2)
C23A	0.82380 (12)	0.81859 (7)	0.55848 (13)	0.0163 (3)
C24	0.87973 (12)	0.85111 (7)	0.48286 (14)	0.0184 (3)
H24	0.9026	0.8244	0.4223	0.022*
C25	0.90064 (13)	0.92333 (8)	0.49918 (14)	0.0209 (3)
H25	0.9366	0.9468	0.4471	0.025*
C26	0.86988 (13)	0.96280 (7)	0.59119 (15)	0.0224 (3)
H26	0.8859	1.0123	0.6000	0.027*
C27	0.81683 (13)	0.93144 (7)	0.66942 (15)	0.0204 (3)
H27	0.7978	0.9580	0.7331	0.025*
C27A	0.79275 (12)	0.85891 (7)	0.64982 (14)	0.0176 (3)
C31	0.76581 (13)	0.60392 (7)	0.55054 (14)	0.0193 (3)
O31	0.71791 (10)	0.59501 (6)	0.42844 (10)	0.0248 (2)
O32	0.88455 (9)	0.60386 (5)	0.62542 (10)	0.0206 (2)
C32	0.95836 (14)	0.60314 (9)	0.54758 (16)	0.0277 (3)
H32A	0.9318	0.6410	0.4775	0.033*
H32B	0.9505	0.5568	0.5011	0.033*
C33	1.08654 (15)	0.61522 (10)	0.64479 (17)	0.0319 (4)
H33A	1.0945	0.6625	0.6855	0.048*
H33B	1.1386	0.6118	0.5956	0.048*

H33C	1.1105	0.5792	0.7169	0.048*
C41	0.65296 (13)	0.48504 (7)	0.63462 (14)	0.0214 (3)
H41	0.5804	0.4593	0.5885	0.026*
C42	0.75590 (13)	0.45149 (7)	0.65934 (14)	0.0203 (3)
H42	0.8284	0.4770	0.7075	0.024*
C421	0.76708 (13)	0.37824 (7)	0.61848 (14)	0.0196 (3)
C422	0.87940 (13)	0.34507 (8)	0.67006 (15)	0.0220 (3)
H422	0.9472	0.3696	0.7339	0.026*
C423	0.89420 (13)	0.27699 (8)	0.63010 (15)	0.0236 (3)
H423	0.9710	0.2548	0.6672	0.028*
C424	0.79541 (14)	0.24190 (7)	0.53548 (15)	0.0213 (3)
Cl44	0.81186 (4)	0.15782 (2)	0.47785 (4)	0.02788 (11)
C425	0.68187 (14)	0.27275 (8)	0.48351 (16)	0.0257 (3)
H425	0.6144	0.2478	0.4198	0.031*
C426	0.66857 (14)	0.34035 (8)	0.52589 (16)	0.0263 (3)
H426	0.5909	0.3615	0.4914	0.032*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0156 (5)	0.0207 (6)	0.0172 (5)	−0.0011 (4)	0.0053 (5)	−0.0001 (4)
C2	0.0158 (6)	0.0183 (6)	0.0142 (6)	−0.0012 (5)	0.0043 (5)	0.0004 (5)
C3	0.0168 (6)	0.0181 (6)	0.0153 (6)	−0.0009 (5)	0.0043 (5)	−0.0002 (5)
C4	0.0161 (6)	0.0184 (6)	0.0172 (6)	−0.0011 (5)	0.0024 (5)	0.0011 (5)
C4A	0.0162 (6)	0.0239 (7)	0.0192 (6)	−0.0028 (5)	0.0042 (5)	0.0023 (5)
C5	0.0246 (8)	0.0277 (8)	0.0320 (8)	−0.0067 (6)	0.0108 (7)	0.0028 (6)
C6	0.0264 (8)	0.0409 (9)	0.0389 (9)	−0.0101 (7)	0.0171 (7)	0.0059 (7)
C7	0.0240 (8)	0.0480 (10)	0.0352 (9)	−0.0042 (7)	0.0185 (7)	0.0015 (7)
C8	0.0209 (7)	0.0343 (8)	0.0258 (7)	−0.0009 (6)	0.0114 (6)	−0.0012 (6)
C8A	0.0149 (6)	0.0249 (7)	0.0171 (6)	−0.0020 (5)	0.0049 (5)	0.0014 (5)
N21	0.0198 (6)	0.0175 (6)	0.0200 (6)	−0.0007 (4)	0.0112 (5)	−0.0014 (4)
C22	0.0160 (6)	0.0169 (6)	0.0164 (6)	0.0000 (5)	0.0053 (5)	−0.0007 (5)
N23	0.0173 (5)	0.0165 (5)	0.0172 (5)	−0.0012 (4)	0.0069 (5)	0.0005 (4)
C23A	0.0141 (6)	0.0159 (6)	0.0162 (6)	0.0006 (5)	0.0035 (5)	−0.0002 (5)
C24	0.0165 (6)	0.0212 (7)	0.0167 (6)	−0.0009 (5)	0.0061 (5)	−0.0010 (5)
C25	0.0193 (7)	0.0212 (7)	0.0214 (7)	−0.0019 (5)	0.0077 (6)	0.0028 (5)
C26	0.0225 (7)	0.0155 (6)	0.0279 (7)	0.0000 (5)	0.0091 (6)	0.0006 (5)
C27	0.0209 (7)	0.0164 (6)	0.0248 (7)	0.0022 (5)	0.0102 (6)	−0.0011 (5)
C27A	0.0143 (6)	0.0179 (6)	0.0196 (6)	0.0010 (5)	0.0061 (5)	0.0014 (5)
C31	0.0246 (7)	0.0127 (6)	0.0218 (7)	−0.0018 (5)	0.0108 (6)	−0.0005 (5)
O31	0.0320 (6)	0.0236 (5)	0.0198 (5)	−0.0064 (4)	0.0116 (4)	−0.0048 (4)
O32	0.0226 (5)	0.0209 (5)	0.0216 (5)	0.0021 (4)	0.0124 (4)	0.0016 (4)
C32	0.0301 (8)	0.0320 (8)	0.0293 (8)	0.0015 (6)	0.0208 (7)	−0.0018 (6)
C33	0.0279 (8)	0.0438 (10)	0.0290 (8)	0.0063 (7)	0.0167 (7)	0.0097 (7)
C41	0.0223 (7)	0.0174 (6)	0.0210 (7)	−0.0040 (5)	0.0053 (6)	0.0011 (5)
C42	0.0211 (7)	0.0190 (7)	0.0178 (6)	−0.0035 (5)	0.0048 (5)	0.0003 (5)
C421	0.0221 (7)	0.0189 (7)	0.0173 (6)	−0.0007 (5)	0.0076 (5)	0.0021 (5)
C422	0.0181 (7)	0.0250 (7)	0.0221 (7)	−0.0029 (5)	0.0076 (6)	0.0002 (5)

C423	0.0185 (7)	0.0254 (7)	0.0280 (8)	0.0010 (6)	0.0105 (6)	0.0016 (6)
C424	0.0258 (7)	0.0182 (7)	0.0235 (7)	0.0001 (5)	0.0138 (6)	0.0014 (5)
Cl44	0.0312 (2)	0.02163 (18)	0.0356 (2)	0.00184 (14)	0.01853 (17)	−0.00355 (14)
C425	0.0233 (7)	0.0209 (7)	0.0256 (7)	−0.0006 (6)	0.0026 (6)	−0.0017 (6)
C426	0.0211 (7)	0.0220 (7)	0.0279 (8)	0.0029 (6)	0.0021 (6)	−0.0005 (6)

Geometric parameters (Å, º)

N1—C2	1.3192 (18)	C26—C27	1.386 (2)
N1—C8A	1.3648 (18)	C26—H26	0.9500
C2—C3	1.4276 (19)	C27—C27A	1.3950 (19)
C2—C22	1.4699 (18)	C27—H27	0.9500
C3—C4	1.3814 (19)	C31—O31	1.2097 (17)
C3—C31	1.5054 (19)	C31—O32	1.3355 (18)
C4—C4A	1.432 (2)	O32—C32	1.4584 (17)
C4—C41	1.4798 (19)	C32—C33	1.497 (2)
C4A—C5	1.418 (2)	C32—H32A	0.9900
C4A—C8A	1.419 (2)	C32—H32B	0.9900
C5—C6	1.368 (2)	C33—H33A	0.9800
C5—H5	0.9500	C33—H33B	0.9800
C6—C7	1.407 (3)	C33—H33C	0.9800
C6—H6	0.9500	C41—C42	1.330 (2)
C7—C8	1.366 (2)	C41—H41	0.9500
C7—H7	0.9500	C42—C421	1.4710 (19)
C8—C8A	1.416 (2)	C42—H42	0.9500
C8—H8	0.9500	C421—C422	1.395 (2)
N21—C22	1.3681 (17)	C421—C426	1.400 (2)
N21—C27A	1.3724 (18)	C422—C423	1.387 (2)
N21—H21	0.832 (19)	C422—H422	0.9500
C22—N23	1.3176 (18)	C423—C424	1.384 (2)
N23—C23A	1.3888 (17)	C423—H423	0.9500
C23A—C24	1.3984 (19)	C424—C425	1.388 (2)
C23A—C27A	1.4104 (19)	C424—Cl44	1.7417 (15)
C24—C25	1.382 (2)	C425—C426	1.384 (2)
C24—H24	0.9500	C425—H425	0.9500
C25—C26	1.406 (2)	C426—H426	0.9500
C25—H25	0.9500		

C2—N1—C8A	118.18 (12)	C26—C27—C27A	116.56 (13)
N1—C2—C3	124.08 (12)	C26—C27—H27	121.7
N1—C2—C22	114.31 (12)	C27A—C27—H27	121.7
C3—C2—C22	121.60 (12)	N21—C27A—C27	132.71 (13)
C4—C3—C2	118.71 (13)	N21—C27A—C23A	105.10 (12)
C4—C3—C31	121.20 (12)	C27—C27A—C23A	122.18 (13)
C2—C3—C31	120.00 (12)	O31—C31—O32	125.06 (13)
C3—C4—C4A	118.24 (13)	O31—C31—C3	123.52 (13)
C3—C4—C41	122.46 (13)	O32—C31—C3	111.42 (11)
C4A—C4—C41	119.29 (12)	C31—O32—C32	115.10 (11)

C5—C4A—C8A	118.29 (14)	O32—C32—C33	108.03 (12)
C5—C4A—C4	123.15 (14)	O32—C32—H32A	110.1
C8A—C4A—C4	118.55 (13)	C33—C32—H32A	110.1
C6—C5—C4A	120.25 (15)	O32—C32—H32B	110.1
C6—C5—H5	119.9	C33—C32—H32B	110.1
C4A—C5—H5	119.9	H32A—C32—H32B	108.4
C5—C6—C7	121.11 (15)	C32—C33—H33A	109.5
C5—C6—H6	119.4	C32—C33—H33B	109.5
C7—C6—H6	119.4	H33A—C33—H33B	109.5
C8—C7—C6	120.31 (15)	C32—C33—H33C	109.5
C8—C7—H7	119.8	H33A—C33—H33C	109.5
C6—C7—H7	119.8	H33B—C33—H33C	109.5
C7—C8—C8A	119.85 (15)	C42—C41—C4	125.13 (13)
C7—C8—H8	120.1	C42—C41—H41	117.4
C8A—C8—H8	120.1	C4—C41—H41	117.4
N1—C8A—C8	117.57 (13)	C41—C42—C421	125.54 (13)
N1—C8A—C4A	122.22 (13)	C41—C42—H42	117.2
C8—C8A—C4A	120.19 (13)	C421—C42—H42	117.2
C22—N21—C27A	106.86 (12)	C422—C421—C426	117.94 (13)
C22—N21—H21	124.9 (12)	C422—C421—C42	119.81 (13)
C27A—N21—H21	128.2 (12)	C426—C421—C42	122.23 (13)
N23—C22—N21	113.56 (12)	C423—C422—C421	121.44 (13)
N23—C22—C2	126.10 (12)	C423—C422—H422	119.3
N21—C22—C2	120.32 (12)	C421—C422—H422	119.3
C22—N23—C23A	104.30 (11)	C424—C423—C422	119.01 (14)
N23—C23A—C24	129.62 (12)	C424—C423—H423	120.5
N23—C23A—C27A	110.17 (12)	C422—C423—H423	120.5
C24—C23A—C27A	120.21 (12)	C423—C424—C425	121.18 (14)
C25—C24—C23A	117.75 (13)	C423—C424—Cl44	120.24 (12)
C25—C24—H24	121.1	C425—C424—Cl44	118.58 (11)
C23A—C24—H24	121.1	C426—C425—C424	118.97 (14)
C24—C25—C26	121.43 (13)	C426—C425—H425	120.5
C24—C25—H25	119.3	C424—C425—H425	120.5
C26—C25—H25	119.3	C425—C426—C421	121.42 (14)
C27—C26—C25	121.83 (13)	C425—C426—H426	119.3
C27—C26—H26	119.1	C421—C426—H426	119.3
C25—C26—H26	119.1		

C8A—N1—C2—C3	0.8 (2)	C22—N23—C23A—C27A	0.58 (15)
C8A—N1—C2—C22	−179.45 (11)	N23—C23A—C24—C25	177.68 (13)
N1—C2—C3—C4	−0.8 (2)	C27A—C23A—C24—C25	−1.25 (19)
C22—C2—C3—C4	179.50 (12)	C23A—C24—C25—C26	1.7 (2)
N1—C2—C3—C31	175.82 (12)	C24—C25—C26—C27	−0.3 (2)
C22—C2—C3—C31	−3.93 (19)	C25—C26—C27—C27A	−1.5 (2)
C2—C3—C4—C4A	0.94 (19)	C22—N21—C27A—C27	179.06 (15)
C31—C3—C4—C4A	−175.59 (12)	C22—N21—C27A—C23A	−0.60 (14)
C2—C3—C4—C41	179.70 (12)	C26—C27—C27A—N21	−177.70 (14)
C31—C3—C4—C41	3.2 (2)	C26—C27—C27A—C23A	1.9 (2)

C3—C4—C4A—C5	177.73 (14)	N23—C23A—C27A—N21	0.03 (15)
C41—C4—C4A—C5	−1.1 (2)	C24—C23A—C27A—N21	179.15 (12)
C3—C4—C4A—C8A	−1.22 (19)	N23—C23A—C27A—C27	−179.68 (12)
C41—C4—C4A—C8A	179.99 (12)	C24—C23A—C27A—C27	−0.6 (2)
C8A—C4A—C5—C6	0.3 (2)	C4—C3—C31—O31	76.74 (18)
C4—C4A—C5—C6	−178.61 (14)	C2—C3—C31—O31	−99.75 (16)
C4A—C5—C6—C7	−1.1 (3)	C4—C3—C31—O32	−102.22 (14)
C5—C6—C7—C8	0.7 (3)	C2—C3—C31—O32	81.29 (15)
C6—C7—C8—C8A	0.4 (2)	O31—C31—O32—C32	9.23 (19)
C2—N1—C8A—C8	−179.52 (13)	C3—C31—O32—C32	−171.82 (11)
C2—N1—C8A—C4A	−1.06 (19)	C31—O32—C32—C33	170.82 (12)
C7—C8—C8A—N1	177.42 (14)	C3—C4—C41—C42	53.3 (2)
C7—C8—C8A—C4A	−1.1 (2)	C4A—C4—C41—C42	−127.99 (16)
C5—C4A—C8A—N1	−177.70 (13)	C4—C41—C42—C421	−178.50 (13)
C4—C4A—C8A—N1	1.3 (2)	C41—C42—C421—C422	−169.18 (14)
C5—C4A—C8A—C8	0.7 (2)	C41—C42—C421—C426	12.4 (2)
C4—C4A—C8A—C8	179.72 (13)	C426—C421—C422—C423	0.8 (2)
C27A—N21—C22—N23	1.05 (16)	C42—C421—C422—C423	−177.66 (13)
C27A—N21—C22—C2	−177.61 (12)	C421—C422—C423—C424	1.0 (2)
N1—C2—C22—N23	−167.50 (13)	C422—C423—C424—C425	−2.0 (2)
C3—C2—C22—N23	12.3 (2)	C422—C423—C424—Cl44	177.41 (11)
N1—C2—C22—N21	10.98 (18)	C423—C424—C425—C426	1.2 (2)
C3—C2—C22—N21	−169.25 (12)	Cl44—C424—C425—C426	−178.24 (12)
N21—C22—N23—C23A	−1.00 (15)	C424—C425—C426—C421	0.7 (2)
C2—C22—N23—C23A	177.57 (13)	C422—C421—C426—C425	−1.6 (2)
C22—N23—C23A—C24	−178.45 (14)	C42—C421—C426—C425	176.78 (14)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N21—H21···O31^{i}	0.832 (19)	2.379 (18)	3.0764 (16)	141.8 (16)
C422—H422···*Cg*1^{ii}	0.95	2.53	3.4420 (17)	168
C426—H426···*Cg*2^{iii}	0.95	2.68	3.5161 (18)	148

Symmetry codes: (i) *x*, −*y*+3/2, *z*+1/2; (ii) −*x*+2, *y*−1/2, −*z*+3/2; (iii) −*x*+1, −*y*+1, −*z*+1.

Ethyl (*E*)-2-(1*H*-benzo[*d*]imidazol-2-yl)-4-(2-methoxystyryl)quinoline-3-carboxylate (IIIb)

Crystal data

$C_{28}H_{23}N_3O_3$
M_r = 449.49
Monoclinic, $P2_1/n$
a = 15.8086 (14) Å
b = 6.9536 (6) Å
c = 20.4101 (19) Å
β = 94.330 (4)°
V = 2237.2 (3) Å^3
Z = 4

$F(000)$ = 944
D_x = 1.335 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5132 reflections
θ = 2.6–27.5°
μ = 0.09 mm^{-1}
T = 100 K
Needle, yellow
0.16 × 0.12 × 0.08 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.817, T_{max} = 0.993
41777 measured reflections
5131 independent reflections
3453 reflections with $I > 2\sigma(I)$
R_{int} = 0.132
θ_{max} = 27.5°, θ_{min} = 2.6°
h = −20→20
k = −8→9
l = −26→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.133
S = 1.06
5131 reflections
312 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0344P)^2 + 1.5171P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Special details

Geometry. 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
N1	0.37557 (11)	0.3293 (2)	0.72892 (9)	0.0195 (4)
C2	0.29526 (13)	0.3281 (3)	0.70558 (11)	0.0186 (4)
C3	0.26834 (13)	0.3275 (3)	0.63748 (11)	0.0186 (4)
C4	0.32852 (13)	0.3338 (3)	0.59196 (11)	0.0182 (4)
C4A	0.41582 (13)	0.3367 (3)	0.61566 (11)	0.0190 (4)
C5	0.48284 (14)	0.3481 (3)	0.57350 (11)	0.0212 (5)
H5	0.4703	0.3529	0.5273	0.025*
C6	0.56541 (14)	0.3524 (3)	0.59865 (12)	0.0240 (5)
H6	0.6096	0.3603	0.5697	0.029*
C7	0.58556 (14)	0.3454 (3)	0.66684 (12)	0.0248 (5)
H7	0.6432	0.3476	0.6837	0.030*
C8	0.52242 (14)	0.3354 (3)	0.70901 (12)	0.0237 (5)
H8	0.5366	0.3302	0.7550	0.028*
C8A	0.43614 (13)	0.3327 (3)	0.68461 (11)	0.0190 (4)
N21	0.25802 (12)	0.3542 (3)	0.82060 (9)	0.0206 (4)
H21	0.3081 (16)	0.383 (3)	0.8370 (12)	0.025*
C22	0.23316 (13)	0.3287 (3)	0.75589 (11)	0.0196 (5)
N23	0.15053 (11)	0.3073 (2)	0.74424 (9)	0.0209 (4)
C23A	0.11934 (14)	0.3222 (3)	0.80584 (11)	0.0202 (5)
C24	0.03559 (14)	0.3150 (3)	0.82354 (12)	0.0238 (5)

H24	−0.0103	0.2947	0.7915	0.029*
C25	0.02187 (15)	0.3381 (3)	0.88899 (12)	0.0259 (5)
H25	−0.0345	0.3347	0.9021	0.031*
C26	0.08929 (15)	0.3665 (3)	0.93656 (12)	0.0252 (5)
H26	0.0774	0.3817	0.9812	0.030*
C27	0.17264 (15)	0.3732 (3)	0.92050 (11)	0.0227 (5)
H27	0.2183	0.3919	0.9529	0.027*
C27A	0.18591 (14)	0.3508 (3)	0.85421 (11)	0.0195 (5)
C31	0.17588 (13)	0.3095 (3)	0.61517 (11)	0.0192 (5)
O31	0.14140 (10)	0.1618 (2)	0.59753 (8)	0.0241 (4)
O32	0.13828 (9)	0.4826 (2)	0.61431 (8)	0.0205 (3)
C32	0.04685 (13)	0.4797 (3)	0.60150 (13)	0.0260 (5)
H32A	0.0203	0.4084	0.6366	0.031*
H32B	0.0310	0.4165	0.5589	0.031*
C33	0.01804 (15)	0.6852 (3)	0.59996 (14)	0.0330 (6)
H33A	0.0361	0.7473	0.6418	0.049*
H33B	−0.0439	0.6899	0.5930	0.049*
H33C	0.0432	0.7529	0.5640	0.049*
C41	0.30552 (13)	0.3388 (3)	0.52082 (11)	0.0203 (5)
H41	0.3322	0.2506	0.4934	0.024*
C42	0.24894 (13)	0.4615 (3)	0.49307 (11)	0.0198 (5)
H42	0.2230	0.5470	0.5218	0.024*
C421	0.22240 (13)	0.4790 (3)	0.42301 (11)	0.0191 (4)
C422	0.17514 (14)	0.6410 (3)	0.40045 (11)	0.0223 (5)
C423	0.14768 (14)	0.6617 (3)	0.33470 (12)	0.0261 (5)
H423	0.1162	0.7722	0.3202	0.031*
C424	0.16638 (15)	0.5204 (4)	0.29022 (12)	0.0283 (5)
H424	0.1469	0.5332	0.2453	0.034*
C425	0.21333 (15)	0.3602 (4)	0.31092 (12)	0.0282 (5)
H425	0.2266	0.2644	0.2802	0.034*
C426	0.24080 (14)	0.3407 (3)	0.37668 (11)	0.0238 (5)
H426	0.2729	0.2306	0.3905	0.029*
O422	0.15917 (11)	0.7714 (2)	0.44836 (8)	0.0311 (4)
C427	0.10924 (16)	0.9370 (3)	0.43045 (14)	0.0325 (6)
H27A	0.1362	1.0092	0.3964	0.049*
H27B	0.1049	1.0190	0.4691	0.049*
H27C	0.0524	0.8966	0.4135	0.049*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0208 (9)	0.0145 (8)	0.0231 (10)	0.0002 (7)	0.0019 (8)	−0.0005 (7)
C2	0.0194 (11)	0.0097 (9)	0.0268 (12)	0.0011 (8)	0.0034 (9)	−0.0009 (8)
C3	0.0192 (11)	0.0106 (9)	0.0262 (12)	0.0004 (8)	0.0017 (9)	−0.0003 (8)
C4	0.0207 (11)	0.0097 (9)	0.0242 (12)	−0.0012 (8)	0.0012 (9)	−0.0001 (8)
C4A	0.0212 (11)	0.0108 (9)	0.0252 (12)	0.0006 (8)	0.0030 (9)	0.0004 (8)
C5	0.0239 (11)	0.0157 (10)	0.0242 (12)	0.0004 (9)	0.0028 (9)	0.0009 (9)
C6	0.0204 (11)	0.0207 (11)	0.0319 (13)	0.0003 (9)	0.0081 (10)	0.0016 (10)

C7	0.0169 (11)	0.0239 (11)	0.0331 (13)	−0.0010 (9)	−0.0016 (9)	0.0024 (10)
C8	0.0225 (11)	0.0242 (11)	0.0239 (12)	−0.0006 (9)	−0.0009 (9)	0.0013 (9)
C8A	0.0194 (10)	0.0130 (10)	0.0247 (12)	0.0009 (8)	0.0031 (9)	0.0006 (9)
N21	0.0201 (9)	0.0176 (9)	0.0243 (10)	−0.0006 (8)	0.0028 (8)	0.0008 (8)
C22	0.0208 (11)	0.0123 (9)	0.0258 (12)	0.0025 (8)	0.0021 (9)	0.0017 (9)
N23	0.0219 (10)	0.0143 (9)	0.0270 (11)	−0.0004 (7)	0.0050 (8)	0.0001 (7)
C23A	0.0224 (11)	0.0127 (9)	0.0258 (12)	0.0007 (8)	0.0046 (9)	0.0021 (9)
C24	0.0227 (11)	0.0170 (10)	0.0319 (13)	−0.0006 (9)	0.0035 (10)	0.0008 (9)
C25	0.0243 (12)	0.0198 (11)	0.0353 (14)	0.0009 (9)	0.0124 (10)	0.0044 (10)
C26	0.0337 (13)	0.0165 (11)	0.0269 (13)	0.0015 (9)	0.0109 (10)	0.0029 (9)
C27	0.0286 (12)	0.0148 (10)	0.0246 (12)	−0.0003 (9)	0.0014 (10)	0.0015 (9)
C27A	0.0223 (11)	0.0107 (9)	0.0260 (12)	0.0018 (8)	0.0059 (9)	0.0033 (9)
C31	0.0196 (11)	0.0163 (10)	0.0221 (12)	0.0012 (8)	0.0047 (9)	0.0015 (9)
O31	0.0242 (8)	0.0177 (8)	0.0302 (9)	−0.0025 (6)	0.0002 (7)	−0.0020 (7)
O32	0.0150 (7)	0.0164 (7)	0.0302 (9)	0.0013 (6)	0.0019 (6)	0.0008 (6)
C32	0.0171 (11)	0.0216 (11)	0.0390 (14)	0.0019 (9)	−0.0003 (10)	0.0017 (10)
C33	0.0234 (12)	0.0247 (12)	0.0507 (17)	0.0047 (10)	0.0021 (11)	0.0009 (11)
C41	0.0198 (11)	0.0169 (10)	0.0244 (12)	−0.0003 (9)	0.0030 (9)	−0.0010 (9)
C42	0.0199 (11)	0.0165 (10)	0.0235 (12)	−0.0018 (8)	0.0044 (9)	−0.0013 (9)
C421	0.0149 (10)	0.0178 (10)	0.0245 (12)	−0.0015 (8)	0.0013 (9)	0.0016 (9)
C422	0.0219 (11)	0.0168 (10)	0.0283 (12)	0.0007 (9)	0.0022 (9)	0.0025 (9)
C423	0.0208 (11)	0.0248 (12)	0.0321 (13)	−0.0007 (9)	−0.0018 (10)	0.0058 (10)
C424	0.0232 (12)	0.0361 (13)	0.0249 (13)	−0.0045 (10)	−0.0023 (10)	0.0030 (11)
C425	0.0259 (12)	0.0318 (13)	0.0270 (13)	−0.0024 (10)	0.0033 (10)	−0.0053 (10)
C426	0.0208 (11)	0.0224 (11)	0.0280 (12)	0.0000 (9)	0.0010 (9)	−0.0012 (10)
O422	0.0402 (10)	0.0194 (8)	0.0329 (10)	0.0113 (7)	−0.0021 (8)	0.0001 (7)
C427	0.0349 (14)	0.0166 (11)	0.0456 (16)	0.0069 (10)	−0.0007 (12)	0.0034 (11)

Geometric parameters (Å, º)

N1—C2	1.322 (3)	C27—C27A	1.393 (3)
N1—C8A	1.366 (3)	C27—H27	0.9500
C2—C3	1.422 (3)	C31—O31	1.205 (3)
C2—C22	1.473 (3)	C31—O32	1.342 (2)
C3—C4	1.380 (3)	O32—C32	1.449 (2)
C3—C31	1.503 (3)	C32—C33	1.499 (3)
C4—C4A	1.428 (3)	C32—H32A	0.9900
C4—C41	1.470 (3)	C32—H32B	0.9900
C4A—C5	1.417 (3)	C33—H33A	0.9800
C4A—C8A	1.420 (3)	C33—H33B	0.9800
C5—C6	1.366 (3)	C33—H33C	0.9800
C5—H5	0.9500	C41—C42	1.331 (3)
C6—C7	1.405 (3)	C41—H41	0.9500
C6—H6	0.9500	C42—C421	1.465 (3)
C7—C8	1.368 (3)	C42—H42	0.9500
C7—H7	0.9500	C421—C426	1.395 (3)
C8—C8A	1.416 (3)	C421—C422	1.410 (3)
C8—H8	0.9500	C422—O422	1.371 (3)

N21—C22	1.361 (3)	C422—C423	1.386 (3)
N21—C27A	1.374 (3)	C423—C424	1.385 (3)
N21—H21	0.86 (3)	C423—H423	0.9500
C22—N23	1.318 (3)	C424—C425	1.386 (3)
N23—C23A	1.389 (3)	C424—H424	0.9500
C23A—C24	1.399 (3)	C425—C426	1.386 (3)
C23A—C27A	1.402 (3)	C425—H425	0.9500
C24—C25	1.379 (3)	C426—H426	0.9500
C24—H24	0.9500	O422—C427	1.428 (3)
C25—C26	1.401 (3)	C427—H27A	0.9800
C25—H25	0.9500	C427—H27B	0.9800
C26—C27	1.382 (3)	C427—H27C	0.9800
C26—H26	0.9500		

C2—N1—C8A	117.64 (19)	N21—C27A—C27	132.4 (2)
N1—C2—C3	124.08 (19)	N21—C27A—C23A	104.95 (19)
N1—C2—C22	114.92 (19)	C27—C27A—C23A	122.7 (2)
C3—C2—C22	121.00 (19)	O31—C31—O32	124.8 (2)
C4—C3—C2	119.16 (19)	O31—C31—C3	124.68 (19)
C4—C3—C31	120.2 (2)	O32—C31—C3	110.43 (17)
C2—C3—C31	120.58 (19)	C31—O32—C32	115.13 (16)
C3—C4—C4A	118.0 (2)	O32—C32—C33	106.74 (18)
C3—C4—C41	122.3 (2)	O32—C32—H32A	110.4
C4A—C4—C41	119.67 (19)	C33—C32—H32A	110.4
C5—C4A—C8A	118.7 (2)	O32—C32—H32B	110.4
C5—C4A—C4	122.9 (2)	C33—C32—H32B	110.4
C8A—C4A—C4	118.44 (19)	H32A—C32—H32B	108.6
C6—C5—C4A	120.7 (2)	C32—C33—H33A	109.5
C6—C5—H5	119.7	C32—C33—H33B	109.5
C4A—C5—H5	119.7	H33A—C33—H33B	109.5
C5—C6—C7	120.7 (2)	C32—C33—H33C	109.5
C5—C6—H6	119.7	H33A—C33—H33C	109.5
C7—C6—H6	119.7	H33B—C33—H33C	109.5
C8—C7—C6	120.2 (2)	C42—C41—C4	122.9 (2)
C8—C7—H7	119.9	C42—C41—H41	118.5
C6—C7—H7	119.9	C4—C41—H41	118.5
C7—C8—C8A	120.6 (2)	C41—C42—C421	127.0 (2)
C7—C8—H8	119.7	C41—C42—H42	116.5
C8A—C8—H8	119.7	C421—C42—H42	116.5
N1—C8A—C8	118.2 (2)	C426—C421—C422	117.5 (2)
N1—C8A—C4A	122.61 (19)	C426—C421—C42	123.1 (2)
C8—C8A—C4A	119.21 (19)	C422—C421—C42	119.41 (19)
C22—N21—C27A	107.01 (19)	O422—C422—C423	124.1 (2)
C22—N21—H21	126.7 (17)	O422—C422—C421	114.6 (2)
C27A—N21—H21	125.8 (17)	C423—C422—C421	121.3 (2)
N23—C22—N21	113.53 (19)	C424—C423—C422	119.6 (2)
N23—C22—C2	125.3 (2)	C424—C423—H423	120.2
N21—C22—C2	121.20 (19)	C422—C423—H423	120.2

C22—N23—C23A	104.06 (19)	C423—C424—C425	120.4 (2)
N23—C23A—C24	129.7 (2)	C423—C424—H424	119.8
N23—C23A—C27A	110.44 (19)	C425—C424—H424	119.8
C24—C23A—C27A	119.9 (2)	C426—C425—C424	119.7 (2)
C25—C24—C23A	117.8 (2)	C426—C425—H425	120.2
C25—C24—H24	121.1	C424—C425—H425	120.2
C23A—C24—H24	121.1	C425—C426—C421	121.5 (2)
C24—C25—C26	121.4 (2)	C425—C426—H426	119.2
C24—C25—H25	119.3	C421—C426—H426	119.2
C26—C25—H25	119.3	C422—O422—C427	118.62 (19)
C27—C26—C25	122.0 (2)	O422—C427—H27A	109.5
C27—C26—H26	119.0	O422—C427—H27B	109.5
C25—C26—H26	119.0	H27A—C427—H27B	109.5
C26—C27—C27A	116.3 (2)	O422—C427—H27C	109.5
C26—C27—H27	121.9	H27A—C427—H27C	109.5
C27A—C27—H27	121.9	H27B—C427—H27C	109.5

C8A—N1—C2—C3	0.9 (3)	C27A—C23A—C24—C25	0.5 (3)
C8A—N1—C2—C22	−178.71 (17)	C23A—C24—C25—C26	−0.5 (3)
N1—C2—C3—C4	−1.9 (3)	C24—C25—C26—C27	0.1 (3)
C22—C2—C3—C4	177.70 (18)	C25—C26—C27—C27A	0.3 (3)
N1—C2—C3—C31	174.88 (19)	C22—N21—C27A—C27	−178.5 (2)
C22—C2—C3—C31	−5.5 (3)	C22—N21—C27A—C23A	0.0 (2)
C2—C3—C4—C4A	1.3 (3)	C26—C27—C27A—N21	178.1 (2)
C31—C3—C4—C4A	−175.49 (18)	C26—C27—C27A—C23A	−0.3 (3)
C2—C3—C4—C41	−178.34 (19)	N23—C23A—C27A—N21	0.4 (2)
C31—C3—C4—C41	4.9 (3)	C24—C23A—C27A—N21	−178.86 (18)
C3—C4—C4A—C5	−178.35 (19)	N23—C23A—C27A—C27	179.15 (19)
C41—C4—C4A—C5	1.3 (3)	C24—C23A—C27A—C27	−0.1 (3)
C3—C4—C4A—C8A	0.1 (3)	C4—C3—C31—O31	78.6 (3)
C41—C4—C4A—C8A	179.70 (19)	C2—C3—C31—O31	−98.1 (3)
C8A—C4A—C5—C6	0.9 (3)	C4—C3—C31—O32	−98.1 (2)
C4—C4A—C5—C6	179.3 (2)	C2—C3—C31—O32	85.2 (2)
C4A—C5—C6—C7	0.1 (3)	O31—C31—O32—C32	10.7 (3)
C5—C6—C7—C8	−0.4 (3)	C3—C31—O32—C32	−172.58 (18)
C6—C7—C8—C8A	−0.2 (3)	C31—O32—C32—C33	−177.8 (2)
C2—N1—C8A—C8	179.57 (19)	C3—C4—C41—C42	49.8 (3)
C2—N1—C8A—C4A	0.6 (3)	C4A—C4—C41—C42	−129.9 (2)
C7—C8—C8A—N1	−177.8 (2)	C4—C41—C42—C421	179.4 (2)
C7—C8—C8A—C4A	1.2 (3)	C41—C42—C421—C426	13.7 (3)
C5—C4A—C8A—N1	177.42 (18)	C41—C42—C421—C422	−167.1 (2)
C4—C4A—C8A—N1	−1.1 (3)	C426—C421—C422—O422	179.99 (19)
C5—C4A—C8A—C8	−1.6 (3)	C42—C421—C422—O422	0.7 (3)
C4—C4A—C8A—C8	179.97 (19)	C426—C421—C422—C423	0.2 (3)
C27A—N21—C22—N23	−0.5 (2)	C42—C421—C422—C423	−179.0 (2)
C27A—N21—C22—C2	178.98 (18)	O422—C422—C423—C424	−179.3 (2)
N1—C2—C22—N23	−172.40 (19)	C421—C422—C423—C424	0.4 (3)
C3—C2—C22—N23	8.0 (3)	C422—C423—C424—C425	−1.0 (3)

N1—C2—C22—N21	8.2 (3)	C423—C424—C425—C426	0.8 (3)
C3—C2—C22—N21	−171.44 (19)	C424—C425—C426—C421	0.0 (3)
N21—C22—N23—C23A	0.7 (2)	C422—C421—C426—C425	−0.4 (3)
C2—C22—N23—C23A	−178.73 (19)	C42—C421—C426—C425	178.8 (2)
C22—N23—C23A—C24	178.5 (2)	C423—C422—O422—C427	1.8 (3)
C22—N23—C23A—C27A	−0.7 (2)	C421—C422—O422—C427	−177.9 (2)
N23—C23A—C24—C25	−178.6 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N21—H21···O31^{i}	0.86 (3)	2.45 (2)	3.081 (3)	130.4 (19)
C7—H7···*Cg*3^{ii}	0.95	2.95	3.704 (2)	138
C33—H33*B*···*Cg*3^{iii}	0.98	2.98	3.768 (3)	139

Symmetry codes: (i) −*x*+1/2, *y*+1/2, −*z*+3/2; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) −*x*, −*y*+1, −*z*+1.

Ethyl (*E*)-2-(1*H*-benzo[*d*]imidazol-2-yl)-4-(4-methylstyryl)quinoline-3-carboxylate (IIIc)

Crystal data

$C_{28}H_{23}N_3O_2$(+solvent)
M_r = 433.49
Monoclinic, $P2_1/n$
a = 20.2611 (7) Å
b = 9.8675 (4) Å
c = 36.8434 (14) Å
β = 104.332 (1)°
V = 7136.7 (5) Å^3
Z = 12
$F(000)$ = 2736
D_x = 1.210 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 16392 reflections
θ = 2.1–27.5°
μ = 0.08 mm^{-1}
T = 100 K
Block, yellow
0.18 × 0.12 × 0.06 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.917, T_{max} = 0.995
151790 measured reflections
17718 independent reflections
10436 reflections with $I > 2\sigma(I)$
R_{int} = 0.188
θ_{max} = 28.3°, θ_{min} = 2.1°
h = −24→27
k = −13→13
l = −49→49

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.096
$wR(F^2)$ = 0.216
S = 1.08
17718 reflections
921 parameters
7 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0622P)^2 + 9.4649P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.71 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Special details

Geometry. 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
N11	0.64456 (15)	0.3471 (3)	0.62137 (9)	0.0355 (7)	
C12	0.66488 (17)	0.4506 (4)	0.60418 (10)	0.0308 (8)	
C13	0.72133 (17)	0.4442 (4)	0.58762 (10)	0.0333 (8)	
C14	0.75387 (19)	0.3205 (4)	0.58717 (11)	0.0420 (9)	
C14A	0.7320 (2)	0.2079 (4)	0.60474 (11)	0.0396 (9)	
C15	0.7616 (2)	0.0754 (5)	0.60593 (13)	0.0511 (11)	
H15	0.7982	0.0604	0.5945	0.061*	
C16	0.7379 (2)	−0.0292 (4)	0.62321 (12)	0.0512 (11)	
H16	0.7573	−0.1169	0.6231	0.061*	
C17	0.6857 (2)	−0.0092 (4)	0.64109 (12)	0.0505 (11)	
H17	0.6704	−0.0827	0.6535	0.061*	
C18	0.6563 (2)	0.1157 (4)	0.64088 (12)	0.0465 (10)	
H18	0.6208	0.1281	0.6533	0.056*	
C18A	0.67797 (19)	0.2270 (4)	0.62234 (10)	0.0364 (8)	
N121	0.57389 (15)	0.5901 (3)	0.61973 (9)	0.0331 (7)	
H121	0.571 (2)	0.544 (4)	0.6359 (12)	0.040*	
C122	0.62381 (17)	0.5755 (4)	0.60133 (10)	0.0299 (7)	
N123	0.62852 (14)	0.6795 (3)	0.57985 (9)	0.0347 (7)	
C13A	0.57782 (17)	0.7680 (4)	0.58403 (11)	0.0337 (8)	
C124	0.5582 (2)	0.8933 (4)	0.56728 (12)	0.0442 (10)	
H124	0.5812	0.9316	0.5502	0.053*	
C125	0.5046 (2)	0.9600 (4)	0.57620 (13)	0.0478 (10)	
H125	0.4905	1.0453	0.5650	0.057*	
C126	0.4704 (2)	0.9040 (4)	0.60171 (13)	0.0466 (10)	
H126	0.4336	0.9526	0.6072	0.056*	
C127	0.48890 (19)	0.7810 (4)	0.61883 (12)	0.0411 (9)	
H127	0.4661	0.7439	0.6362	0.049*	
C17A	0.54308 (17)	0.7130 (4)	0.60929 (10)	0.0330 (8)	
C131	0.74500 (17)	0.5681 (4)	0.57115 (10)	0.0334 (8)	0.765 (7)
O131	0.7853 (5)	0.6465 (5)	0.5902 (2)	0.0331 (13)	0.765 (7)
O132	0.7216 (6)	0.5752 (8)	0.53417 (12)	0.0383 (13)	0.765 (7)
C132	0.7296 (4)	0.7060 (6)	0.51783 (16)	0.0426 (16)	0.765 (7)
H12A	0.7776	0.7195	0.5169	0.051*	0.765 (7)
H12B	0.7168	0.7796	0.5330	0.051*	0.765 (7)
C133	0.6839 (3)	0.7073 (8)	0.47906 (17)	0.069 (2)	0.765 (7)
H13A	0.6989	0.6378	0.4639	0.103*	0.765 (7)
H13B	0.6859	0.7965	0.4677	0.103*	0.765 (7)
H13C	0.6370	0.6884	0.4802	0.103*	0.765 (7)
C431	0.74500 (17)	0.5681 (4)	0.57115 (10)	0.0334 (8)	0.235 (7)

O431	0.7777 (17)	0.669 (2)	0.5846 (8)	0.0331 (13)	0.235 (7)
O432	0.713 (2)	0.568 (3)	0.5347 (4)	0.0383 (13)	0.235 (7)
C432	0.7402 (14)	0.667 (2)	0.5129 (6)	0.0426 (16)	0.235 (7)
H42A	0.7376	0.6310	0.4875	0.051*	0.235 (7)
H42B	0.7885	0.6860	0.5252	0.051*	0.235 (7)
C433	0.6982 (11)	0.793 (2)	0.5108 (6)	0.069 (2)	0.235 (7)
H43A	0.7045	0.8312	0.5359	0.103*	0.235 (7)
H43B	0.6500	0.7705	0.5005	0.103*	0.235 (7)
H43C	0.7127	0.8589	0.4945	0.103*	0.235 (7)
C141	0.8099 (2)	0.3044 (4)	0.56772 (12)	0.0466 (10)	
H141	0.8020	0.2467	0.5465	0.056*	
C142	0.86871 (19)	0.3644 (4)	0.57804 (11)	0.0371 (8)	
H142	0.8761	0.4195	0.5998	0.044*	
C143	0.92535 (18)	0.3559 (3)	0.55940 (10)	0.0324 (8)	
C144	0.98867 (19)	0.4063 (4)	0.57757 (11)	0.0368 (8)	
H144	0.9950	0.4439	0.6019	0.044*	
C145	1.0427 (2)	0.4029 (4)	0.56108 (11)	0.0372 (8)	
H145	1.0859	0.4360	0.5746	0.045*	
C146	1.03553 (19)	0.3523 (3)	0.52527 (10)	0.0332 (8)	
C147	0.9723 (2)	0.3021 (4)	0.50679 (10)	0.0363 (8)	
H147	0.9660	0.2661	0.4823	0.044*	
C148	0.9181 (2)	0.3036 (4)	0.52355 (11)	0.0376 (9)	
H148	0.8753	0.2684	0.5103	0.045*	
C149	1.0933 (2)	0.3530 (4)	0.50624 (11)	0.0435 (10)	
H19A	1.0797	0.4041	0.4828	0.065*	
H19B	1.1043	0.2596	0.5008	0.065*	
H19C	1.1333	0.3956	0.5227	0.065*	
N21	0.36983 (13)	0.6413 (3)	0.74361 (8)	0.0255 (6)	
C22	0.39299 (15)	0.5757 (3)	0.71802 (9)	0.0228 (7)	
C23	0.45502 (16)	0.5028 (3)	0.72552 (9)	0.0240 (7)	
C24	0.49360 (16)	0.4947 (3)	0.76188 (9)	0.0250 (7)	
C24A	0.47137 (16)	0.5669 (3)	0.79006 (9)	0.0259 (7)	
C25	0.50790 (18)	0.5692 (4)	0.82827 (10)	0.0329 (8)	
H25	0.5486	0.5178	0.8361	0.039*	
C26	0.48539 (19)	0.6443 (4)	0.85388 (10)	0.0360 (8)	
H26	0.5110	0.6462	0.8792	0.043*	
C27	0.4247 (2)	0.7186 (4)	0.84299 (11)	0.0398 (9)	
H27	0.4095	0.7703	0.8611	0.048*	
C28	0.38725 (18)	0.7176 (4)	0.80678 (10)	0.0323 (8)	
H28	0.3462	0.7684	0.7998	0.039*	
C28A	0.40938 (16)	0.6410 (3)	0.77963 (9)	0.0255 (7)	
N221	0.28038 (13)	0.5999 (3)	0.67225 (8)	0.0249 (6)	
H221	0.2562 (18)	0.598 (4)	0.6873 (10)	0.030*	
C222	0.34962 (15)	0.5864 (3)	0.67940 (9)	0.0237 (7)	
N223	0.37256 (13)	0.5872 (3)	0.64918 (8)	0.0284 (6)	
C23A	0.31525 (16)	0.6028 (4)	0.62004 (10)	0.0298 (8)	
C224	0.30957 (18)	0.6133 (4)	0.58170 (10)	0.0399 (9)	
H224	0.3487	0.6100	0.5718	0.048*	

C225	0.24521 (19)	0.6287 (4)	0.55859 (11)	0.0428 (10)
H225	0.2398	0.6349	0.5323	0.051*
C226	0.18766 (19)	0.6352 (4)	0.57305 (11)	0.0396 (9)
H226	0.1442	0.6466	0.5562	0.048*
C227	0.19185 (17)	0.6255 (4)	0.61115 (10)	0.0319 (8)
H227	0.1526	0.6298	0.6209	0.038*
C27A	0.25686 (16)	0.6093 (3)	0.63411 (9)	0.0268 (7)
C231	0.48094 (15)	0.4459 (3)	0.69372 (9)	0.0250 (7)
O231	0.52647 (11)	0.4968 (2)	0.68279 (6)	0.0286 (5)
O232	0.44922 (11)	0.3315 (2)	0.68030 (6)	0.0286 (5)
C232	0.4724 (2)	0.2648 (4)	0.65030 (11)	0.0409 (9)
H22A	0.4663	0.1656	0.6519	0.049*
H22B	0.5216	0.2828	0.6535	0.049*
C233	0.4339 (2)	0.3139 (5)	0.61268 (11)	0.0512 (11)
H23A	0.3850	0.2993	0.6098	0.077*
H23B	0.4487	0.2639	0.5931	0.077*
H23C	0.4426	0.4108	0.6104	0.077*
C241	0.55673 (17)	0.4152 (3)	0.77195 (9)	0.0281 (7)
H241	0.5950	0.4540	0.7891	0.034*
C242	0.56372 (17)	0.2923 (3)	0.75858 (10)	0.0299 (7)
H242	0.5242	0.2538	0.7425	0.036*
C243	0.62568 (16)	0.2102 (3)	0.76605 (10)	0.0279 (7)
C244	0.63350 (18)	0.1128 (3)	0.74041 (10)	0.0317 (8)
H244	0.5977	0.0989	0.7186	0.038*
C245	0.69160 (18)	0.0354 (4)	0.74559 (10)	0.0339 (8)
H245	0.6959	−0.0281	0.7269	0.041*
C246	0.74444 (17)	0.0492 (4)	0.77797 (11)	0.0347 (8)
C247	0.73682 (18)	0.1458 (4)	0.80455 (11)	0.0353 (8)
H247	0.7717	0.1569	0.8269	0.042*
C248	0.67847 (18)	0.2256 (4)	0.79839 (10)	0.0341 (8)
H248	0.6744	0.2917	0.8165	0.041*
C249	0.80557 (19)	−0.0432 (4)	0.78489 (12)	0.0421 (9)
H29A	0.7915	−0.1360	0.7888	0.063*
H29B	0.8396	−0.0127	0.8072	0.063*
H29C	0.8253	−0.0410	0.7632	0.063*
N31	−0.06783 (13)	0.4999 (3)	0.67001 (8)	0.0264 (6)
C32	−0.01172 (16)	0.5723 (3)	0.67408 (9)	0.0240 (7)
C33	0.04542 (15)	0.5615 (3)	0.70565 (9)	0.0237 (7)
C34	0.03971 (15)	0.4802 (3)	0.73528 (9)	0.0236 (7)
C34A	−0.02118 (16)	0.4027 (3)	0.73171 (10)	0.0257 (7)
C35	−0.03341 (17)	0.3183 (3)	0.76035 (10)	0.0307 (8)
H35	−0.0006	0.3132	0.7837	0.037*
C36	−0.09204 (17)	0.2437 (3)	0.75474 (11)	0.0345 (8)
H36	−0.0996	0.1878	0.7743	0.041*
C37	−0.14136 (17)	0.2492 (4)	0.72016 (11)	0.0341 (8)
H37	−0.1809	0.1941	0.7161	0.041*
C38	−0.13218 (17)	0.3331 (3)	0.69278 (10)	0.0312 (8)
H38	−0.1661	0.3384	0.6698	0.037*

C38A	−0.07252 (16)	0.4130 (3)	0.69793 (9)	0.0254 (7)
N321	−0.07317 (14)	0.7115 (3)	0.62130 (8)	0.0278 (6)
H321	−0.1103 (19)	0.676 (4)	0.6225 (10)	0.033*
C322	−0.01260 (15)	0.6683 (3)	0.64356 (9)	0.0248 (7)
N323	0.04071 (14)	0.7238 (3)	0.63484 (8)	0.0302 (6)
C33A	0.01310 (17)	0.8090 (3)	0.60491 (10)	0.0311 (8)
C324	0.04516 (19)	0.8953 (4)	0.58475 (11)	0.0377 (9)
H324	0.0934	0.9004	0.5900	0.045*
C325	0.0046 (2)	0.9733 (4)	0.55677 (11)	0.0398 (9)
H325	0.0254	1.0334	0.5428	0.048*
C326	−0.0664 (2)	0.9657 (4)	0.54870 (11)	0.0405 (9)
H326	−0.0927	1.0204	0.5292	0.049*
C327	−0.09926 (19)	0.8814 (3)	0.56811 (10)	0.0344 (8)
H327	−0.1476	0.8775	0.5627	0.041*
C37A	−0.05854 (17)	0.8019 (3)	0.59612 (10)	0.0290 (7)
C331	0.11146 (16)	0.6307 (3)	0.70544 (9)	0.0258 (7)
O331	0.15984 (11)	0.5698 (2)	0.69974 (7)	0.0268 (5)
O332	0.11019 (12)	0.7628 (2)	0.71233 (8)	0.0403 (7)
C332	0.1667 (2)	0.8451 (4)	0.70250 (16)	0.0643 (15)
H32A	0.1831	0.7998	0.6824	0.077*
H32B	0.1502	0.9368	0.6938	0.077*
C333	0.2202 (3)	0.8535 (5)	0.73593 (14)	0.0640 (13)
H33A	0.2022	0.8913	0.7561	0.096*
H33B	0.2565	0.9122	0.7316	0.096*
H33C	0.2385	0.7627	0.7430	0.096*
C341	0.09338 (16)	0.4734 (3)	0.77049 (10)	0.0277 (7)
H341	0.1071	0.3862	0.7805	0.033*
C342	0.12401 (16)	0.5807 (3)	0.78924 (11)	0.0321 (8)
H342	0.1126	0.6665	0.7777	0.039*
C343	0.17340 (18)	0.5806 (4)	0.82569 (11)	0.0377 (9)
C344	0.20722 (19)	0.7005 (4)	0.83935 (13)	0.0450 (10)
H344	0.1983	0.7806	0.8247	0.054*
C345	0.2535 (2)	0.7041 (5)	0.87395 (14)	0.0540 (12)
H345	0.2770	0.7861	0.8823	0.065*
C346	0.2661 (2)	0.5907 (5)	0.89666 (13)	0.0533 (12)
C347	0.2323 (2)	0.4711 (5)	0.88365 (13)	0.0592 (13)
H347	0.2405	0.3918	0.8987	0.071*
C348	0.1865 (2)	0.4667 (4)	0.84858 (12)	0.0482 (11)
H348	0.1637	0.3842	0.8401	0.058*
C349	0.3150 (2)	0.5952 (6)	0.93514 (14)	0.0764 (17)
H39A	0.3598	0.6258	0.9328	0.115*
H39B	0.3192	0.5045	0.9463	0.115*
H39C	0.2978	0.6583	0.9512	0.115*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N11	0.0347 (16)	0.0345 (16)	0.0373 (18)	0.0039 (13)	0.0092 (14)	−0.0038 (14)

C12	0.0246 (17)	0.0376 (19)	0.0294 (19)	−0.0013 (14)	0.0049 (14)	−0.0062 (15)
C13	0.0286 (18)	0.038 (2)	0.032 (2)	−0.0014 (15)	0.0060 (15)	−0.0088 (16)
C14	0.033 (2)	0.054 (2)	0.038 (2)	0.0078 (18)	0.0071 (17)	−0.0084 (19)
C14A	0.040 (2)	0.046 (2)	0.031 (2)	0.0078 (17)	0.0039 (17)	−0.0035 (17)
C15	0.047 (2)	0.056 (3)	0.051 (3)	0.018 (2)	0.014 (2)	−0.005 (2)
C16	0.068 (3)	0.041 (2)	0.042 (2)	0.012 (2)	0.008 (2)	0.0037 (19)
C17	0.069 (3)	0.042 (2)	0.042 (2)	0.007 (2)	0.016 (2)	0.0037 (19)
C18	0.061 (3)	0.039 (2)	0.040 (2)	0.008 (2)	0.015 (2)	−0.0003 (18)
C18A	0.038 (2)	0.041 (2)	0.029 (2)	0.0044 (16)	0.0055 (16)	−0.0051 (16)
N121	0.0319 (16)	0.0360 (17)	0.0352 (18)	−0.0018 (13)	0.0156 (14)	−0.0026 (14)
C122	0.0256 (17)	0.0347 (19)	0.0307 (19)	−0.0030 (14)	0.0093 (14)	−0.0059 (15)
N123	0.0284 (15)	0.0360 (17)	0.0431 (19)	0.0023 (13)	0.0152 (14)	0.0007 (14)
C13A	0.0257 (18)	0.0347 (19)	0.044 (2)	−0.0021 (15)	0.0140 (16)	−0.0029 (16)
C124	0.039 (2)	0.038 (2)	0.059 (3)	−0.0024 (17)	0.020 (2)	0.0023 (19)
C125	0.043 (2)	0.036 (2)	0.065 (3)	0.0023 (18)	0.015 (2)	0.001 (2)
C126	0.036 (2)	0.044 (2)	0.063 (3)	0.0041 (18)	0.020 (2)	−0.010 (2)
C127	0.034 (2)	0.041 (2)	0.053 (3)	−0.0030 (17)	0.0216 (18)	−0.0073 (19)
C17A	0.0289 (18)	0.0328 (19)	0.040 (2)	−0.0020 (15)	0.0131 (16)	−0.0054 (16)
C131	0.0209 (16)	0.047 (2)	0.034 (2)	0.0033 (16)	0.0093 (15)	−0.0116 (17)
O131	0.020 (3)	0.036 (2)	0.041 (3)	0.006 (2)	0.0034 (17)	−0.001 (2)
O132	0.025 (4)	0.0526 (19)	0.0392 (16)	−0.0086 (15)	0.0124 (13)	−0.0011 (13)
C132	0.041 (3)	0.043 (4)	0.045 (3)	−0.002 (3)	0.013 (2)	−0.001 (3)
C133	0.062 (4)	0.092 (5)	0.049 (4)	−0.036 (4)	0.007 (3)	0.006 (3)
C431	0.0209 (16)	0.047 (2)	0.034 (2)	0.0033 (16)	0.0093 (15)	−0.0116 (17)
O431	0.020 (3)	0.036 (2)	0.041 (3)	0.006 (2)	0.0034 (17)	−0.001 (2)
O432	0.025 (4)	0.0526 (19)	0.0392 (16)	−0.0086 (15)	0.0124 (13)	−0.0011 (13)
C432	0.041 (3)	0.043 (4)	0.045 (3)	−0.002 (3)	0.013 (2)	−0.001 (3)
C433	0.062 (4)	0.092 (5)	0.049 (4)	−0.036 (4)	0.007 (3)	0.006 (3)
C141	0.041 (2)	0.048 (2)	0.050 (3)	0.0078 (19)	0.0087 (19)	−0.007 (2)
C142	0.043 (2)	0.036 (2)	0.033 (2)	0.0046 (17)	0.0111 (17)	0.0024 (16)
C143	0.038 (2)	0.0268 (17)	0.033 (2)	0.0070 (15)	0.0096 (16)	0.0024 (15)
C144	0.046 (2)	0.0339 (19)	0.030 (2)	0.0018 (17)	0.0095 (17)	−0.0018 (16)
C145	0.040 (2)	0.0323 (19)	0.039 (2)	0.0001 (16)	0.0106 (17)	0.0001 (16)
C146	0.042 (2)	0.0258 (17)	0.033 (2)	0.0068 (15)	0.0117 (17)	0.0048 (15)
C147	0.050 (2)	0.0306 (19)	0.0268 (19)	0.0072 (17)	0.0059 (17)	0.0005 (15)
C148	0.041 (2)	0.0320 (19)	0.038 (2)	0.0060 (16)	0.0067 (17)	0.0028 (16)
C149	0.055 (3)	0.037 (2)	0.043 (2)	0.0140 (18)	0.021 (2)	0.0094 (18)
N21	0.0252 (14)	0.0245 (14)	0.0274 (15)	−0.0022 (11)	0.0076 (12)	0.0022 (12)
C22	0.0212 (15)	0.0199 (15)	0.0289 (18)	−0.0022 (12)	0.0097 (13)	−0.0006 (13)
C23	0.0248 (16)	0.0198 (15)	0.0273 (18)	−0.0031 (12)	0.0064 (13)	0.0000 (13)
C24	0.0252 (16)	0.0225 (16)	0.0281 (18)	−0.0025 (13)	0.0082 (14)	0.0022 (13)
C24A	0.0295 (17)	0.0222 (16)	0.0274 (18)	−0.0061 (13)	0.0096 (14)	0.0004 (13)
C25	0.0351 (19)	0.0317 (18)	0.031 (2)	−0.0033 (15)	0.0070 (16)	0.0019 (15)
C26	0.042 (2)	0.040 (2)	0.0241 (19)	−0.0074 (17)	0.0054 (16)	−0.0023 (16)
C27	0.049 (2)	0.040 (2)	0.034 (2)	−0.0040 (18)	0.0179 (18)	−0.0096 (17)
C28	0.0343 (19)	0.0339 (19)	0.0301 (19)	−0.0031 (15)	0.0110 (16)	−0.0017 (15)
C28A	0.0282 (17)	0.0231 (16)	0.0266 (18)	−0.0048 (13)	0.0093 (14)	0.0015 (13)
N221	0.0200 (13)	0.0308 (15)	0.0252 (15)	0.0001 (11)	0.0083 (11)	−0.0024 (12)

C222	0.0197 (15)	0.0211 (15)	0.0306 (18)	0.0008 (12)	0.0066 (13)	0.0001 (13)
N223	0.0234 (14)	0.0347 (16)	0.0275 (15)	0.0041 (12)	0.0073 (12)	0.0024 (12)
C23A	0.0213 (16)	0.0376 (19)	0.0296 (19)	0.0053 (14)	0.0048 (14)	−0.0015 (15)
C224	0.0283 (19)	0.063 (3)	0.031 (2)	0.0084 (18)	0.0105 (16)	0.0011 (18)
C225	0.036 (2)	0.062 (3)	0.030 (2)	0.0106 (19)	0.0053 (17)	0.0009 (19)
C226	0.0283 (19)	0.052 (2)	0.034 (2)	0.0048 (17)	0.0003 (16)	−0.0021 (18)
C227	0.0217 (16)	0.041 (2)	0.033 (2)	0.0036 (14)	0.0064 (14)	−0.0035 (16)
C27A	0.0238 (16)	0.0284 (17)	0.0278 (18)	0.0019 (13)	0.0053 (14)	−0.0032 (14)
C231	0.0202 (15)	0.0251 (16)	0.0281 (18)	0.0042 (13)	0.0031 (13)	0.0048 (14)
O231	0.0214 (11)	0.0360 (13)	0.0294 (13)	0.0006 (10)	0.0085 (10)	0.0001 (10)
O232	0.0268 (12)	0.0271 (12)	0.0320 (13)	−0.0001 (10)	0.0073 (10)	−0.0049 (10)
C232	0.039 (2)	0.041 (2)	0.042 (2)	0.0036 (17)	0.0095 (18)	−0.0151 (18)
C233	0.053 (3)	0.062 (3)	0.038 (2)	0.012 (2)	0.013 (2)	−0.012 (2)
C241	0.0278 (17)	0.0302 (18)	0.0250 (18)	0.0011 (14)	0.0041 (14)	0.0045 (14)
C242	0.0268 (17)	0.0298 (18)	0.0310 (19)	−0.0035 (14)	0.0029 (14)	0.0046 (15)
C243	0.0243 (17)	0.0230 (16)	0.035 (2)	−0.0033 (13)	0.0049 (15)	0.0037 (14)
C244	0.0323 (19)	0.0254 (17)	0.033 (2)	0.0017 (14)	−0.0004 (15)	0.0050 (15)
C245	0.036 (2)	0.0307 (18)	0.035 (2)	0.0017 (15)	0.0089 (16)	0.0021 (15)
C246	0.0272 (18)	0.0287 (18)	0.046 (2)	−0.0009 (14)	0.0057 (16)	0.0096 (16)
C247	0.0282 (18)	0.0303 (18)	0.043 (2)	−0.0034 (15)	−0.0005 (16)	0.0018 (16)
C248	0.0335 (19)	0.0285 (18)	0.036 (2)	−0.0009 (15)	0.0000 (16)	0.0021 (15)
C249	0.034 (2)	0.036 (2)	0.057 (3)	0.0052 (16)	0.0105 (19)	0.0093 (19)
N31	0.0245 (14)	0.0273 (14)	0.0302 (16)	−0.0004 (11)	0.0122 (12)	−0.0050 (12)
C32	0.0238 (16)	0.0239 (16)	0.0267 (17)	0.0018 (13)	0.0109 (13)	−0.0045 (13)
C33	0.0220 (15)	0.0189 (15)	0.0319 (18)	0.0029 (12)	0.0103 (14)	−0.0037 (13)
C34	0.0208 (15)	0.0173 (15)	0.0327 (18)	0.0060 (12)	0.0067 (13)	−0.0054 (13)
C34A	0.0240 (16)	0.0192 (15)	0.0367 (19)	0.0054 (12)	0.0128 (14)	−0.0022 (14)
C35	0.0246 (17)	0.0295 (18)	0.038 (2)	0.0072 (14)	0.0077 (15)	0.0073 (15)
C36	0.0319 (19)	0.0258 (17)	0.050 (2)	0.0071 (14)	0.0187 (17)	0.0103 (16)
C37	0.0224 (17)	0.0323 (19)	0.049 (2)	−0.0001 (14)	0.0122 (16)	0.0023 (17)
C38	0.0286 (17)	0.0293 (18)	0.036 (2)	−0.0010 (14)	0.0086 (15)	−0.0041 (15)
C38A	0.0219 (16)	0.0245 (16)	0.0327 (19)	0.0027 (13)	0.0126 (14)	−0.0017 (14)
N321	0.0230 (14)	0.0278 (15)	0.0326 (16)	0.0000 (11)	0.0069 (12)	−0.0008 (12)
C322	0.0210 (15)	0.0235 (16)	0.0316 (18)	0.0027 (13)	0.0094 (14)	−0.0014 (14)
N323	0.0264 (15)	0.0297 (15)	0.0370 (17)	0.0045 (12)	0.0129 (13)	0.0027 (13)
C33A	0.0323 (18)	0.0292 (18)	0.035 (2)	0.0047 (14)	0.0133 (16)	0.0031 (15)
C324	0.0330 (19)	0.035 (2)	0.049 (2)	0.0031 (16)	0.0177 (18)	0.0051 (17)
C325	0.049 (2)	0.0283 (19)	0.045 (2)	0.0011 (17)	0.0173 (19)	0.0083 (17)
C326	0.047 (2)	0.0301 (19)	0.041 (2)	0.0055 (17)	0.0042 (18)	0.0050 (17)
C327	0.0331 (19)	0.0303 (18)	0.039 (2)	0.0026 (15)	0.0065 (16)	0.0007 (16)
C37A	0.0297 (18)	0.0238 (16)	0.035 (2)	0.0006 (14)	0.0105 (15)	−0.0022 (15)
C331	0.0259 (17)	0.0207 (15)	0.0312 (19)	0.0032 (13)	0.0079 (14)	0.0010 (14)
O331	0.0215 (11)	0.0214 (11)	0.0393 (14)	0.0040 (9)	0.0109 (10)	−0.0009 (10)
O332	0.0267 (13)	0.0208 (12)	0.080 (2)	−0.0030 (10)	0.0251 (13)	−0.0107 (12)
C332	0.035 (2)	0.040 (2)	0.112 (4)	0.0104 (19)	0.007 (3)	−0.031 (3)
C333	0.072 (3)	0.053 (3)	0.072 (4)	0.012 (2)	0.026 (3)	−0.007 (3)
C341	0.0226 (16)	0.0238 (16)	0.037 (2)	0.0045 (13)	0.0079 (14)	−0.0009 (14)
C342	0.0250 (17)	0.0257 (17)	0.045 (2)	0.0044 (14)	0.0078 (15)	−0.0027 (16)

C343	0.0266 (18)	0.0330 (19)	0.049 (2)	0.0028 (15)	0.0013 (16)	−0.0106 (17)
C344	0.034 (2)	0.034 (2)	0.062 (3)	0.0004 (16)	0.005 (2)	−0.0107 (19)
C345	0.030 (2)	0.047 (3)	0.078 (3)	−0.0024 (18)	0.001 (2)	−0.029 (2)
C346	0.029 (2)	0.064 (3)	0.058 (3)	0.010 (2)	−0.0061 (19)	−0.031 (2)
C347	0.060 (3)	0.050 (3)	0.051 (3)	0.013 (2)	−0.018 (2)	−0.011 (2)
C348	0.049 (2)	0.034 (2)	0.048 (3)	0.0007 (18)	−0.013 (2)	−0.0105 (19)
C349	0.049 (3)	0.098 (4)	0.067 (3)	0.015 (3)	−0.014 (2)	−0.047 (3)

Geometric parameters (Å, º)

N11—C12	1.320 (5)	C225—C226	1.398 (5)
N11—C18A	1.361 (5)	C225—H225	0.9500
C12—C13	1.425 (5)	C226—C227	1.388 (5)
C12—C122	1.476 (5)	C226—H226	0.9500
C13—C14	1.389 (5)	C227—C27A	1.387 (5)
C13—C131	1.496 (5)	C227—H227	0.9500
C14—C14A	1.412 (6)	C231—O231	1.204 (4)
C14—C141	1.493 (5)	C231—O232	1.332 (4)
C14A—C18A	1.416 (5)	O232—C232	1.461 (4)
C14A—C15	1.435 (6)	C232—C233	1.494 (6)
C15—C16	1.360 (6)	C232—H22A	0.9900
C15—H15	0.9500	C232—H22B	0.9900
C16—C17	1.392 (6)	C233—H23A	0.9800
C16—H16	0.9500	C233—H23B	0.9800
C17—C18	1.369 (6)	C233—H23C	0.9800
C17—H17	0.9500	C241—C242	1.330 (5)
C18—C18A	1.419 (6)	C241—H241	0.9500
C18—H18	0.9500	C242—C243	1.462 (5)
N121—C122	1.358 (4)	C242—H242	0.9500
N121—C17A	1.375 (5)	C243—C244	1.384 (5)
N121—H121	0.76 (4)	C243—C248	1.399 (5)
C122—N123	1.314 (4)	C244—C245	1.376 (5)
N123—C13A	1.385 (4)	C244—H244	0.9500
C13A—C124	1.395 (5)	C245—C246	1.399 (5)
C13A—C17A	1.407 (5)	C245—H245	0.9500
C124—C125	1.377 (5)	C246—C247	1.402 (5)
C124—H124	0.9500	C246—C249	1.508 (5)
C125—C126	1.412 (6)	C247—C248	1.391 (5)
C125—H125	0.9500	C247—H247	0.9500
C126—C127	1.377 (6)	C248—H248	0.9500
C126—H126	0.9500	C249—H29A	0.9800
C127—C17A	1.403 (5)	C249—H29B	0.9800
C127—H127	0.9500	C249—H29C	0.9800
C131—O131	1.214 (5)	N31—C32	1.319 (4)
C131—O132	1.329 (5)	N31—C38A	1.361 (4)
O132—C132	1.450 (7)	C32—C33	1.428 (5)
C132—C133	1.498 (8)	C32—C322	1.467 (5)
C132—H12A	0.9900	C33—C34	1.382 (5)

C132—H12B	0.9900	C33—C331	1.504 (4)
C133—H13A	0.9800	C34—C34A	1.430 (4)
C133—H13B	0.9800	C34—C341	1.474 (5)
C133—H13C	0.9800	C34A—C35	1.413 (5)
O432—C432	1.456 (11)	C34A—C38A	1.414 (5)
C432—C433	1.499 (12)	C35—C36	1.369 (5)
C432—H42A	0.9900	C35—H35	0.9500
C432—H42B	0.9900	C36—C37	1.412 (5)
C433—H43A	0.9800	C36—H36	0.9500
C433—H43B	0.9800	C37—C38	1.353 (5)
C433—H43C	0.9800	C37—H37	0.9500
C141—C142	1.301 (5)	C38—C38A	1.416 (4)
C141—H141	0.9500	C38—H38	0.9500
C142—C143	1.478 (5)	N321—C322	1.365 (4)
C142—H142	0.9500	N321—C37A	1.371 (4)
C143—C144	1.384 (5)	N321—H321	0.84 (4)
C143—C148	1.391 (5)	C322—N323	1.320 (4)
C144—C145	1.379 (5)	N323—C33A	1.389 (4)
C144—H144	0.9500	C33A—C324	1.392 (5)
C145—C146	1.384 (5)	C33A—C37A	1.409 (5)
C145—H145	0.9500	C324—C325	1.383 (5)
C146—C147	1.385 (5)	C324—H324	0.9500
C146—C149	1.505 (5)	C325—C326	1.396 (5)
C147—C148	1.386 (5)	C325—H325	0.9500
C147—H147	0.9500	C326—C327	1.372 (5)
C148—H148	0.9500	C326—H326	0.9500
C149—H19A	0.9800	C327—C37A	1.393 (5)
C149—H19B	0.9800	C327—H327	0.9500
C149—H19C	0.9800	C331—O331	1.212 (4)
N21—C22	1.322 (4)	C331—O332	1.330 (4)
N21—C28A	1.370 (4)	O332—C332	1.519 (5)
C22—C23	1.414 (4)	C332—C333	1.427 (7)
C22—C222	1.479 (5)	C332—H32A	0.9900
C23—C24	1.376 (5)	C332—H32B	0.9900
C23—C231	1.507 (4)	C333—H33A	0.9800
C24—C24A	1.421 (4)	C333—H33B	0.9800
C24—C241	1.468 (4)	C333—H33C	0.9800
C24A—C25	1.419 (5)	C341—C342	1.331 (5)
C24A—C28A	1.422 (5)	C341—H341	0.9500
C25—C26	1.363 (5)	C342—C343	1.463 (5)
C25—H25	0.9500	C342—H342	0.9500
C26—C27	1.403 (5)	C343—C348	1.391 (6)
C26—H26	0.9500	C343—C344	1.396 (5)
C27—C28	1.362 (5)	C344—C345	1.383 (6)
C27—H27	0.9500	C344—H344	0.9500
C28—C28A	1.412 (5)	C345—C346	1.383 (7)
C28—H28	0.9500	C345—H345	0.9500
N221—C222	1.368 (4)	C346—C347	1.389 (6)

N221—C27A	1.371 (4)	C346—C349	1.516 (6)
N221—H221	0.82 (4)	C347—C348	1.393 (6)
C222—N223	1.309 (4)	C347—H347	0.9500
N223—C23A	1.381 (4)	C348—H348	0.9500
C23A—C224	1.392 (5)	C349—H39A	0.9800
C23A—C27A	1.406 (4)	C349—H39B	0.9800
C224—C225	1.378 (5)	C349—H39C	0.9800
C224—H224	0.9500		

C12—N11—C18A	118.3 (3)	C227—C226—C225	122.3 (3)
N11—C12—C13	123.4 (3)	C227—C226—H226	118.9
N11—C12—C122	116.3 (3)	C225—C226—H226	118.9
C13—C12—C122	120.3 (3)	C27A—C227—C226	115.8 (3)
C14—C13—C12	118.5 (3)	C27A—C227—H227	122.1
C14—C13—C131	120.8 (3)	C226—C227—H227	122.1
C12—C13—C131	120.6 (3)	N221—C27A—C227	132.0 (3)
C13—C14—C14A	118.9 (3)	N221—C27A—C23A	105.3 (3)
C13—C14—C141	121.2 (4)	C227—C27A—C23A	122.6 (3)
C14A—C14—C141	119.9 (4)	O231—C231—O232	124.7 (3)
C14—C14A—C18A	118.1 (3)	O231—C231—C23	123.2 (3)
C14—C14A—C15	123.8 (4)	O232—C231—C23	112.1 (3)
C18A—C14A—C15	118.2 (4)	C231—O232—C232	116.5 (3)
C16—C15—C14A	120.8 (4)	O232—C232—C233	111.3 (3)
C16—C15—H15	119.6	O232—C232—H22A	109.4
C14A—C15—H15	119.6	C233—C232—H22A	109.4
C15—C16—C17	120.7 (4)	O232—C232—H22B	109.4
C15—C16—H16	119.6	C233—C232—H22B	109.4
C17—C16—H16	119.6	H22A—C232—H22B	108.0
C18—C17—C16	120.4 (4)	C232—C233—H23A	109.5
C18—C17—H17	119.8	C232—C233—H23B	109.5
C16—C17—H17	119.8	H23A—C233—H23B	109.5
C17—C18—C18A	120.8 (4)	C232—C233—H23C	109.5
C17—C18—H18	119.6	H23A—C233—H23C	109.5
C18A—C18—H18	119.6	H23B—C233—H23C	109.5
N11—C18A—C14A	122.7 (4)	C242—C241—C24	124.0 (3)
N11—C18A—C18	118.3 (3)	C242—C241—H241	118.0
C14A—C18A—C18	119.0 (4)	C24—C241—H241	118.0
C122—N121—C17A	107.3 (3)	C241—C242—C243	127.0 (3)
C122—N121—H121	123 (3)	C241—C242—H242	116.5
C17A—N121—H121	128 (3)	C243—C242—H242	116.5
N123—C122—N121	113.1 (3)	C244—C243—C248	117.7 (3)
N123—C122—C12	124.8 (3)	C244—C243—C242	119.7 (3)
N121—C122—C12	122.0 (3)	C248—C243—C242	122.7 (3)
C122—N123—C13A	105.0 (3)	C245—C244—C243	122.0 (3)
N123—C13A—C124	130.3 (3)	C245—C244—H244	119.0
N123—C13A—C17A	109.7 (3)	C243—C244—H244	119.0
C124—C13A—C17A	120.0 (3)	C244—C245—C246	120.8 (4)
C125—C124—C13A	118.3 (4)	C244—C245—H245	119.6

C125—C124—H124	120.9	C246—C245—H245	119.6
C13A—C124—H124	120.9	C245—C246—C247	117.9 (3)
C124—C125—C126	121.2 (4)	C245—C246—C249	120.6 (4)
C124—C125—H125	119.4	C247—C246—C249	121.4 (3)
C126—C125—H125	119.4	C248—C247—C246	120.5 (3)
C127—C126—C125	121.8 (4)	C248—C247—H247	119.8
C127—C126—H126	119.1	C246—C247—H247	119.8
C125—C126—H126	119.1	C247—C248—C243	121.1 (3)
C126—C127—C17A	116.6 (4)	C247—C248—H248	119.4
C126—C127—H127	121.7	C243—C248—H248	119.4
C17A—C127—H127	121.7	C246—C249—H29A	109.5
N121—C17A—C127	132.9 (3)	C246—C249—H29B	109.5
N121—C17A—C13A	105.0 (3)	H29A—C249—H29B	109.5
C127—C17A—C13A	122.1 (3)	C246—C249—H29C	109.5
O131—C131—O132	125.3 (5)	H29A—C249—H29C	109.5
O131—C131—C13	121.6 (5)	H29B—C249—H29C	109.5
O132—C131—C13	113.0 (4)	C32—N31—C38A	118.3 (3)
C131—O132—C132	115.1 (5)	N31—C32—C33	123.7 (3)
O132—C132—C133	107.3 (5)	N31—C32—C322	114.6 (3)
O132—C132—H12A	110.3	C33—C32—C322	121.7 (3)
C133—C132—H12A	110.3	C34—C33—C32	118.5 (3)
O132—C132—H12B	110.3	C34—C33—C331	121.1 (3)
C133—C132—H12B	110.3	C32—C33—C331	120.3 (3)
H12A—C132—H12B	108.5	C33—C34—C34A	118.5 (3)
C132—C133—H13A	109.5	C33—C34—C341	122.3 (3)
C132—C133—H13B	109.5	C34A—C34—C341	119.2 (3)
H13A—C133—H13B	109.5	C35—C34A—C38A	118.2 (3)
C132—C133—H13C	109.5	C35—C34A—C34	123.6 (3)
H13A—C133—H13C	109.5	C38A—C34A—C34	118.2 (3)
H13B—C133—H13C	109.5	C36—C35—C34A	120.7 (3)
O432—C432—C433	107.0 (14)	C36—C35—H35	119.7
O432—C432—H42A	110.3	C34A—C35—H35	119.7
C433—C432—H42A	110.3	C35—C36—C37	120.6 (3)
O432—C432—H42B	110.3	C35—C36—H36	119.7
C433—C432—H42B	110.3	C37—C36—H36	119.7
H42A—C432—H42B	108.6	C38—C37—C36	119.9 (3)
C432—C433—H43A	109.5	C38—C37—H37	120.0
C432—C433—H43B	109.5	C36—C37—H37	120.0
H43A—C433—H43B	109.5	C37—C38—C38A	120.7 (3)
C432—C433—H43C	109.5	C37—C38—H38	119.6
H43A—C433—H43C	109.5	C38A—C38—H38	119.6
H43B—C433—H43C	109.5	N31—C38A—C34A	122.5 (3)
C142—C141—C14	124.2 (4)	N31—C38A—C38	117.8 (3)
C142—C141—H141	117.9	C34A—C38A—C38	119.7 (3)
C14—C141—H141	117.9	C322—N321—C37A	107.3 (3)
C141—C142—C143	126.8 (4)	C322—N321—H321	121 (3)
C141—C142—H142	116.6	C37A—N321—H321	131 (3)
C143—C142—H142	116.6	N323—C322—N321	113.1 (3)

C144—C143—C148	117.3 (3)	N323—C322—C32	126.8 (3)
C144—C143—C142	119.2 (3)	N321—C322—C32	120.1 (3)
C148—C143—C142	123.4 (3)	C322—N323—C33A	104.6 (3)
C145—C144—C143	121.4 (4)	N323—C33A—C324	130.1 (3)
C145—C144—H144	119.3	N323—C33A—C37A	110.0 (3)
C143—C144—H144	119.3	C324—C33A—C37A	119.9 (3)
C144—C145—C146	121.4 (4)	C325—C324—C33A	117.9 (3)
C144—C145—H145	119.3	C325—C324—H324	121.0
C146—C145—H145	119.3	C33A—C324—H324	121.0
C145—C146—C147	117.7 (3)	C324—C325—C326	121.3 (3)
C145—C146—C149	122.1 (4)	C324—C325—H325	119.3
C147—C146—C149	120.2 (3)	C326—C325—H325	119.3
C146—C147—C148	121.0 (3)	C327—C326—C325	121.9 (4)
C146—C147—H147	119.5	C327—C326—H326	119.0
C148—C147—H147	119.5	C325—C326—H326	119.0
C147—C148—C143	121.2 (4)	C326—C327—C37A	116.9 (3)
C147—C148—H148	119.4	C326—C327—H327	121.5
C143—C148—H148	119.4	C37A—C327—H327	121.5
C146—C149—H19A	109.5	N321—C37A—C327	132.9 (3)
C146—C149—H19B	109.5	N321—C37A—C33A	105.1 (3)
H19A—C149—H19B	109.5	C327—C37A—C33A	122.0 (3)
C146—C149—H19C	109.5	O331—C331—O332	125.0 (3)
H19A—C149—H19C	109.5	O331—C331—C33	122.3 (3)
H19B—C149—H19C	109.5	O332—C331—C33	112.6 (3)
C22—N21—C28A	117.6 (3)	C331—O332—C332	115.3 (3)
N21—C22—C23	124.4 (3)	C333—C332—O332	106.3 (5)
N21—C22—C222	114.8 (3)	C333—C332—H32A	110.5
C23—C22—C222	120.8 (3)	O332—C332—H32A	110.5
C24—C23—C22	118.9 (3)	C333—C332—H32B	110.5
C24—C23—C231	120.8 (3)	O332—C332—H32B	110.5
C22—C23—C231	120.1 (3)	H32A—C332—H32B	108.7
C23—C24—C24A	118.4 (3)	C332—C333—H33A	109.5
C23—C24—C241	121.8 (3)	C332—C333—H33B	109.5
C24A—C24—C241	119.8 (3)	H33A—C333—H33B	109.5
C25—C24A—C24	123.4 (3)	C332—C333—H33C	109.5
C25—C24A—C28A	117.9 (3)	H33A—C333—H33C	109.5
C24—C24A—C28A	118.6 (3)	H33B—C333—H33C	109.5
C26—C25—C24A	120.9 (3)	C342—C341—C34	124.6 (3)
C26—C25—H25	119.5	C342—C341—H341	117.7
C24A—C25—H25	119.5	C34—C341—H341	117.7
C25—C26—C27	120.4 (4)	C341—C342—C343	127.0 (3)
C25—C26—H26	119.8	C341—C342—H342	116.5
C27—C26—H26	119.8	C343—C342—H342	116.5
C28—C27—C26	120.8 (3)	C348—C343—C344	117.6 (4)
C28—C27—H27	119.6	C348—C343—C342	122.8 (3)
C26—C27—H27	119.6	C344—C343—C342	119.6 (4)
C27—C28—C28A	120.0 (3)	C345—C344—C343	121.0 (4)
C27—C28—H28	120.0	C345—C344—H344	119.5

C28A—C28—H28	120.0	C343—C344—H344	119.5
N21—C28A—C28	118.2 (3)	C346—C345—C344	121.2 (4)
N21—C28A—C24A	121.9 (3)	C346—C345—H345	119.4
C28—C28A—C24A	119.9 (3)	C344—C345—H345	119.4
C222—N221—C27A	106.5 (3)	C345—C346—C347	118.5 (4)
C222—N221—H221	128 (3)	C345—C346—C349	121.5 (4)
C27A—N221—H221	125 (3)	C347—C346—C349	120.0 (5)
N223—C222—N221	113.5 (3)	C346—C347—C348	120.3 (4)
N223—C222—C22	124.6 (3)	C346—C347—H347	119.8
N221—C222—C22	121.9 (3)	C348—C347—H347	119.8
C222—N223—C23A	104.8 (3)	C343—C348—C347	121.3 (4)
N223—C23A—C224	129.7 (3)	C343—C348—H348	119.3
N223—C23A—C27A	109.9 (3)	C347—C348—H348	119.3
C224—C23A—C27A	120.3 (3)	C346—C349—H39A	109.5
C225—C224—C23A	117.6 (3)	C346—C349—H39B	109.5
C225—C224—H224	121.2	H39A—C349—H39B	109.5
C23A—C224—H224	121.2	C346—C349—H39C	109.5
C224—C225—C226	121.3 (4)	H39A—C349—H39C	109.5
C224—C225—H225	119.3	H39B—C349—H39C	109.5
C226—C225—H225	119.3		

C18A—N11—C12—C13	−2.0 (5)	C222—N223—C23A—C27A	−0.8 (4)
C18A—N11—C12—C122	175.5 (3)	N223—C23A—C224—C225	−179.9 (4)
N11—C12—C13—C14	4.6 (5)	C27A—C23A—C224—C225	−0.7 (6)
C122—C12—C13—C14	−172.8 (3)	C23A—C224—C225—C226	0.8 (6)
N11—C12—C13—C131	−175.3 (3)	C224—C225—C226—C227	−0.5 (7)
C122—C12—C13—C131	7.3 (5)	C225—C226—C227—C27A	0.2 (6)
C12—C13—C14—C14A	−3.1 (5)	C222—N221—C27A—C227	−179.3 (4)
C131—C13—C14—C14A	176.8 (3)	C222—N221—C27A—C23A	−0.9 (4)
C12—C13—C14—C141	175.3 (4)	C226—C227—C27A—N221	178.1 (4)
C131—C13—C14—C141	−4.8 (6)	C226—C227—C27A—C23A	−0.1 (5)
C13—C14—C14A—C18A	−0.5 (6)	N223—C23A—C27A—N221	1.1 (4)
C141—C14—C14A—C18A	−179.0 (4)	C224—C23A—C27A—N221	−178.3 (3)
C13—C14—C14A—C15	179.8 (4)	N223—C23A—C27A—C227	179.7 (3)
C141—C14—C14A—C15	1.3 (6)	C224—C23A—C27A—C227	0.4 (5)
C14—C14A—C15—C16	−179.7 (4)	C24—C23—C231—O231	−71.5 (4)
C18A—C14A—C15—C16	0.6 (6)	C22—C23—C231—O231	103.2 (4)
C14A—C15—C16—C17	−1.9 (7)	C24—C23—C231—O232	106.4 (3)
C15—C16—C17—C18	1.4 (7)	C22—C23—C231—O232	−78.9 (4)
C16—C17—C18—C18A	0.3 (7)	O231—C231—O232—C232	0.3 (5)
C12—N11—C18A—C14A	−2.0 (5)	C23—C231—O232—C232	−177.6 (3)
C12—N11—C18A—C18	179.9 (3)	C231—O232—C232—C233	−90.0 (4)
C14—C14A—C18A—N11	3.2 (6)	C23—C24—C241—C242	−43.0 (5)
C15—C14A—C18A—N11	−177.0 (4)	C24A—C24—C241—C242	137.7 (3)
C14—C14A—C18A—C18	−178.6 (4)	C24—C241—C242—C243	177.1 (3)
C15—C14A—C18A—C18	1.1 (6)	C241—C242—C243—C244	−155.0 (3)
C17—C18—C18A—N11	176.7 (4)	C241—C242—C243—C248	24.8 (5)
C17—C18—C18A—C14A	−1.6 (6)	C248—C243—C244—C245	−1.7 (5)

C17A—N121—C122—N123	0.5 (4)	C242—C243—C244—C245	178.1 (3)
C17A—N121—C122—C12	−176.2 (3)	C243—C244—C245—C246	2.4 (5)
N11—C12—C122—N123	−167.4 (3)	C244—C245—C246—C247	−1.2 (5)
C13—C12—C122—N123	10.2 (5)	C244—C245—C246—C249	175.2 (3)
N11—C12—C122—N121	8.9 (5)	C245—C246—C247—C248	−0.5 (5)
C13—C12—C122—N121	−173.5 (3)	C249—C246—C247—C248	−176.9 (3)
N121—C122—N123—C13A	−0.7 (4)	C246—C247—C248—C243	1.1 (5)
C12—C122—N123—C13A	175.9 (3)	C244—C243—C248—C247	0.0 (5)
C122—N123—C13A—C124	−178.4 (4)	C242—C243—C248—C247	−179.8 (3)
C122—N123—C13A—C17A	0.7 (4)	C38A—N31—C32—C33	2.1 (4)
N123—C13A—C124—C125	179.1 (4)	C38A—N31—C32—C322	−176.9 (3)
C17A—C13A—C124—C125	0.0 (6)	N31—C32—C33—C34	−6.2 (5)
C13A—C124—C125—C126	0.2 (6)	C322—C32—C33—C34	172.7 (3)
C124—C125—C126—C127	0.1 (7)	N31—C32—C33—C331	170.2 (3)
C125—C126—C127—C17A	−0.8 (6)	C322—C32—C33—C331	−10.8 (4)
C122—N121—C17A—C127	179.4 (4)	C32—C33—C34—C34A	4.8 (4)
C122—N121—C17A—C13A	0.0 (4)	C331—C33—C34—C34A	−171.6 (3)
C126—C127—C17A—N121	−178.3 (4)	C32—C33—C34—C341	−173.9 (3)
C126—C127—C17A—C13A	1.0 (6)	C331—C33—C34—C341	9.7 (4)
N123—C13A—C17A—N121	−0.4 (4)	C33—C34—C34A—C35	−178.7 (3)
C124—C13A—C17A—N121	178.8 (3)	C341—C34—C34A—C35	0.0 (4)
N123—C13A—C17A—C127	−179.9 (3)	C33—C34—C34A—C38A	0.1 (4)
C124—C13A—C17A—C127	−0.7 (6)	C341—C34—C34A—C38A	178.8 (3)
C14—C13—C131—O131	−93.5 (7)	C38A—C34A—C35—C36	3.1 (5)
C12—C13—C131—O131	86.4 (7)	C34—C34A—C35—C36	−178.2 (3)
C14—C13—C131—O132	81.5 (8)	C34A—C35—C36—C37	0.3 (5)
C12—C13—C131—O132	−98.6 (7)	C35—C36—C37—C38	−2.8 (5)
O131—C131—O132—C132	−18.8 (15)	C36—C37—C38—C38A	1.9 (5)
C13—C131—O132—C132	166.4 (7)	C32—N31—C38A—C34A	3.3 (4)
C131—O132—C132—C133	−164.1 (8)	C32—N31—C38A—C38	−178.3 (3)
C13—C14—C141—C142	65.7 (6)	C35—C34A—C38A—N31	174.5 (3)
C14A—C14—C141—C142	−115.9 (5)	C34—C34A—C38A—N31	−4.3 (4)
C14—C141—C142—C143	−178.1 (4)	C35—C34A—C38A—C38	−4.0 (4)
C141—C142—C143—C144	−168.6 (4)	C34—C34A—C38A—C38	177.2 (3)
C141—C142—C143—C148	13.7 (6)	C37—C38—C38A—N31	−177.0 (3)
C148—C143—C144—C145	−1.0 (5)	C37—C38—C38A—C34A	1.5 (5)
C142—C143—C144—C145	−178.8 (3)	C37A—N321—C322—N323	0.1 (4)
C143—C144—C145—C146	1.7 (6)	C37A—N321—C322—C32	178.6 (3)
C144—C145—C146—C147	−1.5 (5)	N31—C32—C322—N323	−159.5 (3)
C144—C145—C146—C149	177.5 (3)	C33—C32—C322—N323	21.5 (5)
C145—C146—C147—C148	0.5 (5)	N31—C32—C322—N321	22.2 (4)
C149—C146—C147—C148	−178.5 (3)	C33—C32—C322—N321	−156.8 (3)
C146—C147—C148—C143	0.2 (5)	N321—C322—N323—C33A	0.0 (4)
C144—C143—C148—C147	0.1 (5)	C32—C322—N323—C33A	−178.4 (3)
C142—C143—C148—C147	177.8 (3)	C322—N323—C33A—C324	178.6 (4)
C28A—N21—C22—C23	1.5 (5)	C322—N323—C33A—C37A	−0.1 (4)
C28A—N21—C22—C222	−176.8 (3)	N323—C33A—C324—C325	−177.7 (4)
N21—C22—C23—C24	2.2 (5)	C37A—C33A—C324—C325	1.0 (5)

C222—C22—C23—C24	−179.6 (3)	C33A—C324—C325—C326	−0.4 (6)
N21—C22—C23—C231	−172.5 (3)	C324—C325—C326—C327	0.4 (6)
C222—C22—C23—C231	5.6 (4)	C325—C326—C327—C37A	−0.8 (6)
C22—C23—C24—C24A	−3.7 (4)	C322—N321—C37A—C327	−176.9 (4)
C231—C23—C24—C24A	171.0 (3)	C322—N321—C37A—C33A	−0.1 (4)
C22—C23—C24—C241	176.9 (3)	C326—C327—C37A—N321	177.7 (4)
C231—C23—C24—C241	−8.4 (5)	C326—C327—C37A—C33A	1.4 (5)
C23—C24—C24A—C25	−178.2 (3)	N323—C33A—C37A—N321	0.2 (4)
C241—C24—C24A—C25	1.2 (5)	C324—C33A—C37A—N321	−178.7 (3)
C23—C24—C24A—C28A	1.7 (4)	N323—C33A—C37A—C327	177.4 (3)
C241—C24—C24A—C28A	−178.9 (3)	C324—C33A—C37A—C327	−1.5 (5)
C24—C24A—C25—C26	177.6 (3)	C34—C33—C331—O331	74.2 (4)
C28A—C24A—C25—C26	−2.3 (5)	C32—C33—C331—O331	−102.2 (4)
C24A—C25—C26—C27	1.3 (5)	C34—C33—C331—O332	−105.4 (3)
C25—C26—C27—C28	−0.1 (6)	C32—C33—C331—O332	78.3 (4)
C26—C27—C28—C28A	0.0 (6)	O331—C331—O332—C332	16.0 (5)
C22—N21—C28A—C28	176.2 (3)	C33—C331—O332—C332	−164.4 (3)
C22—N21—C28A—C24A	−3.6 (4)	C331—O332—C332—C333	−93.7 (4)
C27—C28—C28A—N21	179.2 (3)	C33—C34—C341—C342	47.0 (5)
C27—C28—C28A—C24A	−1.0 (5)	C34A—C34—C341—C342	−131.6 (3)
C25—C24A—C28A—N21	−178.1 (3)	C34—C341—C342—C343	174.7 (3)
C24—C24A—C28A—N21	2.0 (5)	C341—C342—C343—C348	−10.9 (6)
C25—C24A—C28A—C28	2.1 (5)	C341—C342—C343—C344	172.0 (4)
C24—C24A—C28A—C28	−177.8 (3)	C348—C343—C344—C345	1.8 (6)
C27A—N221—C222—N223	0.4 (4)	C342—C343—C344—C345	179.1 (4)
C27A—N221—C222—C22	178.9 (3)	C343—C344—C345—C346	−2.2 (6)
N21—C22—C222—N223	147.3 (3)	C344—C345—C346—C347	1.4 (7)
C23—C22—C222—N223	−31.0 (5)	C344—C345—C346—C349	−178.0 (4)
N21—C22—C222—N221	−31.0 (4)	C345—C346—C347—C348	−0.5 (7)
C23—C22—C222—N221	150.7 (3)	C349—C346—C347—C348	179.0 (4)
N221—C222—N223—C23A	0.2 (4)	C344—C343—C348—C347	−0.9 (6)
C22—C222—N223—C23A	−178.2 (3)	C342—C343—C348—C347	−178.0 (4)
C222—N223—C23A—C224	178.4 (4)	C346—C347—C348—C343	0.2 (7)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N121—H121···O231	0.76 (4)	2.19 (4)	2.876 (4)	150 (4)
N221—H221···O331	0.84 (4)	2.13 (4)	2.882 (4)	151 (4)
N321—H321···O131^{i}	0.85 (4)	2.18 (4)	2.885 (4)	142 (3)
C127—H127···N223	0.95	2.58	3.432 (5)	149
C227—H227···N323	0.95	2.62	3.522 (5)	159
C332—H32*B*···*Cg*4^{ii}	0.95	2.80	3.702 (5)	152
C347—H347···*Cg*5^{iii}	0.95	2.70	3.548 (5)	149

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) −*x*+1/2, *y*+1/2, −*z*+3/2; (iii) −*x*+1/2, *y*−1/2, −*z*+3/2.