Acta Cryst C STRUCTURAL CHEMISTRY
ISSN 2053-2296

Design, synthesis and crystallographic study of novel indole-based cyano derivatives as key building blocks for heteropolycyclic compounds of major complexity

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Received 3 October 2017
Accepted 30 October 2017

Edited by D. S. Yufit, University of Durham, England

Keywords: synthetic design; synthesis; heterocyclic compounds; chalcones; dihydrochalcones; enolate esters; crystal structure; molecular conformation; hydrogen bonding; supramolecular assembly; privileged substructures.

CCDC references: 1582887; 1582886; 1582885; 1582884; 1582883; 1582882

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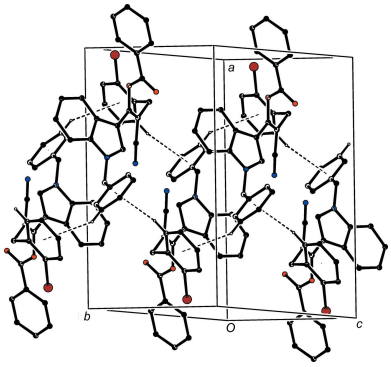
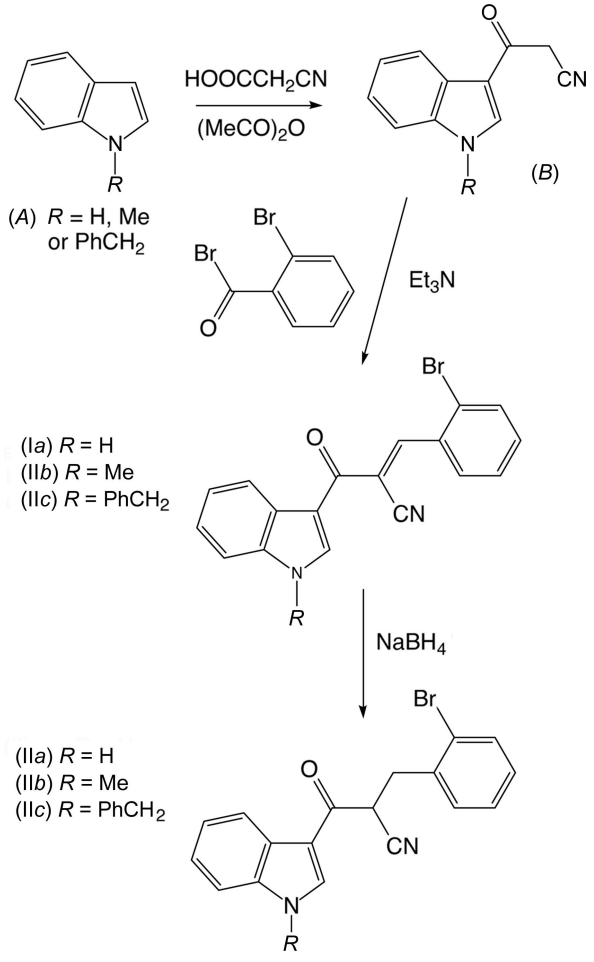
A four-stage reaction sequence has been designed and developed for the synthesis of highly functionalized enolate esters as key building blocks for the synthesis of novel heteropolycyclic compounds of potential pharmaceutical value. The sequence starts with simple commercially available indoles and proceeds *via* 3-(indol-3-yl)-3-oxopropanenitriles, which react with 2-bromobenzaldehyde to form the corresponding chalcones; these are readily reduced to dihydrochalcones, which are in turn acylated to form the enolate esters. The compounds in this sequence have been characterized by IR and ^{1}H and ^{13}C NMR spectroscopy, by mass spectrometry and by elemental analysis. The molecular and supramolecular structures are reported for representative examples, namely (*E*)-3-(2-bromophenyl)-2-(1-methyl-1*H*-indole-3-carbonyl)-acrylonitrile, $C_{19}H_{13}BrN_2O$, (I*b*), (2*RS*)-2-(2-bromobenzyl)-3-(1-methyl-1*H*-indol-3-yl)-3-oxopropanenitrile, $C_{19}H_{15}BrN_2O$, (II*b*), and (2*RS*)-3-(1-benzyl-1*H*-indol-3-yl)-2-(2-bromobenzyl)-3-oxopropanenitrile, $C_{25}H_{19}BrN_2O$, (II*c*), the latter two of which crystallize with Z' = 2, and (*E*)-1-(1-acetyl-1*H*-indol-3-yl)-3-(2-bromophenyl)-2-cyanoprop-1-en-1-yl acetate, $C_{22}H_{17}BrN_2O$, (III), and (*E*)-1-(1-benzyl-1*H*-indol-3-yl)-3-(2-bromophenyl)-2-cyanoprop-1-en-1-yl benzoate, $C_{32}H_{23}BrN_2O$, (IV). The structure of the related chalcone (*E*)-2-benzoyl-3-(2-bromophenyl)prop-2-enenitrile, (V), has been redetermined at 100 K, where it is monoclinic, as opposed to the triclinic form reported at ambient temperature.

1. Introduction

The indole core is present in a wide range of naturally occurring and synthetic compounds and it is regarded as one of the most important 'privileged substructures' because of the wide spectrum of biological activities displayed by many of its derivatives (Biswal *et al.*, 2012), particularly indole-based heteropolycyclic compounds, such as the anticancer agent ellipticine (Stiborová *et al.*, 2011), the antihypertensive drug reserpine (Baumeister *et al.*, 2003) and the anti-obesity agent evodiamine (Wang *et al.*, 2008). As part of our current program on the development of novel routes for the synthesis of new heterocyclic derivatives of biological interest (Abonía, 2014; Abonía *et al.*, 2016; Insuasty *et al.*, 2017; Ramírez-Prada *et al.*, 2017), we now report the design, synthesis and structural study of novel cyano derivatives, containing the indole core in their structures, as valuable key intermediates for the construction of novel indole-based heteropolycyclic compounds of major structural complexity.



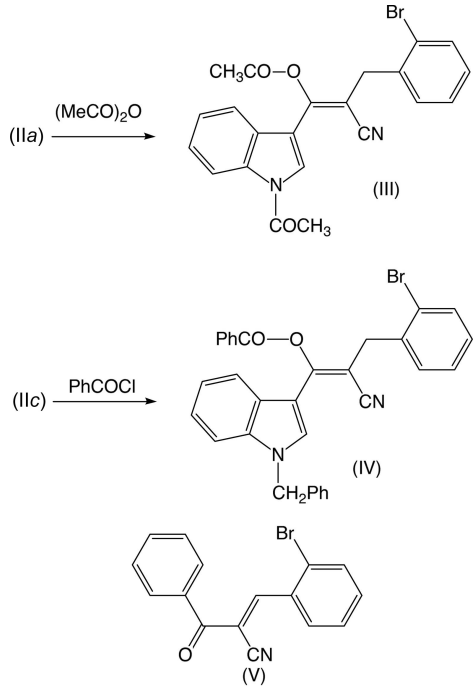
Thus, we describe here a newly designed and developed four-step sequence for the preparation of the esters (*E*)-1-(1-acetyl-1*H*-indol-3-yl)-3-(2-bromophenyl)-2-cyanoprop-1-en-1-yl acetate and (*E*)-1-(1-benzyl-1*H*-indol-3-yl)-3-(2-bromophenyl)-2-cyanoprop-1-en-1-yl benzoate as important synthetic intermediates starting with the cyanoacetylation of the 1*H*-indole [(*A*), where *R* = H, Me or $PhCH_2$] (see Scheme 1) by reaction with cyanoacetic acid in acetic anhydride to provide the corresponding indolyl(oxo)propanenitriles (*B*). The intermediates (*B*) undergo a condensation reaction with 2-bromobenzaldehyde under basic conditions to give the corresponding cyanoheterochalcones (I), in yields of 92–95%, and we report here the structure of the derivative containing an *N*-methyl substituent, namely (*E*)-3-(2-bromophenyl)-2-(1-methyl-1*H*-indole-3-carbonyl)acrylonitrile, (I*b*).



Scheme 1

Reduction of the cyanoheterochalcones using sodium borohydride forms the dihydro analogues (II) and we report the structures of two of these, containing either an *N*-methyl substituent, *i.e.* (2*RS*)-2-(2-bromobenzyl)-3-(1-methyl-1*H*-indol-3-yl)-3-oxopropanenitrile, (II*b*), or an *N*-benzyl substituent, *i.e.* (2*RS*)-3-(1-benzyl-1*H*-indol-3-yl)-2-(2-bromobenzyl)-3-oxopropanenitrile, (II*c*). This reduction of the C=C double bond in chalcones (I) is highly specific, with yields in the range 82–93%, and with no evidence for any reduction of either the carbonyl or the cyano groups.

Acetylation, using acetic anhydride, of the dihydrochalcone containing an N—H group, compound (II*a*), gives the diacetyl product (*E*)-1-(1-acetyl-1*H*-indol-3-yl)-3-(2-bromophenyl)-2-cyanoprop-1-en-1-yl acetate, (III), while benzoylation using benzoyl chloride of the *N*-benzyl analogue (II*c*) yielded (*E*)-1-(1-benzyl-1*H*-indol-3-yl)-3-(2-bromophenyl)-2-cyanoprop-1-en-1-yl benzoate, (IV) (see Scheme 2), and we report here the structures of both (III) and (IV), which have been designed as synthetic precursors in order to generate complex heteropentacyclic derivatives.



Scheme 2

We also report here a redetermination of the structure of the chalcone 2-benzoyl-3-(2-bromophenyl)acrylonitrile, (V), which is related to chalcone (I), but with a simple phenyl group in place of the substituted indolyl unit. The structure of this chalcone has been reported recently at ambient temperature, in the space group $P\overline{1}$, with $Z' = 2$ (Dhiman *et al.*, 2016), but the structure was refined only to $R1 = 0.157$ and $wR2 = 0.341$, with $S = 1.410$. As we had occasion to prepare this compound at an early stage in the present study, we have taken the opportunity to collect a new data set at 100 K, where the compound is monoclinic, space group $P2_1/n$ with $Z' = 1$, suggesting the possibility of temperature-dependent polymorphism.

2. Experimental

2.1. Synthesis and crystallization

The initial indoles (*A*) (see Scheme 1), where *R* = H, Me or $PhCH_2$, are all commercially available. For the synthesis of the indolyl(oxo)propanenitrile precursors (*B*), a mixture of the appropriate indole (1.0 mmol), cyanoacetic acid (1.2 mmol) and acetic anhydride (2–3 ml) was held at 380 K for 1.5 h, with stirring throughout, and the progress of the reactions was

monitored using thin-layer chromatography (TLC). When the indole component had been completely consumed, the solutions were held at 273 K overnight, and the resulting solid products (*B*) were collected by filtration and washed with cold methanol (2 × 1.0 ml).

3-(1*H*-Indol-3-yl)-3-oxopropanenitrile: yield 79%, m.p. 516 K; IR (KBr): 3225 (N—H), 2253 (CN), 1643 (C═O), 1581 (C═C) cm^{-1}; ^{1}H NMR (DMSO-d_6): δ 4.47 (*s*, CH_2), 7.19–7.27 (*m*, 2H), 7.47–7.52 (*m*, 1H), 8.10–8.16 (*m*, 1H), 8.36 (*s*, 1H), 12.14 (*s*, 1H, NH); MS (70 eV) *m*/*z* (%): 184 (23, M^+) 144 (100), 116 (27), 89 (26).

3-(1-Methyl-1*H*-indol-3-yl)-3-oxopropanenitrile: yield 73%, m.p. 426 K; IR (KBr): 3110 (C—H), 2250 (CN), 1643 (C═O), 1581 (C═C) cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 3.88 (*s*, 2H, CH_2), 3.90 (*s*, 3H, CH_3), 7.31–7.48 (*m*, 3H), 7.81 (*s*, 1H), 8.27–8.37 (*m*, 1H); ^{13}C NMR ($CDCl_3$): δ 29.6 (CH_2), 33.9 (CH_3), 110.1, 114.2 (Cq), 114.9 (CN), 122.3, 123.5, 124.2, 126.1 (Cq), 136.2, 137.5 (Cq), 180.5 (C═O).

3-(1-Benzyl-1*H*-indol-3-yl)-3-oxopropanenitrtile: yield 72%, m.p. 397 K; IR (KBr): 3105 (C—H), 2252 (CN), 1662 (C═O), 1602 (C═C) cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 3.90 (*s*, 2H, CH_2CN), 5.41 (*s*, 2H, CH_2Ph), 7.21 (*dd*, *J* = 7.5, 2.1 Hz, 2H), 7.30–7.40 (*m*, 6H), 7.85 (*s*, 1H), 8.37 (*dd*, *J* = 8.4, 1.7 Hz, 1H), ^{13}C NMR ($CDCl_3$): δ 29.8 (CH_2), 51.1 (CH_2Ph), 110.6, 114.7 (Cq), 114.8 (CN), 122.5, 123.6, 124.4, 126.3 (Cq), 127.2, 128.5, 129.2, 135.1 (Cq), 135.2, 137.1 (Cq), 180.6 (C═O). Analysis found: C 78.9, H 5.4, N 9.9%; $C_{18}H_{14}N_2O$ requires: C 78.8, H 5.1, N 10.2%.

For the synthesis of chalcones (I), a mixture of the corresponding oxopropanenitrile (*B*) (1.0 mmol), 2-bromobenzaldehyde (1.2 mmol) and triethylamine (1.2 ml) in ethanol (4 ml) was heated under reflux for 2 h. Once precursor (*B*) had been consumed, as shown by TLC, the mixtures were cooled to ambient temperature and the resulting solid products (I) were collected by filtration and washed with cold methanol (2 × 1.0 ml); no further purification was required.

(*E*)-3-(2-Bromophenyl)-2-(1*H*-indole-3-carbonyl)acrylonitrile, (I*a*): yield 95%, m.p. 522 K; IR (ATR): 3174 (N—H), 2218 (CN), 1631 (C═O), 1579 (C═C) cm^{-1}; ^{1}H NMR (DMSO-d_6): δ 7.26–7.35 (*m*, 2H), 7.53 (*t*, *J* = 8.1 Hz, 1H), 7.58 (*d*, *J* = 8.0 Hz, 1H), 7.64 (*t*, *J* = 8.0 Hz, 1H), 7.86 (*d*, *J* = 7.9 Hz, 1H), 8.13 (*d*, *J* = 7.9 Hz, 1H), 8.22 (*d*, *J* = 8.0 Hz, 1H), 8.28 (*s*, 1H), 8.52 (*s*, 1H), 12.39 (*s*, 1H, NH); ^{13}C NMR (DMSO-d_6): δ 113.1 (Cq), 113.9, 115.9 (CN), 117.0, 121.8, 122.8 (Cq), 123.1 (Cq), 125.0, 126.5, 128.7 (Cq), 130.6, 131.4, 133.2 (Cq), 133.7, 136.8 (Cq), 137.2, 151.0, 180.9 (C═O); MS (70 eV) *m*/*z* (%): 352/350 (22/22, M^+) 144 (100), 271 (41), 116 (37), 89 (62). Analysis found: C 61.4, H 3.0, N 8.1%; $C_{18}H_{11}BrN_2O$ requires: C 61.6, H 3.2, N 8.0%.

(*E*)-3-(2-Bromophenyl)-2-(1-methyl-1*H*-indole-3-carbonyl)-acrylonitrile, (I*b*): yield 92%, m.p. 436 K; IR (ATR): 3018 (C—H), 2212 (CN), 1635 (C═O), 1581 (C═C) cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 3.95 (*s*, 3H, CH_3), 7.35–7.57 (*m*, 5H), 7.75 (*dd*, *J* = 8.0, 1.3 Hz, 1H), 8.20 (*dd*, *J* = 7.8, 1.7 Hz, 1H), 8.35 (*s*, 1H), 8.44–8.54 (*m*, 1H), 8.58 (*s*, 1H); ^{13}C NMR ($CDCl_3$): 34.0 (CH_3), 110.0, 113.9 (Cq), 114.1 (Cq), 117.8 (CN), 123.1, 123.5, 124.3, 126.1 (Cq), 127.4 (Cq), 128.0, 130.0, 132.8 (Cq), 132.9, 133.5, 137.2, 137.4 (Cq), 152.2, 178.6 (C═O); MS (70 eV) *m*/*z* (%): 366/364 (38/40, M^+), 285 (100), 236 (19), 158 (66), 152 (35). Analysis found: C 62.1, H 3.6, N 8.0%; $C_{19}H_{13}BrN_2O$ requires: C 62.5, H 3.6, N 7.7%.

(*E*)-2-(1-Benzyl-1*H*-indole-3-carbonyl)-3-(2-bromophenyl)-acrylonitrile, (I*c*): yield 92%, m.p. 453 K; IR (ATR): 3068 (C—H), 2225 (CN), 1616 (C═O), 1517 (C═C); ^{1}H NMR ($CDCl_3$): δ 5.44 (*s*, 2H), 7.19–7.29 (*m*, 2H), 7.33–7.45 (*m*, 7H), 7.50 (*td*, *J* = 7.7, 1.2 Hz, 1H), 7.73 (*dd*, *J* = 8.0, 1.1 Hz, 1H), 8.19 (*dd*, *J* = 7.8, 1.6 Hz, 1H), 8.36 (*s*, 1H), 8.46–8.63 (*m*, 2H); ^{13}C NMR ($CDCl_3$): δ 51.2 (CH_2Ph), 110.5, 114.2 (Cq), 114.5 (Cq), 117,4 (CN), 123.1, 123.6, 124.4, 126.1 (Cq), 127.3, 127.6 (Cq), 128.0, 128.5, 129.2, 130.0, 132.7 (Cq), 132.9, 133.4, 135.1 (Cq), 136.6, 137.0 (Cq), 151.9, 178.3 (C═O); MS (70 eV) *m*/*z* (%): 442/440 (8/8, M^+), 361 (12), 234 (5), 91 (100). Analysis found: C 67.9, H 4.0, N 6.5%; $C_{26}H_{17}BrN_2O$ requires: C 68.0, H 3.9, N 6.4%.

For the reduction of chalcones (I) to dihydrochalcones (II), the appropriate chalcone (I) (1.0 mmol) was dissolved in a mixture of 1,4-dioxane and methanol (1:4 *v*/*v*, 5 ml) and the solution were stirred vigorously at 343 K as sodium borohydride (1.0 equivalent) was added portionwise. When the starting chalcone had been consumed, as indicated by TLC, the solutions were cooled to ambient temperature and the resulting precipitates were collected by filtration and washed with cold methanol (2 × 1.0 ml). No further purification was necessary.

2-(2-Bromobenzyl)-3-(1*H*-indol-3-yl)-3-oxopropanenitrile, (II*a*): yield 82%, m.p. 519 K; IR (ATR): 3259 (N—H), 2247 (CN), 1616 (C═O), 1579 (C═C) cm^{-1}; ^{1}H NMR (DMSO-d_6): δ 3.34–3.37 (*m*, 2H), 5.06 (*t*, *J* = 9.6 Hz, 1H), 7.21–7.29 (*m*, 3H), 7.38 (*t*, *J* = 8.3 Hz, 1H), 7.52 (*bt*, 2H), 7.64 (*d*, *J* = 8.3 Hz, 1H), 8.18 (*d*, *J* = 8.4 Hz, 1H), 8.61 (*s*, 1H), 12.27 (*s*, 1H, NH). ^{13}C (DMSO-d_6): δ 35.4, 39.9, 112.5, 113.7 (Cq), 118.4 (CN), 121.5, 122.5, 123.6, 124.2 (Cq), 125.4 (Cq), 127.8, 128.4, 129.3 (Cq), 131.6, 132.7, 136.0 (Cq), 136.8, 184.3 (C═O); MS (70 eV) *m*/*z* (%): 354/352 (2.9/2.5, M^+) 144 (100), 273 (43), 116 (23), 89 (13). Analysis found: C 61.1, H 3.9, N 7.8%; $C_{18}H_{13}BrN_2O$ requires: C 61.2, H 3.7, N 7.9%.

2-(2-Bromobenzyl)-3-(1-methyl-1*H*-indol-3-yl)-3-oxopropanenitrile, (II*b*): yield 93%, m.p. 419 K; IR (ATR): 3059 (C—H), 2249 (CN), 1641 (C═O), 1577 (C═C) cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 3.38 (*dd*, *J* = 13.8, 9.5 Hz, 1H), 3.61 (*dd*, *J* = 13.8, 6.1 Hz, 1H), 3.89 (*s*, 3H, CH_3), 4.53 (*dd*, *J* = 9.5, 6.1 Hz, 1H), 7.19 (*td*, *J* = 7.7, 1.7 Hz, 1H), 7.28–7.50 (*m*, 5H), 7.61 (*dd*, *J* = 8.0, 1.3 Hz, 1H), 7.89 (*s*, 1H), 8.43 (*ddd*, *J* = 6.8, 3.0, 1.5 Hz, 1H); ^{13}C NMR ($CDCl_3$): δ 33.9 (NCH_3), 36.5 (CH_2), 40.4 (CH), 110, 114.1 (Cq), 117.9 (CN), 122.7, 123.5, 124.2 (Cq), 124.3, 126.5 (Cq), 128.1, 129.4, 132.4, 132.9, 135.8 (Cq), 136.2, 137.6 (Cq), 183.1 (C═O); MS (70 eV) *m*/*z* (%): 368/366 (2/2, M^+), 287 (9), 158 (100), 130 (17), 83 (61). Analysis found: C 62.3, H 4.1, N 8.0%; $C_{19}H_{15}BrN_2O$ requires: C 62.1, H 4.1, N 7.6%.

3-(1-Benzyl-1*H*-indol-3-yl)-2-(2-bromobenzyl)-3-oxopropanenitrile, (II*c*): yield 91%, m.p. 419 K; IR (ATR): 3059 (C—H), 2250 (CN), 1641 (C═O), 1568 (C═C) cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 3.34 (*dd*, *J* = 13.7, 9.8 Hz, 1H), 3.55 (*dd*, *J* = 13.6, 6.0 Hz, 1H), 4.50 (*dd*, *J* = 9.8, 6.0 Hz, 1H), 5.29–5.45 (*m*, 2H), 7.12–7.32 (*m*, 4H), 7.33–7.47 (*m*, 7H), 7.56 (*dd*, *J* = 8.0, 1.2 Hz, 1H), 7.89 (*s*, 1H), 8.42–8.50 (*m*, 1H); ^{13}C ($CDCl_3$): δ 37.1

(CH_2), 40.7 (CH), 51.1 (CH_2Ph), 110.4, 114.5 (Cq), 117.6 (CN), 122.8, 123.7, 124.2 (Cq), 124.4, 126.7 (Cq), 127.6, 128.1, 128.7, 129.3, 129.4, 132.4, 132.9, 134.7 (Cq), 135.3, 135.6 (Cq), 137.3 (Cq), 183.6 (C═O); MS (70 eV) *m*/*z* (%): 444/442 (8/8, M^+), 234 (15), 149 (8), 91 (100). Analysis found: C 66.8, H 4.3, N 6.9%; $C_{25}H_{19}BrN_2O$ requires: C 67.7, H 4.3, N 6.3%.

For the synthesis of (*E*)-1-(1-acetyl-1*H*-indol-3-yl)-3-(2-bromophenyl)-2-cyanoprop-1-en-1-yl acetate, (III), a mixture of dihydrochalcone (II*a*) (1.4 mmol) and acetic anhydride (5 ml) was stirred at 373 K. When the dihydrochalcone had all been consumed, as indicated by TLC, the residual acetyl compounds were removed under reduced pressure and the resulting solid was purified by flash chromatography on silica gel using hexane/diethyl ether (60/40, then 50/50, then 60/40 *v*/*v*) as eluent to give (III): yield 56%, m.p. 424 K; IR (ATR): 3007 (C—H), 2242 (CN), 1721 (C═O), 1680 (C═O), 1533 (C═C) cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 2.30 (*s*, 3H, CH_3), 2.73 (*s*, 3H, CH_3), 3.89 (*s*, 2H, CH_2), 7.22 (*td*, *J* = 7.7, 1.8 Hz, 1H), 7.32–7.50 (*m*, 4H), 7.57–7.78 (*m*, 2H), 8.18 (*s*, 1H), 8.46–8.60 (*m*, 1H), ^{13}C NMR ($CDCl_3$): δ 20.7 (CH_3), 23.9 (CH_3), 34.6 (CH_2), 101.9 (Cq), 114.5 (Cq), 117.2, 118.1 (CN), 119.7, 124.5 (Cq), 124.6, 126.2, 127.3 (Cq), 127.8, 127.9, 129.2, 130.7, 133.2, 135.5 (Cq), 135.6 (Cq), 154.9 (Cq), 167.0 (OC═O), 168.6 (NC═O); MS (70 eV) *m*/*z* (%): 438/436 (12/12, M^+), 396/394 (60/59), 273 (100), 186 (14). Analysis found: C60.7, H 4.1, N 6.2%; $C_{22}H_{17}BrN_2O_3$ requires: C 60.4, H 3.9, N 6.4%.

For the synthesis of (*E*)-1-(1-benzyl-1*H*-indol-3-yl)-3-(2-bromophenyl)-2-cyanoprop-1-en-1-yl benzoate, (IV), a mixture of dihydrochalcone (II*c*) (1.0 mmol), chloroform (7.0 ml) and triethylamine (5.0 mmol) was stirred for 20 min at ambient temperature. Benzoyl chloride (4.0 mmol) was then added dropwise and stirring was continued until TLC showed that the dihydrochalcone had all been consumed. The mixture was then extracted with a saturated aqueous solution of sodium hydrogen carbonate (2 × 3 ml) and the organic phase was dried over anhydrous magnesium sulfate. The solvent was removed under reduced pressure and the crude product (IV) was purified by flash chromatography using dichloromethane/hexane (45/55, then 50/50, then 55/45 and finally 60/40 *v*/*v*) to give an 87/13 mixture of the *E* and *Z* isomers. Yield 88%, m.p. 394–395 K; IR (ATR): 3067 (C—H), 2210 (CN), 1737 (C═O), 1560 (C═C), 1238 (C—O—C) cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 3.90 (*s*, 2H, CH_2Ar), 5.39 (*s*, 2H, CH_2Ph), 6.89–7.19 (*m*, 16H), 8.05–8.32 (*m*, 3H); ^{13}C NMR ($CDCl_3$): δ 34.7 (CH_2Ar), 50.9 (CH_2Ph), 96.8 (Cq), 108.3 (Cq), 110.8, 119.3 (CN), 120.5, 121.6, 123.0, 124.7 (Cq), 126.1 (Cq), 127.0, 127.8, 128.1, 128.2 (Cq), 128.8, 128.9 (×2 C, CH and Cq), 129.0, 130.4, 130.8, 131.9, 133.0, 134.2, 135.9 (Cq), 136.3 (Cq), 157.2 (Cq), 163.1 (OC═O); MS (70 eV) *m*/*z* (%): 547/545 (32/30, M^+), 234 (40), 105 (90), 90 (100). Analysis found: C 70.0, H 4.5, N 5.0%; $C_{32}H_{21}BrN_2O$ requires: C 70.2, H 4.2, N 5.1%.

(*E*)-2-Benzoyl-3-(2-bromophenyl)acrylonitrile, (V), was obtained as an orange solid in a manner similar to that used for the the preparation of chalcones (I), but using benzoylacetonitrile and 2-bromobenzaldehyde. Yield 52%, m.p. 399–400 K (399–401 K; Dhiman *et al.*, 2016); IR (ATR): 3088 (C—H), 2225 (CN), 1651 (C═O), 1577 (C═C) cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 7.43 (*t*, *J* = 7.7 Hz, 1H), 7.48–7.63 (*m*, 3H), 7.65–7.79 (*m*, 2H), 7.88–8.03 (*m*, 2H), 8.27 (*d*, *J* = 7.9 Hz, 1H), 8.38 (*s*, 1H); ^{13}C NMR ($CDCl_3$): δ 113.7 (Cq), 115.8 (CN), 126.5 (Cq), 128.2, 128.9, 129.6, 130.2, 132.2 (Cq), 133.6, 133.7, 133.8, 135.5 (Cq), 154.1, 188.7 (C═O).

Crystals of compounds (I*b*), (II*b*), (II*c*) and (V) suitable for single-crystal X-ray diffraction were grown by slow evaporation, at ambient temperature and in the presence of air, of solutions in chloroform; crystals of (III) and (IV) were grown, under similar conditions from solutions in acetic acid/acetic anhydride and dichloromethane/hexane (1:1 *v*/*v*), respectively.

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. For all the compounds described here, the atom labelling is based on the systematic chemical names. Several low-angle reflections which had been attenuated by the beam stop were omitted from the refinements; thus, for (III), 120 and $\bar{1}$22, and for (V), 110. All H atoms were located in difference maps and then treated as riding atoms in geometrically idealized positions, with C—H = 0.95 (aromatic), 0.98 (CH_3), 0.99 (CH_2) or 1.00 Å (aliphatic), 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. In the final analysis of variance for (II*c*), there was a large value, *i.e.* 3.292, of K = mean(F_o^2)/mean(F_c^2) for the group of 1060 very weak reflections having F_c/F_c(max) in the range 0.000 < F_c/F_c(max) < 0.010; the corresponding value for (III) was 1.727 for 440 reflections having F_c/F_c(max) in the range 0.000 < F_c/F_c(max) < 0.008, and for (IV) was 1.612 for 728 reflections having F_c/F_c(max) in the range 0.000 < F_c/F_c(max) < 0.014.

3. Results and discussion

Chalcone (I*b*) and esters (III) and (IV) all crystallize with Z' = 1, but dihydrochalcones (II*b*) and (II*c*) both crystallize with Z' = 2, albeit in different space groups, *viz.* $P2_1/n$ and $P\bar{1}$, respectively. For each of (II*b*) and (II*c*), examination of the atomic coordinates using *PLATON* (Spek, 2009) revealed no additional symmetry, although the two molecules in the selected asymmetric unit of (II*c*) are approximately, but not exactly, related by a noncrystallographic inversion centre at ($\frac{1}{4}$, $\frac{3}{4}$, $\frac{3}{4}$).

In each of the molecules in the structures reported here, there is a series of substituents, *i.e.* benzyl or benzylidene, carbonyl or carboxylate, cyano, and indolyl or phenyl, disposed about a central C—C bond, and the relative orientations of these substituents, as indicated by the relevant torsion angles (Table 2), show some interesting variations. Thus, for example, the carbonyl and cyano substituents in compounds (I*b*) and (V) adopt mutually *trans* and *cis* arrangements, respectively; likewise, in both (III) and (IV), these two substituents are *trans* to one another, whereas these substituents exhibit synclinal arrangements in all of the independent molecules of (II*b*) and (II*c*). In general, the bromo

Table 1
Experimental details.

	(I*b*)	(II*b*)	(II*c*)
Crystal data			
Chemical formula	$C_{19}H_{13}BrN_2O$	$C_{19}H_{15}BrN_2O$	$C_{25}H_{19}BrN_2O$
M_r	365.22	367.24	443.32
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/n$	Triclinic, $P\overline{1}$
Temperature (K)	100	100	100
a, b, c (Å)	9.188 (5), 9.308 (5), 10.887 (5)	13.728 (6), 14.901 (6), 15.507 (6)	10.912 (4), 12.544 (8), 15.583 (7)
α, β, γ (°)	113.56 (2), 90.782 (18), 116.173 (16)	90, 92.165 (19), 90	89.23 (4), 79.49 (3), 73.20 (3)
V (Å^3)	745.3 (7)	3170 (2)	2006.0 (18)
Z	2	8	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	2.76	2.60	2.07
Crystal size (mm)	0.33 × 0.21 × 0.19	0.40 × 0.30 × 0.10	0.22 × 0.21 × 0.10
Data collection			
Diffractometer	Bruker D8 Venture	Bruker D8 Venture	Bruker D8 Venture
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)	Multi-scan (*SADABS*; Bruker, 2016)	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.446, 0.591	0.489, 0.771	0.737, 0.813
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	46632, 3467, 3296	79963, 7297, 5456	96931, 9244, 6872
R_{int}	0.040	0.090	0.112
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.653	0.651	0.651
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.020, 0.049, 1.07	0.034, 0.072, 1.05	0.042, 0.084, 1.02
No. of reflections	3467	7297	9244
No. of parameters	209	417	523
H-atom treatment	H-atom parameters constrained $w = 1/[\sigma^2(F_o^2) + (0.0202P)^2 + 0.6373P]$ where $P = (F_o^2 + 2F_c^2)/3$	H-atom parameters constrained $w = 1/[\sigma^2(F_o^2) + (0.0217P)^2 + 3.7392P]$ where $P = (F_o^2 + 2F_c^2)/3$	H-atom parameters constrained $w = 1/[\sigma^2(F_o^2) + (0.025P)^2 + 2.2423P]$ where $P = (F_o^2 + 2F_c^2)/3$
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.40, −0.39	0.54, −0.76	0.40, −0.57

	(III)	(IV)	(V)
Crystal data			
Chemical formula	$C_{22}H_{17}BrN_2O_3$	$C_{32}H_{23}BrN_2O_2$	$C_{16}H_{10}BrNO$
M_r	437.28	547.42	312.15
Crystal system, space group	Monoclinic, $C2/c$	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/n$
Temperature (K)	100	100	100
a, b, c (Å)	19.406 (9), 8.773 (4), 23.028 (9)	14.886 (4), 8.129 (3), 21.432 (6)	10.986 (4), 8.977 (4), 13.209 (5)
α, β, γ (°)	90, 103.318 (15), 90	90, 104.638 (12), 90	90, 97.140 (17), 90
V (Å^3)	3815 (3)	2509.3 (14)	1292.6 (9)
Z	8	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	2.18	1.67	3.17
Crystal size (mm)	0.42 × 0.32 × 0.24	0.21 × 0.12 × 0.12	0.27 × 0.26 × 0.23
Data collection			
Diffractometer	Bruker D8 Venture	Bruker D8 Venture	Bruker D8 Venture
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)	Multi-scan (*SADABS*; Bruker, 2016)	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.420, 0.594	0.676, 0.818	0.366, 0.482
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	49228, 4379, 3435	87392, 6999, 5828	30048, 2985, 2754
R_{int}	0.057	0.074	0.036
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.650	0.693	0.652
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.056, 0.136, 1.08	0.036, 0.086, 1.05	0.022, 0.053, 1.11
No. of reflections	4379	6999	2985
No. of parameters	255	334	172
H-atom treatment	H-atom parameters constrained $w = 1/[\sigma^2(F_o^2) + (0.0427P)^2 + 18.903P]$ where $P = (F_o^2 + 2F_c^2)/3$	H-atom parameters constrained $w = 1/[\sigma^2(F_o^2) + (0.0363P)^2 + 2.1078P]$ where $P = (F_o^2 + 2F_c^2)/3$	H-atom parameters constrained $w = 1/[\sigma^2(F_o^2) + (0.023P)^2 + 0.9733P]$ where $P = (F_o^2 + 2F_c^2)/3$
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.80, −1.90	0.46, −0.94	0.34, −0.52

Computer programs: *APEX3* (Bruker, 2016), *SAINT* (Bruker, 2016), *SIR92* (Altomare *et al.*, 1994), *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

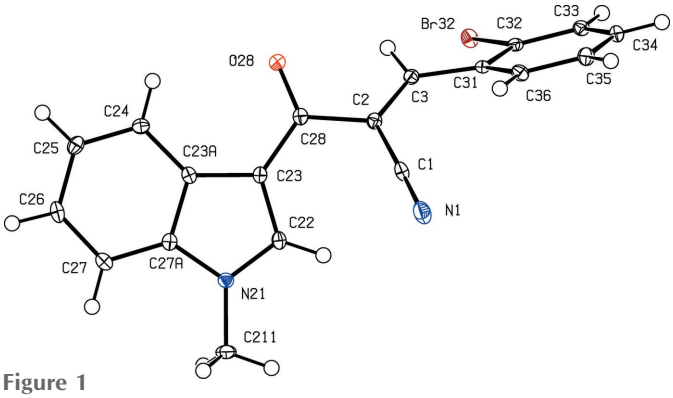


Figure 1
The molecular structure of compound (I*b*), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

substituents occupy locations remote from the cyano substituents (*cf.* Figs. 1–6).

In chalcone (I*b*) (Fig. 1), the central spacer unit comprising atoms N1/C1–C3/C28/C31 is planar, as expected, but the two cyclic substituents are both twisted out of this plane. Thus, the dihedral angle between the planes of the spacer unit and the phenyl ring is 39.45 (6)°, while the dihedral angle between the spacer and the indolyl unit is 36.23 (5)°, with a dihedral angle between the indolyl and phenyl ring systems of 75.25 (4)°; hence, atoms C23 and O28 are displaced on either side the plane of the central spacer by 0.581 (2) and 0.507 (2) Å, respectively. The molecule of compound (I*b*) thus exhibits no internal symmetry and so it is conformationally chiral; the centrosymmetric space group (Table 1) indicates that equal numbers of the two conformational enantiomers are present. The bond lengths present no unusual features (*cf.* Allen *et al.*, 1987); in particular, they provide no evidence for any delocalization of charge from indole atom N21 onto carbonyl atom O28.

The molecules of dihydrochalcones (II*b*) and (II*c*) (Figs. 2 and 3) both contain a stereogenic centre. For each of these compounds, the asymmetric units were selected such that the type 1 molecule, containing atom Br12, has an *R* configuration at atom C12, while the type 2 molecule, containing atom Br22, has an *S* configuration at atom C22. This ensures that the two independent molecules in the selected asymmetric units are linked within the asymmetric units by hydrogen bonds having an aromatic C—H unit as the donor (Table 3), and having an O- or N-atom acceptor, respectively. The centrosymmetric space groups for (II*b*) and (II*c*) (Table 1) confirm that each of

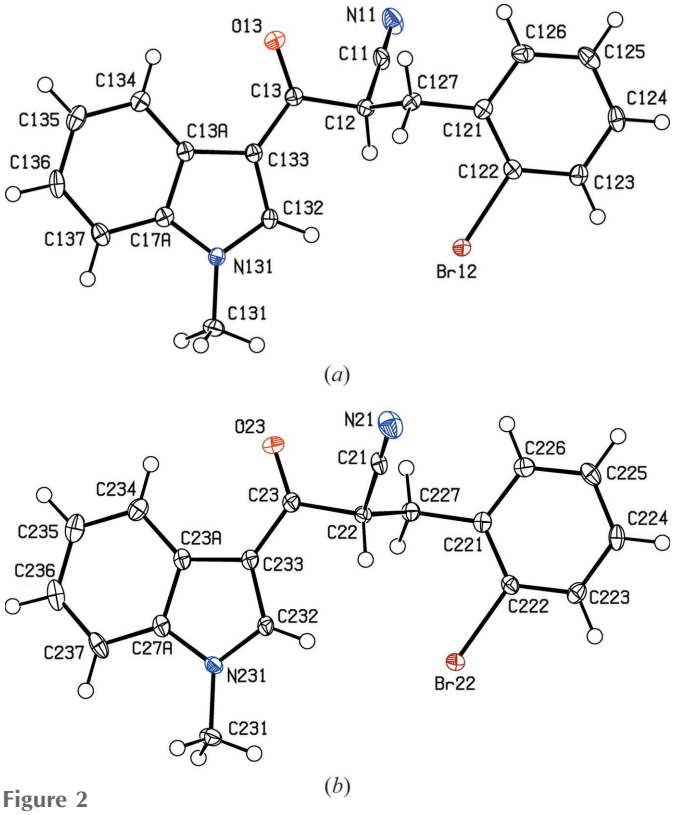


Figure 2
The structures of the two independent molecules in the structure of compound (II*b*), showing the atom-labelling scheme for (*a*) a type 1 molecule having the (2*R*)-configuration and (*b*) a type 2 molecule having the (2*S*)-configuration. Displacement ellipsoids are drawn at the 50% probability level.

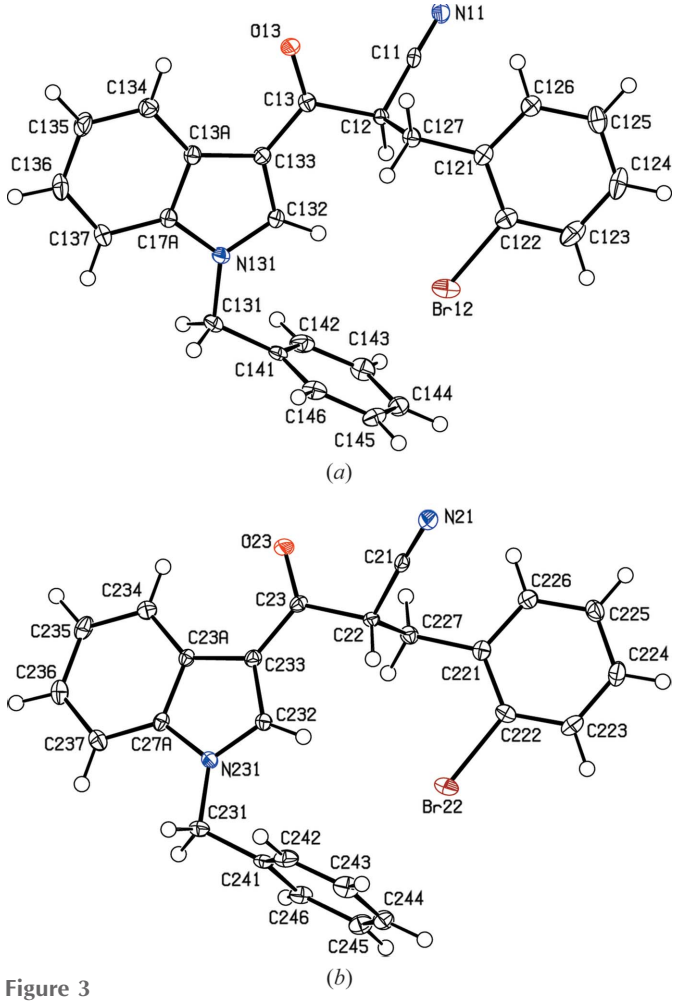


Figure 3
The structures of the two independent molecules in the structure of compound (II*c*), showing the atom-labelling scheme for (*a*) a type 1 molecule having the (2*R*)-configuration and (*b*) a type 2 moleucle having the (2*S*)-configuration. Displacement ellipsoids are drawn at the 50% probability level.

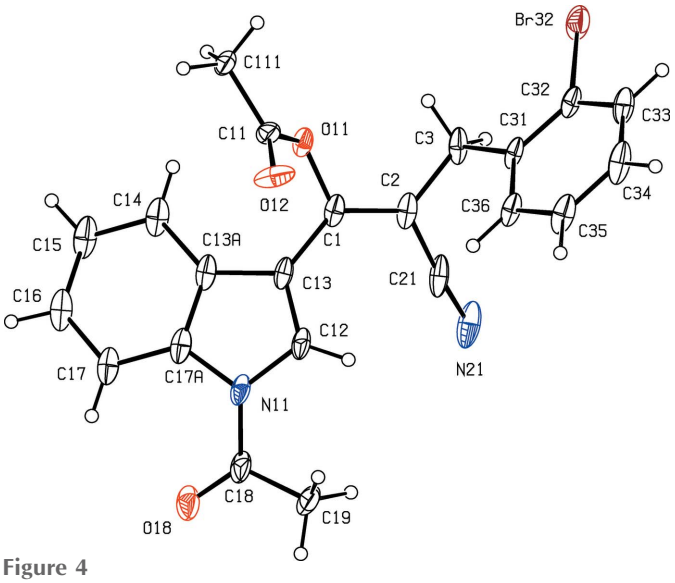


Figure 4
The molecular structure of compound (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

these compounds has crystallized as a racemic mixture, as expected, since the synthetic pathway (see Scheme 1) involves no reagents capable of promoting enantioselectivity. In each of the independent molecules of compound (II*b*), the atoms of the benzyl unit are effectively coplanar, as are those of the indolyl-C(=O)—C unit; the maximum deviation of any of the component atoms from this latter plane is 0.0447 (19) Å for atom C12, with an r.m.s. deviation of 0.026 Å in the type 1 molecule and of 0.024 (2) Å for atom C22 with an r.m.s. deviation of 0.012 Å in the type 2 molecule. Moreover, these

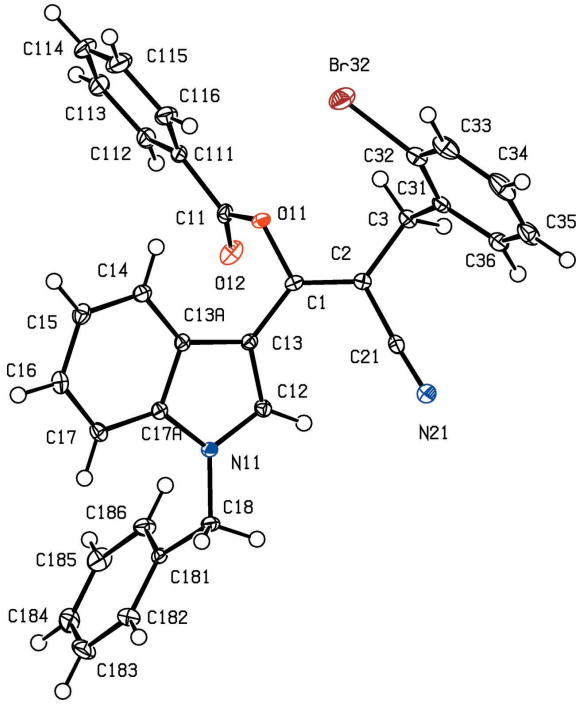


Figure 5
The molecular structure of compound (IV), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

Table 2
Selected torsion angles (°).

	(Indolyl)C—C—C—C(N)[a]	O—C—C—C(N)[b]	(NC)C—C—C—C(Br)[c]
(I*b*)	−29.8 (2)	152.77 (14)	147.66 (16)
(II*b*), *x* = 1	141.1 (2)	−39.4 (3)	−77.7 (3)
(II*b*), *x* = 2	−139.9 (2)	41.5 (3)	50.5 (3)
(II*c*), *x* = 1	151.1 (2)	−31.9 (3)	−93.1 (3)
(II*c*), *x* = 2	−150.1 (2)	34.5 (3)	97.4 (3)
(III)	6.6 (5)	−167.3 (3)	171.0 (3)
(IV)	1.0 (2)	174.10 (13)	−98.04 (10)
(V)	−153.71 (14)	27.4 (2)	134.3 (16)

Notes: (*a*) In (I*b*), C23—C28—C2—C1; in (II*b*) and (II*c*), C*x*33═C*x*34—C*x*2—C*x*1 (*x* = 1 or 2); in (III) and (IV), C13—C1—C2—C21; in (V), C21—C27—C2—C1. (*b*) In (I*b*), O28—C28—C2—C1; in (II*b*) and (II*c*), O*x*23—C*x*3—C*x*2—C*x*1 (*x* = 1 or 2); in (III) and (IV), O11—C1—C2—C21; in (V), O27—C27—C2—C1. (*b*) In (I*b*) and (V), C2—C3—C31—C32; in (II*b*) and (II*c*), C*x*2—C*x*27—C*x*21—C*x*22 (*x* = 1 or 2); in (III) and (IV), C2—C3—C31—C32.

Table 3
Hydrogen bonds and short intra- and intermolecular contacts (Å, °).

*Cg*1, *Cg*2 and *Cg*3 represent the centroids of the C31–C36, C13*A*/C14–C17/C17*A* and C181–C196 rings, respectively.

Compound	*D*—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
(I*b*)	C24—H24···*Cg*1^{i}	0.95	2.77	3.544 (3)	139
(II*b*)	C125—H125···O23	0.95	2.57	3.446 (3)	153
(II*c*)	C225—H225···N11	0.95	2.62	3.304 (4)	129
	C231—H23*A*···O23^{ii}	0.95	2.57	3.415 (4)	143
(III)	C17—H17···O18^{iii}	0.95	2.45	3.133 (5)	129
	C35—H35···*Cg*2^{iv}	0.95	2.76	3.582 (5)	146
(IV)	C36—H36···*Cg*3^{v}	0.95	2.92	3.739 (2)	146
	C183—H183···*Cg*1^{vi}	0.95	2.97	2.807 (2)	148
(V)	C33—H33···N1^{i}	0.95	2.55	3.425 (3)	154
	C36—H36···O27^{vii}	0.95	2.59	3.459 (2)	153
	C3—H3···*Cg*1^{vi}	0.95	2.73	3.431 (2)	131
	C23—H23···*Cg*1^{viii}	0.95	2.69	3.506 (2)	144

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

two units are almost parallel in each molecule, with dihedral angles between them of only 5.78 (5) and 6.10 (5)° in the molecules of types 1 and 2, respectively (Fig. 2). Entirely similar comments apply to the molecules of compound (II*c*) (Fig. 3), where the dihedral angles between the planes of the benzyl and indolylcarbonyl units are 11.91 (6) and 13.09 (7)° in the molecules of types 1 and 2, respectively; the dihedral

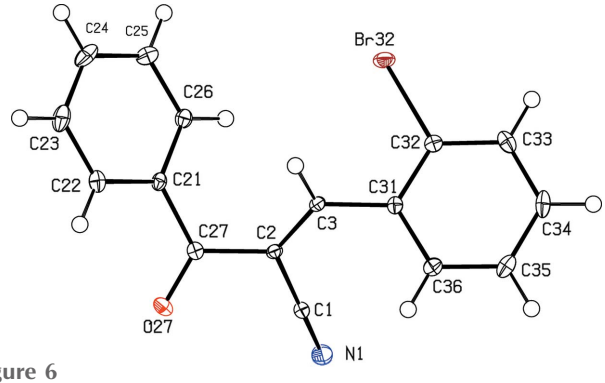


Figure 6
The molecular structure of compound (V), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

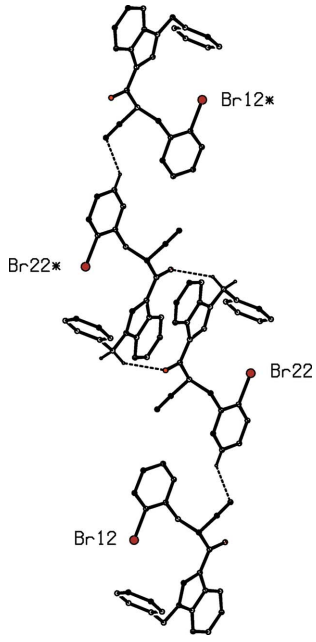


Figure 7
Part of the crystal structure of compound (II*c*), showing the formation of a centrosymmetric four-molecule aggregate. For the sake of clarity, the unit-cell outline and H atoms bonded to those C atoms not involved in the motif shown have all been omitted. The Br atoms marked with an asterisk (*) are at the symmetry position ($-x-1, -y+2, -z+2$).

angles between the planes of the indolyl units and the C atoms of the *N*-benzyl units are 82.52 (7) and 82.07 (7)°, respectively.

Atoms C1, C2, C3, C21 and N21 in the central spacer unit of compound (III) are coplanar, but substituent atoms O11 and C13 are displaced on either side of this plane by 0.283 (4) and 0.087 (6) Å, respectively, indicating a slight twist around the C1═C2 double bond. In contrast, in compound (IV), both of these atoms are displaced to the same side of the C1–C3/C21/N21 plane by 0.114 (2) and 0.038 (3) Å, respectively. The dihedral angles between the indolyl and bromobenzyl units in (III) and (IV) are 36.91 (9) and 79.27 (4)°, respectively.

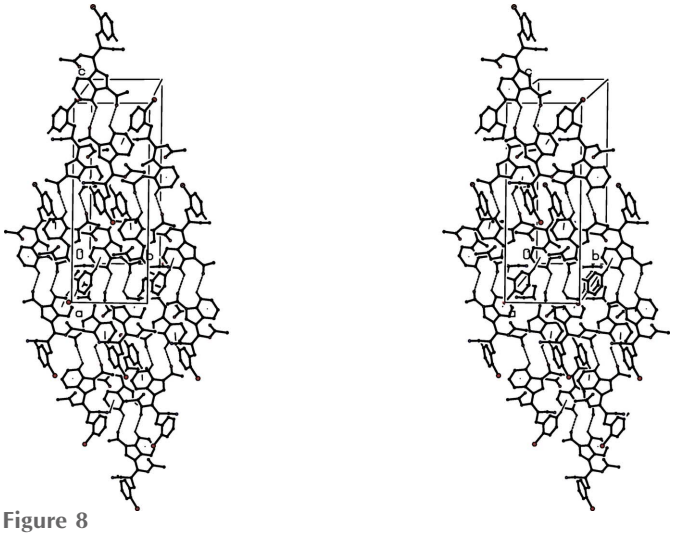


Figure 8
A stereoview of part of the crystal structure of compound (III), showing the formation of a hydrogen-bonded sheet lying parallel to ($10\bar{1}$). For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

As noted above, the structure of compound (V) has recently been reported at ambient temperature and refined in the space group $P\bar{1}$ with $Z' = 2$ (Dhiman *et al.*, 2016). Comparison of the atomic coordinates for corresponding pairs of atoms in this structure showed that the two molecules in the selected asymmetric unit are very precisely related by a noncrystallographic 2_1 screw axis along $(x, \frac{3}{4}, \frac{3}{4})$, and a detailed examination of this structure for additional symmetry elements (*PLATON*; Spek, 2009) found a 100% fit to space group $P2_1/n$ with $Z' = 1$, in a unit cell with dimensions $a = 10.896$ Å, $b = 8.952$ Å, $c = 13.292$ Å and $\beta = 96.15°$, very similar to the monoclinic unit cell found here at 100 K. Although a request to the authors of the original report for a copy of their .hkl file proved fruitless, we are nonetheless reasonably confident that compound (V) does not, in fact, exhibit temperature-dependent polymorphism, but that the ambient-temperature structure was refined in the wrong space group. The high values of the *R* factors and the goodness-of-fit parameter resulting from the ambient temperature refinement are probably, in large part, a consequence of the rather indifferent quality of the data set used, where $R_{\rm int} = 0.386$. At 100 K, the conformation of compound (V) resembles that of (I*b*), with dihedral angles between the central spacer unit on the one hand and the substituted and unsubstituted phenyl rings on the other of 48.63 (6) and 51.91 (7)°, respectively.

The supramolecular assembly in compounds (I*b*), (II*b*) and (II*c*) is very simple. In the structure of (I*b*), a single C—H···π(arene) hydrogen bond (Table 3) links molecules related by translation into simple chains running parallel to the [010] direction, while in (II*b*), the two molecules in the selected asymmetric unit are linked by a C—H···O hydrogen bond, but there are no direction-specific interactions between adjacent bimolecular units. The two independent molecules in compound (II*c*) are linked within the selected asymmetric unit by a C—H···N hydrogen bond and pairs of type 2 molecules are linked by paired C—H···O hydrogen bonds to form a cyclic centrosymmetric $R_2^2(14)$ ring, so forming a four-molecule aggregate in which the type 1 molecules are simply pendent from the central ring (Fig. 7).

In contrast to the rather simple assembly in compounds (I*b*), (II*b*) and (II*c*), that in compound (III) is more complex, and a combination of C—H···O and C—H···π(arene) hydrogen bonds links the molecules into sheets. Inversion related pairs of molecules are linked by symmetry-related C—H···O hydrogen bonds to form a cyclic centrosymmetric dimer, characterized by an $R_2^2(12)$ motif (Etter, 1990; Etter *et al.*, 1990; Bernstein *et al.*, 1995) and centred at $(\frac{1}{2}, \frac{1}{2}, 0)$. This dimeric unit is directly linked by the C—H···π hydrogen bond to four other dimers centred respectively at $(0, 0, -\frac{1}{2})$, $(0, 1, -\frac{1}{2})$, $(1, 0, \frac{1}{2})$ and $(1, 1, \frac{1}{2})$, so forming a sheet lying parallel to ($10\bar{1}$) (Fig. 8). Two sheets of this type, related to one another by the C-centring operation pass through each unit cell, but there are no direction-specific interactions between adjacent sheets. Two independent C—H···π(arene) hydrogen bonds link the molecules of compound (IV) into a chain of centro-

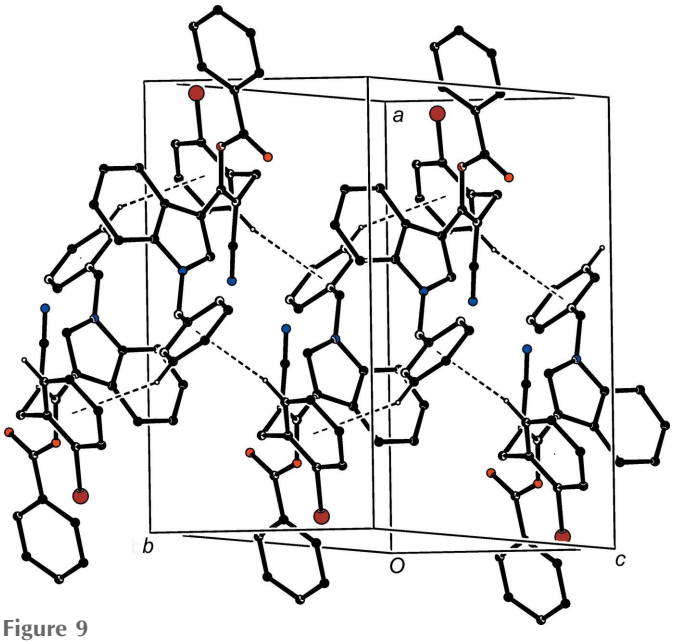


Figure 9
Part of the crystal structure of compound (IV), showing a hydrogen-bonded chain of rings running parallel to [010]. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

symmetric rings running parallel to the [010] direction, in which two types ring alternate, involving the *C*-benzyl and *N*-benzyl rings, respectively (Table 3 and Fig. 9).

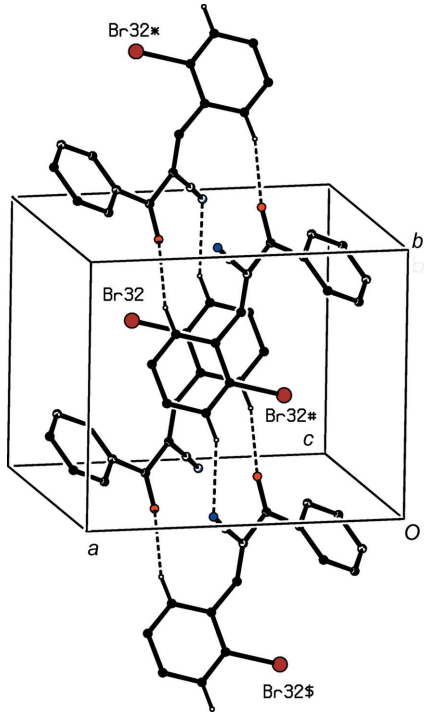


Figure 10
Part of the crystal structure of compound (V), showing the formation of a ribbon containing alternating $R_2^2(14)$ and $R_4^4(22)$ rings, running parallel to the [010] direction. Hydrogen bonds are shown as dashed lines and, for the sake of clarity, H atoms not involved in the motifs shown have been omitted. The Br atoms marked with an asterisk (*), a hash (#) or a dollar sign ($) are at the symmetry positions $(x, y+1, z)$, $(-x+1, -y+1, -z+1)$ and $(-x+1, -y, -z+1)$, respectively.

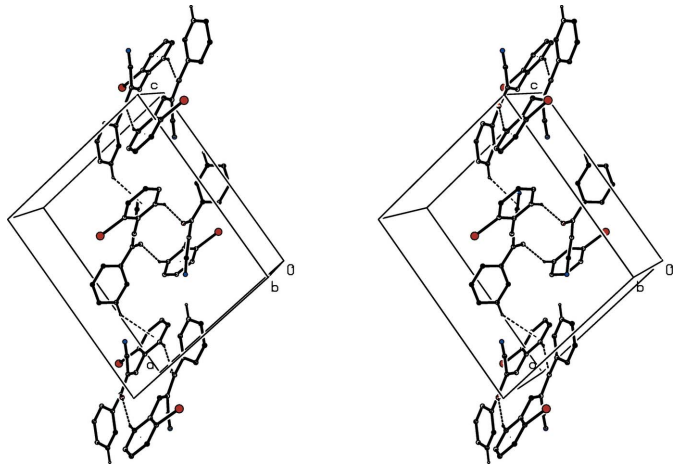


Figure 11
A stereoview of part of the crystal structure of compound (V), showing the formation of a hydrogen-bonded chain running parallel to the [10$\bar{1}$] direction. Hydrogen bonds are shown as dashed lines and, for the sake of clarity, H atoms not involved in the motifs shown have been omitted.

The supramolecular assembly of compound (V) was not mentioned in the original report (Dhiman *et al.*, 2016) and we therefore discuss it here. Four hydrogen bonds are present (Table 3) and these together link the molecules into very complex sheets, whose formation can, however, be readily analysed in terms of two simpler one-dimensional substructures (Ferguson *et al.*, 1998*a*,*b*; Gregson *et al.*, 2000). The C—H···N and C—H···O hydrogen bonds combine to form a ribbon running parallel to the [010] direction, in which $R_2^2(14)$ rings centred at $(\frac{1}{2}, n, \frac{1}{2})$ alternate with $R_4^4(22)$ rings centred at $(\frac{1}{2}, n+\frac{1}{2}, \frac{1}{2})$, where n represents an integer in each case (Fig. 10). There are also two C—H···π(arene) hydrogen bonds present in the structure, both involving the same aryl ring as the acceptor with the two donor group approaching opposite faces of the ring, such that the H3^{i}···*Cg*2···H23^{ii} angle is 174°

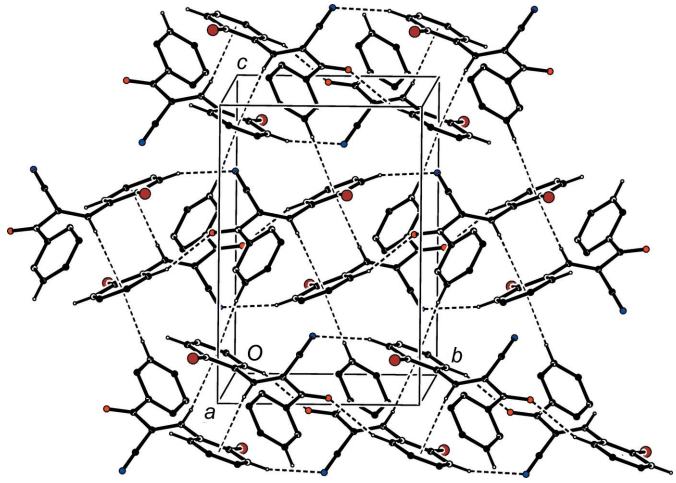


Figure 12
Part of the crystal structure of compound (V), showing the formation of a hydrogen-bonded sheet lying parallel to (101). Hydrogen bonds are shown as dashed lines and, for the sake of clarity, H atoms not involved in the motifs shown have been omitted.

[symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x-\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$]. One of these, that involving atom H3, lies within the ribbon generated by the C—H···N and C—H···O hydrogen bonds, while the second, involving atom H23, links $R_2^2(14)$ dimers generated by paired C—H···O hydrogen bonds into a chain running parallel to the $[10\bar{1}]$ direction (Fig. 11). The combination of the ribbon along [010] and the chain along $[10\bar{1}]$ generates a complex sheet structure lying parallel to (101) (Fig. 12).

Acknowledgements

The authors thank the Centro de Instrumentación Científico-Técnica of the Universidad de Jaén and the staff for the data collection.

Funding information

Funding for this research was provided by: COLCIENCIAS and Universidad del Valle, Consejería de Economía, Innovación, Ciencia y Empleo (Junta de Andalucía, Spain) and the Universidad de Jaén.

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supporting information

Acta Cryst. (2017). C**73**, 1040-1049 [https://doi.org/10.1107/S2053229617015789]

Design, synthesis and crystallographic study of novel indole-based cyano derivatives as key building blocks for heteropolycyclic compounds of major complexity

Andrés C. García, Rodrigo Abonía, Luz M. Jaramillo-Gómez, Justo Cobo and Christopher Glidewell

Computing details

For all structures, data collection: *APEX3* (Bruker, 2016); cell refinement: *SAINT* (Bruker, 2016); data reduction: *SAINT* (Bruker, 2016); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

(*E*)-3-(2-Bromophenyl)-2-(1-methyl-1*H*-indole-3-carbonyl)acrylonitrile (Ib)

Crystal data

$C_{19}H_{13}BrN_2O$
M_r = 365.22
Triclinic, $P\overline{1}$
a = 9.188 (5) Å
b = 9.308 (5) Å
c = 10.887 (5) Å
α = 113.56 (2)°
β = 90.782 (18)°
γ = 116.173 (16)°
V = 745.3 (7) Å^3
Z = 2
$F(000)$ = 368
D_x = 1.628 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3467 reflections
θ = 2.5–27.7°
μ = 2.76 mm^{-1}
T = 100 K
Block, yellow
0.33 × 0.21 × 0.19 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} = 0.446, T_{max} = 0.591
46632 measured reflections
3467 independent reflections
3296 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 27.7°, θ_{min} = 2.5°
h = −11→11
k = −12→12
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.049
S = 1.07
3467 reflections
209 parameters
0 restraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0202P)^2 + 0.6373P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ 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}
N1	0.29057 (17)	0.14946 (19)	0.81720 (15)	0.0189 (3)
C1	0.41876 (18)	0.2564 (2)	0.82078 (15)	0.0119 (3)
C2	0.57679 (18)	0.39150 (19)	0.82260 (14)	0.0100 (3)
C3	0.72260 (18)	0.40212 (19)	0.86046 (14)	0.0098 (3)
H3	0.8179	0.4924	0.8513	0.012*
C28	0.58375 (18)	0.52896 (19)	0.77826 (14)	0.0098 (3)
O28	0.71080 (13)	0.67527 (14)	0.82765 (11)	0.0150 (2)
N21	0.21687 (15)	0.33281 (16)	0.50736 (12)	0.0101 (2)
C22	0.31281 (17)	0.31821 (19)	0.59208 (14)	0.0100 (3)
H22	0.2916	0.2088	0.5923	0.012*
C23	0.44624 (17)	0.48493 (18)	0.67852 (14)	0.0087 (3)
C23A	0.43162 (17)	0.61089 (19)	0.64119 (14)	0.0090 (3)
C24	0.52727 (18)	0.79570 (19)	0.68562 (14)	0.0105 (3)
H24	0.6262	0.8660	0.7565	0.013*
C25	0.47404 (19)	0.87307 (19)	0.62369 (15)	0.0126 (3)
H25	0.5386	0.9982	0.6519	0.015*
C26	0.32693 (19)	0.7718 (2)	0.51994 (15)	0.0131 (3)
H26	0.2934	0.8302	0.4807	0.016*
C27	0.22994 (18)	0.5887 (2)	0.47382 (15)	0.0120 (3)
H27	0.1303	0.5194	0.4037	0.014*
C27A	0.28581 (17)	0.51137 (19)	0.53536 (14)	0.0094 (3)
C211	0.07277 (18)	0.1855 (2)	0.39592 (16)	0.0150 (3)
H21A	0.0497	0.0716	0.3949	0.022*
H21B	0.0963	0.1829	0.3077	0.022*
H21C	−0.0244	0.2029	0.4103	0.022*
C31	0.75642 (17)	0.29564 (18)	0.91350 (14)	0.0091 (3)
C32	0.88829 (17)	0.25981 (18)	0.88333 (14)	0.0092 (3)
Br32	1.00583 (2)	0.32293 (2)	0.75519 (2)	0.01222 (5)
C33	0.93663 (18)	0.17583 (19)	0.94167 (15)	0.0114 (3)
H33	1.0270	0.1542	0.9195	0.014*
C34	0.85086 (18)	0.1236 (2)	1.03316 (15)	0.0127 (3)
H34	0.8831	0.0669	1.0751	0.015*
C35	0.71789 (19)	0.1542 (2)	1.06365 (15)	0.0130 (3)
H35	0.6585	0.1167	1.1255	0.016*

C36	0.67111 (18)	0.2388 (2)	1.00457 (15)	0.0116 (3)
H36	0.5797	0.2586	1.0263	0.014*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0142 (6)	0.0248 (7)	0.0258 (7)	0.0094 (6)	0.0057 (5)	0.0187 (6)
C1	0.0131 (7)	0.0160 (7)	0.0128 (7)	0.0096 (6)	0.0042 (5)	0.0092 (6)
C2	0.0114 (7)	0.0103 (6)	0.0092 (6)	0.0055 (6)	0.0033 (5)	0.0050 (5)
C3	0.0109 (6)	0.0092 (6)	0.0086 (6)	0.0044 (5)	0.0021 (5)	0.0039 (5)
C28	0.0111 (6)	0.0108 (6)	0.0096 (6)	0.0068 (5)	0.0041 (5)	0.0048 (5)
O28	0.0135 (5)	0.0108 (5)	0.0181 (5)	0.0036 (4)	−0.0017 (4)	0.0067 (4)
N21	0.0087 (5)	0.0106 (6)	0.0105 (6)	0.0039 (5)	0.0019 (5)	0.0053 (5)
C22	0.0115 (6)	0.0121 (7)	0.0105 (6)	0.0073 (6)	0.0051 (5)	0.0070 (6)
C23	0.0092 (6)	0.0099 (6)	0.0097 (6)	0.0059 (5)	0.0045 (5)	0.0055 (5)
C23A	0.0097 (6)	0.0120 (7)	0.0086 (6)	0.0068 (5)	0.0046 (5)	0.0058 (5)
C24	0.0107 (6)	0.0105 (7)	0.0088 (6)	0.0050 (6)	0.0026 (5)	0.0031 (5)
C25	0.0154 (7)	0.0097 (6)	0.0125 (7)	0.0066 (6)	0.0050 (6)	0.0043 (6)
C26	0.0175 (7)	0.0170 (7)	0.0133 (7)	0.0125 (6)	0.0061 (6)	0.0098 (6)
C27	0.0116 (7)	0.0153 (7)	0.0108 (7)	0.0075 (6)	0.0025 (5)	0.0064 (6)
C27A	0.0105 (6)	0.0110 (6)	0.0093 (6)	0.0063 (5)	0.0051 (5)	0.0056 (5)
C211	0.0111 (7)	0.0123 (7)	0.0134 (7)	0.0015 (6)	−0.0020 (6)	0.0035 (6)
C31	0.0082 (6)	0.0079 (6)	0.0086 (6)	0.0027 (5)	−0.0002 (5)	0.0030 (5)
C32	0.0078 (6)	0.0090 (6)	0.0082 (6)	0.0019 (5)	0.0010 (5)	0.0039 (5)
Br32	0.01107 (7)	0.01656 (8)	0.01307 (8)	0.00725 (6)	0.00570 (5)	0.00963 (6)
C33	0.0092 (6)	0.0110 (7)	0.0129 (7)	0.0048 (5)	0.0012 (5)	0.0047 (6)
C34	0.0134 (7)	0.0117 (7)	0.0134 (7)	0.0050 (6)	0.0003 (5)	0.0074 (6)
C35	0.0137 (7)	0.0140 (7)	0.0123 (7)	0.0055 (6)	0.0038 (5)	0.0082 (6)
C36	0.0099 (6)	0.0140 (7)	0.0115 (7)	0.0062 (6)	0.0027 (5)	0.0059 (6)

Geometric parameters (Å, º)

N1—C1	1.146 (2)	C25—H25	0.9500
C1—C2	1.435 (2)	C26—C27	1.384 (2)
C2—C3	1.345 (2)	C26—H26	0.9500
C2—C28	1.516 (2)	C27—C27A	1.390 (2)
C3—C31	1.462 (2)	C27—H27	0.9500
C3—H3	0.9500	C211—H21A	0.9800
C28—O28	1.2253 (19)	C211—H21B	0.9800
C28—C23	1.447 (2)	C211—H21C	0.9800
N21—C22	1.3528 (19)	C31—C36	1.397 (2)
N21—C27A	1.385 (2)	C31—C32	1.402 (2)
N21—C211	1.459 (2)	C32—C33	1.381 (2)
C22—C23	1.385 (2)	C32—Br32	1.8959 (15)
C22—H22	0.9500	C33—C34	1.385 (2)
C23—C23A	1.441 (2)	C33—H33	0.9500
C23A—C24	1.398 (2)	C34—C35	1.388 (2)
C23A—C27A	1.411 (2)	C34—H34	0.9500

C24—C25	1.379 (2)	C35—C36	1.384 (2)
C24—H24	0.9500	C35—H35	0.9500
C25—C26	1.405 (2)	C36—H36	0.9500

N1—C1—C2	178.21 (16)	C26—C27—C27A	116.53 (14)
C3—C2—C1	123.11 (14)	C26—C27—H27	121.7
C3—C2—C28	117.10 (13)	C27A—C27—H27	121.7
C1—C2—C28	119.78 (13)	N21—C27A—C27	128.84 (14)
C2—C3—C31	129.71 (13)	N21—C27A—C23A	108.03 (12)
C2—C3—H3	115.1	C27—C27A—C23A	123.12 (14)
C31—C3—H3	115.1	N21—C211—H21A	109.5
O28—C28—C23	122.07 (13)	N21—C211—H21B	109.5
O28—C28—C2	117.42 (13)	H21A—C211—H21B	109.5
C23—C28—C2	120.49 (13)	N21—C211—H21C	109.5
C22—N21—C27A	108.76 (12)	H21A—C211—H21C	109.5
C22—N21—C211	126.51 (13)	H21B—C211—H21C	109.5
C27A—N21—C211	124.52 (12)	C36—C31—C32	116.99 (13)
N21—C22—C23	110.62 (13)	C36—C31—C3	122.58 (13)
N21—C22—H22	124.7	C32—C31—C3	120.20 (13)
C23—C22—H22	124.7	C33—C32—C31	122.72 (13)
C22—C23—C23A	105.98 (13)	C33—C32—Br32	117.14 (11)
C22—C23—C28	128.88 (13)	C31—C32—Br32	120.13 (11)
C23A—C23—C28	124.91 (13)	C32—C33—C34	118.77 (14)
C24—C23A—C27A	119.08 (13)	C32—C33—H33	120.6
C24—C23A—C23	134.34 (14)	C34—C33—H33	120.6
C27A—C23A—C23	106.58 (13)	C33—C34—C35	120.09 (13)
C25—C24—C23A	118.17 (14)	C33—C34—H34	120.0
C25—C24—H24	120.9	C35—C34—H34	120.0
C23A—C24—H24	120.9	C36—C35—C34	120.46 (14)
C24—C25—C26	121.77 (14)	C36—C35—H35	119.8
C24—C25—H25	119.1	C34—C35—H35	119.8
C26—C25—H25	119.1	C35—C36—C31	120.95 (14)
C27—C26—C25	121.31 (14)	C35—C36—H36	119.5
C27—C26—H26	119.3	C31—C36—H36	119.5
C25—C26—H26	119.3		

C1—C2—C3—C31	−4.1 (2)	C22—N21—C27A—C27	179.90 (14)
C28—C2—C3—C31	176.84 (13)	C211—N21—C27A—C27	4.9 (2)
C3—C2—C28—O28	−28.16 (19)	C22—N21—C27A—C23A	0.57 (15)
C1—C2—C28—O28	152.77 (14)	C211—N21—C27A—C23A	−174.47 (13)
C3—C2—C28—C23	150.23 (14)	C26—C27—C27A—N21	−178.17 (14)
C1—C2—C28—C23	−28.8 (2)	C26—C27—C27A—C23A	1.1 (2)
C27A—N21—C22—C23	0.38 (16)	C24—C23A—C27A—N21	178.21 (12)
C211—N21—C22—C23	175.30 (13)	C23—C23A—C27A—N21	−1.26 (15)
N21—C22—C23—C23A	−1.15 (16)	C24—C23A—C27A—C27	−1.2 (2)
N21—C22—C23—C28	−175.81 (13)	C23—C23A—C27A—C27	179.37 (13)
O28—C28—C23—C22	164.56 (14)	C2—C3—C31—C36	−38.0 (2)
C2—C28—C23—C22	−13.7 (2)	C2—C3—C31—C32	147.66 (16)

O28—C28—C23—C23A	−9.2 (2)	C36—C31—C32—C33	−1.3 (2)
C2—C28—C23—C23A	172.51 (13)	C3—C31—C32—C33	173.30 (13)
C22—C23—C23A—C24	−177.89 (15)	C36—C31—C32—Br32	177.45 (10)
C28—C23—C23A—C24	−3.0 (2)	C3—C31—C32—Br32	−7.92 (18)
C22—C23—C23A—C27A	1.45 (15)	C31—C32—C33—C34	0.4 (2)
C28—C23—C23A—C27A	176.39 (13)	Br32—C32—C33—C34	−178.40 (11)
C27A—C23A—C24—C25	0.2 (2)	C32—C33—C34—C35	0.7 (2)
C23—C23A—C24—C25	179.45 (15)	C33—C34—C35—C36	−0.8 (2)
C23A—C24—C25—C26	0.9 (2)	C34—C35—C36—C31	−0.1 (2)
C24—C25—C26—C27	−1.0 (2)	C32—C31—C36—C35	1.2 (2)
C25—C26—C27—C27A	0.0 (2)	C3—C31—C36—C35	−173.32 (14)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C24—H24···*Cg*1^{i}	0.95	2.77	3.544 (3)	139

Symmetry code: (i) *x*, *y*+1, *z*.

(2*RS*)-2-(2-Bromobenzyl)-3-(1-methyl-1*H*-indol-3-yl)-\ 3-oxopropanenitrile (IIb)

Crystal data

$C_{19}H_{15}BrN_2O$
M_r = 367.24
Monoclinic, $P2_1/n$
a = 13.728 (6) Å
b = 14.901 (6) Å
c = 15.507 (6) Å
β = 92.165 (19)°
V = 3170 (2) Å^3
Z = 8
$F(000)$ = 1488
D_x = 1.539 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 7297 reflections
θ = 2.4–27.6°
μ = 2.60 mm^{-1}
T = 100 K
Plate, yellow-brown
0.40 × 0.30 × 0.10 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} = 0.489, T_{max} = 0.771
79963 measured reflections
7297 independent reflections
5456 reflections with $I > 2\sigma(I)$
R_{int} = 0.090
θ_{max} = 27.6°, θ_{min} = 2.4°
h = −17→17
k = −18→19
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.072
S = 1.05
7297 reflections
417 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0217P)^2 + 3.7392P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.76 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}
N11	0.18232 (18)	0.42519 (15)	0.29826 (14)	0.0248 (5)
C11	0.2371 (2)	0.47754 (16)	0.27615 (16)	0.0168 (5)
C12	0.30553 (18)	0.54607 (16)	0.24554 (15)	0.0133 (5)
H12	0.2725	0.6059	0.2438	0.016*
C13	0.33425 (18)	0.51972 (15)	0.15332 (15)	0.0133 (5)
O13	0.34791 (14)	0.44049 (11)	0.13777 (11)	0.0198 (4)
C127	0.39870 (19)	0.55156 (16)	0.30549 (15)	0.0146 (5)
H12A	0.4337	0.4936	0.3035	0.018*
H12B	0.4423	0.5985	0.2834	0.018*
C121	0.37709 (18)	0.57268 (16)	0.39815 (16)	0.0134 (5)
C122	0.35456 (18)	0.65859 (16)	0.42667 (16)	0.0131 (5)
Br12	0.34405 (2)	0.75572 (2)	0.34672 (2)	0.01692 (7)
C123	0.33681 (19)	0.67677 (17)	0.51225 (16)	0.0153 (5)
H123	0.3223	0.7361	0.5300	0.018*
C124	0.34047 (19)	0.60709 (17)	0.57167 (16)	0.0186 (6)
H124	0.3284	0.6188	0.6305	0.022*
C125	0.3616 (2)	0.52069 (17)	0.54579 (17)	0.0194 (6)
H125	0.3636	0.4730	0.5865	0.023*
C126	0.37979 (19)	0.50432 (17)	0.45965 (16)	0.0181 (5)
H126	0.3945	0.4449	0.4422	0.022*
N131	0.33943 (15)	0.72482 (13)	0.02457 (13)	0.0130 (4)
C132	0.32742 (18)	0.68181 (16)	0.10035 (15)	0.0129 (5)
H132	0.3100	0.7100	0.1525	0.016*
C133	0.34426 (18)	0.59041 (16)	0.09058 (15)	0.0132 (5)
C13A	0.36750 (18)	0.57742 (16)	0.00110 (15)	0.0126 (5)
C134	0.38778 (18)	0.50255 (17)	−0.04959 (15)	0.0156 (5)
H134	0.3906	0.4442	−0.0250	0.019*
C135	0.40367 (19)	0.51530 (18)	−0.13629 (17)	0.0196 (6)
H135	0.4165	0.4649	−0.1716	0.024*
C136	0.40119 (19)	0.60133 (18)	−0.17306 (16)	0.0196 (6)
H136	0.4134	0.6081	−0.2326	0.023*
C137	0.38127 (19)	0.67663 (17)	−0.12446 (16)	0.0163 (5)
H137	0.3798	0.7350	−0.1492	0.020*
C17A	0.36354 (18)	0.66288 (16)	−0.03762 (15)	0.0132 (5)
C131	0.3262 (2)	0.82032 (16)	0.00842 (17)	0.0190 (6)
H13A	0.3095	0.8504	0.0621	0.029*
H13B	0.2734	0.8291	−0.0351	0.029*
H13C	0.3867	0.8457	−0.0126	0.029*
N21	0.22989 (19)	0.33176 (16)	0.79585 (15)	0.0273 (6)

C21	0.2823 (2)	0.27789 (16)	0.77344 (16)	0.0164 (5)
C22	0.34802 (18)	0.20737 (16)	0.74254 (15)	0.0126 (5)
H22	0.3133	0.1484	0.7423	0.015*
C23	0.37571 (18)	0.23082 (15)	0.64954 (15)	0.0137 (5)
O23	0.39429 (14)	0.30937 (11)	0.63232 (11)	0.0198 (4)
C227	0.44284 (18)	0.20000 (16)	0.79972 (15)	0.0140 (5)
H22A	0.4848	0.1527	0.7760	0.017*
H22B	0.4787	0.2575	0.7973	0.017*
C221	0.42413 (18)	0.17826 (16)	0.89260 (15)	0.0130 (5)
C222	0.40507 (18)	0.09155 (16)	0.92183 (15)	0.0138 (5)
Br22	0.39344 (2)	−0.00513 (2)	0.84159 (2)	0.01913 (7)
C223	0.39041 (19)	0.07332 (17)	1.00809 (16)	0.0166 (5)
H223	0.3797	0.0135	1.0266	0.020*
C224	0.39156 (19)	0.14326 (17)	1.06685 (16)	0.0177 (5)
H224	0.3811	0.1313	1.1260	0.021*
C225	0.40795 (19)	0.23065 (17)	1.04010 (16)	0.0177 (5)
H225	0.4074	0.2787	1.0804	0.021*
C226	0.42517 (18)	0.24722 (16)	0.95389 (16)	0.0159 (5)
H226	0.4380	0.3069	0.9360	0.019*
N231	0.36915 (16)	0.02132 (13)	0.52880 (13)	0.0140 (4)
C232	0.35817 (18)	0.06868 (16)	0.60173 (15)	0.0136 (5)
H232	0.3381	0.0442	0.6548	0.016*
C233	0.38029 (18)	0.15829 (15)	0.58849 (15)	0.0126 (5)
C23A	0.40570 (18)	0.16573 (16)	0.49902 (15)	0.0133 (5)
C234	0.43411 (19)	0.23648 (17)	0.44611 (16)	0.0180 (5)
H234	0.4402	0.2958	0.4682	0.022*
C235	0.4530 (2)	0.21792 (19)	0.36101 (17)	0.0231 (6)
H235	0.4708	0.2655	0.3240	0.028*
C236	0.4466 (2)	0.13043 (19)	0.32817 (17)	0.0237 (6)
H236	0.4615	0.1198	0.2697	0.028*
C237	0.41896 (19)	0.05918 (18)	0.37893 (16)	0.0190 (6)
H237	0.4144	−0.0002	0.3568	0.023*
C27A	0.39814 (18)	0.07874 (16)	0.46411 (15)	0.0143 (5)
C231	0.3488 (2)	−0.07415 (16)	0.51695 (17)	0.0177 (5)
H23A	0.3322	−0.1007	0.5723	0.027*
H23B	0.4065	−0.1041	0.4953	0.027*
H23C	0.2939	−0.0817	0.4753	0.027*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N11	0.0269 (14)	0.0297 (12)	0.0177 (12)	−0.0120 (11)	−0.0031 (10)	0.0050 (10)
C11	0.0179 (15)	0.0211 (13)	0.0111 (12)	−0.0018 (11)	−0.0050 (10)	0.0003 (10)
C12	0.0141 (14)	0.0124 (11)	0.0133 (12)	−0.0004 (9)	−0.0005 (10)	0.0006 (9)
C13	0.0116 (13)	0.0140 (12)	0.0142 (12)	−0.0020 (9)	−0.0007 (10)	−0.0007 (9)
O13	0.0268 (11)	0.0137 (9)	0.0189 (9)	−0.0008 (8)	0.0017 (8)	−0.0017 (7)
C127	0.0138 (14)	0.0134 (11)	0.0166 (12)	0.0003 (10)	−0.0009 (10)	−0.0030 (9)
C121	0.0084 (13)	0.0160 (12)	0.0157 (12)	−0.0027 (10)	−0.0020 (10)	−0.0021 (9)

C122	0.0092 (13)	0.0160 (12)	0.0139 (12)	−0.0031 (9)	−0.0021 (10)	0.0011 (9)
Br12	0.02524 (15)	0.01184 (11)	0.01379 (12)	−0.00209 (10)	0.00211 (10)	−0.00004 (9)
C123	0.0123 (14)	0.0182 (12)	0.0152 (13)	−0.0036 (10)	−0.0026 (10)	−0.0023 (10)
C124	0.0149 (14)	0.0287 (14)	0.0119 (12)	−0.0048 (11)	−0.0021 (10)	0.0005 (10)
C125	0.0158 (14)	0.0234 (13)	0.0186 (13)	−0.0012 (11)	−0.0044 (11)	0.0083 (10)
C126	0.0164 (14)	0.0149 (12)	0.0225 (13)	0.0012 (10)	−0.0049 (10)	0.0013 (10)
N131	0.0130 (11)	0.0134 (10)	0.0125 (10)	−0.0015 (8)	−0.0028 (8)	−0.0008 (8)
C132	0.0106 (13)	0.0164 (12)	0.0116 (12)	−0.0013 (9)	−0.0032 (10)	−0.0013 (9)
C133	0.0116 (13)	0.0150 (12)	0.0127 (12)	−0.0009 (9)	−0.0010 (10)	−0.0001 (9)
C13A	0.0085 (13)	0.0163 (12)	0.0128 (12)	−0.0021 (9)	−0.0022 (10)	−0.0002 (9)
C134	0.0123 (13)	0.0176 (12)	0.0170 (12)	0.0009 (10)	0.0004 (10)	−0.0021 (10)
C135	0.0133 (14)	0.0265 (14)	0.0191 (13)	0.0007 (11)	0.0019 (11)	−0.0081 (11)
C136	0.0143 (14)	0.0343 (15)	0.0099 (12)	−0.0018 (11)	−0.0011 (10)	−0.0010 (10)
C137	0.0116 (14)	0.0231 (13)	0.0138 (12)	−0.0025 (10)	−0.0032 (10)	0.0032 (10)
C17A	0.0074 (13)	0.0164 (12)	0.0155 (12)	−0.0020 (9)	−0.0042 (10)	−0.0014 (9)
C131	0.0241 (16)	0.0135 (12)	0.0191 (13)	0.0009 (11)	−0.0026 (11)	0.0026 (10)
N21	0.0259 (15)	0.0317 (13)	0.0243 (13)	0.0088 (11)	0.0008 (11)	−0.0055 (10)
C21	0.0172 (14)	0.0205 (12)	0.0111 (12)	0.0001 (11)	−0.0023 (10)	−0.0016 (10)
C22	0.0133 (13)	0.0122 (11)	0.0122 (12)	0.0009 (9)	0.0000 (10)	−0.0005 (9)
C23	0.0118 (13)	0.0157 (12)	0.0133 (12)	0.0026 (10)	−0.0029 (10)	0.0006 (9)
O23	0.0284 (12)	0.0114 (8)	0.0197 (10)	0.0007 (7)	0.0013 (8)	0.0009 (7)
C227	0.0127 (14)	0.0147 (11)	0.0145 (12)	0.0006 (10)	−0.0017 (10)	0.0005 (9)
C221	0.0081 (13)	0.0168 (12)	0.0140 (12)	0.0021 (9)	−0.0009 (10)	0.0008 (9)
C222	0.0126 (13)	0.0155 (12)	0.0130 (12)	0.0032 (10)	−0.0037 (10)	−0.0019 (9)
Br22	0.03058 (16)	0.01197 (12)	0.01466 (12)	0.00296 (11)	−0.00141 (10)	−0.00100 (9)
C223	0.0155 (14)	0.0176 (12)	0.0165 (13)	0.0019 (10)	−0.0025 (10)	0.0039 (10)
C224	0.0140 (14)	0.0280 (13)	0.0110 (12)	0.0004 (11)	−0.0033 (10)	0.0019 (10)
C225	0.0135 (14)	0.0226 (13)	0.0168 (13)	0.0003 (10)	−0.0040 (10)	−0.0066 (10)
C226	0.0130 (13)	0.0155 (11)	0.0189 (12)	−0.0002 (10)	−0.0028 (10)	0.0002 (10)
N231	0.0160 (12)	0.0124 (10)	0.0135 (10)	0.0012 (8)	−0.0028 (9)	−0.0027 (8)
C232	0.0138 (14)	0.0157 (11)	0.0111 (12)	0.0050 (10)	−0.0028 (10)	0.0005 (9)
C233	0.0094 (13)	0.0158 (11)	0.0124 (12)	0.0033 (9)	−0.0028 (10)	0.0009 (9)
C23A	0.0091 (13)	0.0183 (12)	0.0123 (12)	0.0039 (10)	−0.0022 (10)	−0.0002 (9)
C234	0.0157 (14)	0.0196 (13)	0.0188 (13)	0.0020 (10)	0.0009 (10)	0.0042 (10)
C235	0.0196 (15)	0.0330 (15)	0.0169 (13)	0.0001 (12)	0.0021 (11)	0.0058 (11)
C236	0.0188 (16)	0.0404 (16)	0.0117 (13)	0.0026 (12)	−0.0005 (11)	−0.0025 (11)
C237	0.0122 (14)	0.0295 (14)	0.0149 (13)	0.0022 (11)	−0.0044 (11)	−0.0086 (11)
C27A	0.0094 (13)	0.0200 (12)	0.0132 (12)	0.0025 (10)	−0.0044 (10)	−0.0007 (9)
C231	0.0190 (15)	0.0135 (12)	0.0205 (13)	−0.0011 (10)	−0.0032 (11)	−0.0034 (10)

Geometric parameters (Å, º)

N11—C11	1.145 (3)	N21—C21	1.141 (3)
C11—C12	1.478 (3)	C21—C22	1.476 (3)
C12—C13	1.548 (3)	C22—C23	1.545 (3)
C12—C127	1.555 (3)	C22—C227	1.551 (3)
C12—H12	1.0000	C22—H22	1.0000
C13—O13	1.221 (3)	C23—O23	1.229 (3)

C13—C133	1.444 (3)	C23—C233	1.440 (3)
C127—C121	1.511 (3)	C227—C221	1.508 (3)
C127—H12A	0.9900	C227—H22A	0.9900
C127—H12B	0.9900	C227—H22B	0.9900
C121—C122	1.393 (3)	C221—C222	1.397 (3)
C121—C126	1.395 (3)	C221—C226	1.399 (3)
C122—C123	1.385 (3)	C222—C223	1.387 (3)
C122—Br12	1.908 (2)	C222—Br22	1.906 (2)
C123—C124	1.388 (3)	C223—C224	1.384 (4)
C123—H123	0.9500	C223—H223	0.9500
C124—C125	1.383 (4)	C224—C225	1.388 (4)
C124—H124	0.9500	C224—H224	0.9500
C125—C126	1.390 (4)	C225—C226	1.389 (3)
C125—H125	0.9500	C225—H225	0.9500
C126—H126	0.9500	C226—H226	0.9500
N131—C132	1.354 (3)	N231—C232	1.346 (3)
N131—C17A	1.384 (3)	N231—C27A	1.388 (3)
N131—C131	1.455 (3)	N231—C231	1.460 (3)
C132—C133	1.391 (3)	C232—C233	1.386 (3)
C132—H132	0.9500	C232—H232	0.9500
C133—C13A	1.449 (3)	C233—C23A	1.448 (3)
C13A—C134	1.399 (3)	C23A—C234	1.400 (3)
C13A—C17A	1.408 (3)	C23A—C27A	1.407 (3)
C134—C135	1.383 (4)	C234—C235	1.382 (4)
C134—H134	0.9500	C234—H234	0.9500
C135—C136	1.403 (4)	C235—C236	1.401 (4)
C135—H135	0.9500	C235—H235	0.9500
C136—C137	1.385 (4)	C236—C237	1.383 (4)
C136—H136	0.9500	C236—H236	0.9500
C137—C17A	1.393 (3)	C237—C27A	1.393 (3)
C137—H137	0.9500	C237—H237	0.9500
C131—H13A	0.9800	C231—H23A	0.9800
C131—H13B	0.9800	C231—H23B	0.9800
C131—H13C	0.9800	C231—H23C	0.9800
N11—C11—C12	178.2 (3)	N21—C21—C22	178.4 (3)
C11—C12—C13	107.97 (19)	C21—C22—C23	108.47 (19)
C11—C12—C127	111.3 (2)	C21—C22—C227	112.0 (2)
C13—C12—C127	109.5 (2)	C23—C22—C227	108.6 (2)
C11—C12—H12	109.4	C21—C22—H22	109.2
C13—C12—H12	109.4	C23—C22—H22	109.2
C127—C12—H12	109.4	C227—C22—H22	109.2
O13—C13—C133	123.6 (2)	O23—C23—C233	123.9 (2)
O13—C13—C12	118.3 (2)	O23—C23—C22	118.6 (2)
C133—C13—C12	118.1 (2)	C233—C23—C22	117.5 (2)
C121—C127—C12	113.1 (2)	C221—C227—C22	113.1 (2)
C121—C127—H12A	109.0	C221—C227—H22A	109.0
C12—C127—H12A	109.0	C22—C227—H22A	109.0

C121—C127—H12B	109.0	C221—C227—H22B	109.0
C12—C127—H12B	109.0	C22—C227—H22B	109.0
H12A—C127—H12B	107.8	H22A—C227—H22B	107.8
C122—C121—C126	117.0 (2)	C222—C221—C226	117.1 (2)
C122—C121—C127	123.2 (2)	C222—C221—C227	123.3 (2)
C126—C121—C127	119.8 (2)	C226—C221—C227	119.6 (2)
C123—C122—C121	122.2 (2)	C223—C222—C221	122.0 (2)
C123—C122—Br12	117.56 (18)	C223—C222—Br22	118.02 (18)
C121—C122—Br12	120.21 (18)	C221—C222—Br22	119.95 (18)
C122—C123—C124	119.1 (2)	C224—C223—C222	119.3 (2)
C122—C123—H123	120.4	C224—C223—H223	120.4
C124—C123—H123	120.4	C222—C223—H223	120.4
C125—C124—C123	120.4 (2)	C223—C224—C225	120.5 (2)
C125—C124—H124	119.8	C223—C224—H224	119.7
C123—C124—H124	119.8	C225—C224—H224	119.7
C124—C125—C126	119.3 (2)	C224—C225—C226	119.4 (2)
C124—C125—H125	120.3	C224—C225—H225	120.3
C126—C125—H125	120.3	C226—C225—H225	120.3
C125—C126—C121	121.9 (2)	C225—C226—C221	121.7 (2)
C125—C126—H126	119.1	C225—C226—H226	119.1
C121—C126—H126	119.1	C221—C226—H226	119.1
C132—N131—C17A	109.22 (19)	C232—N231—C27A	109.2 (2)
C132—N131—C131	126.4 (2)	C232—N231—C231	126.1 (2)
C17A—N131—C131	124.3 (2)	C27A—N231—C231	124.6 (2)
N131—C132—C133	110.0 (2)	N231—C232—C233	110.4 (2)
N131—C132—H132	125.0	N231—C232—H232	124.8
C133—C132—H132	125.0	C233—C232—H232	124.8
C132—C133—C13	128.4 (2)	C232—C233—C23	127.6 (2)
C132—C133—C13A	106.2 (2)	C232—C233—C23A	106.1 (2)
C13—C133—C13A	125.3 (2)	C23—C233—C23A	126.2 (2)
C134—C13A—C17A	119.1 (2)	C234—C23A—C27A	119.1 (2)
C134—C13A—C133	134.5 (2)	C234—C23A—C233	134.6 (2)
C17A—C13A—C133	106.3 (2)	C27A—C23A—C233	106.3 (2)
C135—C134—C13A	118.6 (2)	C235—C234—C23A	118.4 (2)
C135—C134—H134	120.7	C235—C234—H234	120.8
C13A—C134—H134	120.7	C23A—C234—H234	120.8
C134—C135—C136	121.2 (2)	C234—C235—C236	121.4 (3)
C134—C135—H135	119.4	C234—C235—H235	119.3
C136—C135—H135	119.4	C236—C235—H235	119.3
C137—C136—C135	121.4 (2)	C237—C236—C235	121.5 (2)
C137—C136—H136	119.3	C237—C236—H236	119.3
C135—C136—H136	119.3	C235—C236—H236	119.3
C136—C137—C17A	116.9 (2)	C236—C237—C27A	116.7 (2)
C136—C137—H137	121.5	C236—C237—H237	121.6
C17A—C137—H137	121.5	C27A—C237—H237	121.6
N131—C17A—C137	129.1 (2)	N231—C27A—C237	129.1 (2)
N131—C17A—C13A	108.2 (2)	N231—C27A—C23A	108.0 (2)
C137—C17A—C13A	122.7 (2)	C237—C27A—C23A	122.9 (2)

N131—C131—H13A	109.5	N231—C231—H23A	109.5
N131—C131—H13B	109.5	N231—C231—H23B	109.5
H13A—C131—H13B	109.5	H23A—C231—H23B	109.5
N131—C131—H13C	109.5	N231—C231—H23C	109.5
H13A—C131—H13C	109.5	H23A—C231—H23C	109.5
H13B—C131—H13C	109.5	H23B—C231—H23C	109.5
C11—C12—C13—O13	−39.4 (3)	C21—C22—C23—O23	41.5 (3)
C127—C12—C13—O13	81.8 (3)	C227—C22—C23—O23	−80.5 (3)
C11—C12—C13—C133	141.1 (2)	C21—C22—C23—C233	−139.9 (2)
C127—C12—C13—C133	−97.6 (3)	C227—C22—C23—C233	98.1 (3)
C11—C12—C127—C121	−58.0 (3)	C21—C22—C227—C221	59.6 (3)
C13—C12—C127—C121	−177.27 (19)	C23—C22—C227—C221	179.33 (19)
C12—C127—C121—C122	−77.7 (3)	C22—C227—C221—C222	80.5 (3)
C12—C127—C121—C126	102.7 (3)	C22—C227—C221—C226	−99.3 (3)
C126—C121—C122—C123	0.9 (4)	C226—C221—C222—C223	−1.8 (4)
C127—C121—C122—C123	−178.7 (2)	C227—C221—C222—C223	178.4 (2)
C126—C121—C122—Br12	−177.55 (19)	C226—C221—C222—Br22	175.73 (18)
C127—C121—C122—Br12	2.8 (3)	C227—C221—C222—Br22	−4.1 (3)
C121—C122—C123—C124	−0.7 (4)	C221—C222—C223—C224	2.1 (4)
Br12—C122—C123—C124	177.78 (19)	Br22—C222—C223—C224	−175.5 (2)
C122—C123—C124—C125	0.0 (4)	C222—C223—C224—C225	−0.5 (4)
C123—C124—C125—C126	0.4 (4)	C223—C224—C225—C226	−1.3 (4)
C124—C125—C126—C121	−0.2 (4)	C224—C225—C226—C221	1.6 (4)
C122—C121—C126—C125	−0.4 (4)	C222—C221—C226—C225	−0.1 (4)
C127—C121—C126—C125	179.2 (2)	C227—C221—C226—C225	179.7 (2)
C17A—N131—C132—C133	0.6 (3)	C27A—N231—C232—C233	−0.6 (3)
C131—N131—C132—C133	178.6 (2)	C231—N231—C232—C233	−177.4 (2)
N131—C132—C133—C13	−176.3 (2)	N231—C232—C233—C23	178.6 (2)
N131—C132—C133—C13A	−0.5 (3)	N231—C232—C233—C23A	0.8 (3)
O13—C13—C133—C132	177.6 (3)	O23—C23—C233—C232	−178.1 (3)
C12—C13—C133—C132	−2.9 (4)	C22—C23—C233—C232	3.3 (4)
O13—C13—C133—C13A	2.6 (4)	O23—C23—C233—C23A	−0.7 (4)
C12—C13—C133—C13A	−177.9 (2)	C22—C23—C233—C23A	−179.3 (2)
C132—C133—C13A—C134	−177.3 (3)	C232—C233—C23A—C234	179.7 (3)
C13—C133—C13A—C134	−1.4 (5)	C23—C233—C23A—C234	1.9 (5)
C132—C133—C13A—C17A	0.3 (3)	C232—C233—C23A—C27A	−0.6 (3)
C13—C133—C13A—C17A	176.2 (2)	C23—C233—C23A—C27A	−178.5 (2)
C17A—C13A—C134—C135	0.3 (4)	C27A—C23A—C234—C235	0.5 (4)
C133—C13A—C134—C135	177.5 (3)	C233—C23A—C234—C235	−180.0 (3)
C13A—C134—C135—C136	1.0 (4)	C23A—C234—C235—C236	−1.6 (4)
C134—C135—C136—C137	−1.0 (4)	C234—C235—C236—C237	1.4 (4)
C135—C136—C137—C17A	−0.2 (4)	C235—C236—C237—C27A	−0.1 (4)
C132—N131—C17A—C137	179.1 (3)	C232—N231—C27A—C237	179.3 (3)
C131—N131—C17A—C137	1.1 (4)	C231—N231—C27A—C237	−4.0 (4)
C132—N131—C17A—C13A	−0.4 (3)	C232—N231—C27A—C23A	0.2 (3)
C131—N131—C17A—C13A	−178.5 (2)	C231—N231—C27A—C23A	177.0 (2)
C136—C137—C17A—N131	−178.0 (2)	C236—C237—C27A—N231	−180.0 (3)

C136—C137—C17A—C13A	1.5 (4)	C236—C237—C27A—C23A	−1.0 (4)
C134—C13A—C17A—N131	178.1 (2)	C234—C23A—C27A—N231	180.0 (2)
C133—C13A—C17A—N131	0.1 (3)	C233—C23A—C27A—N231	0.3 (3)
C134—C13A—C17A—C137	−1.5 (4)	C234—C23A—C27A—C237	0.9 (4)
C133—C13A—C17A—C137	−179.5 (2)	C233—C23A—C27A—C237	−178.8 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C125—H125···O23	0.95	2.57	3.446 (3)	153

(2*RS*)-3-(1-benzyl-1*H*-indol-3-yl)-\ 2-(2-bromobenzyl)-3-oxopropanenitrile (IIc)

Crystal data

$C_{25}H_{19}BrN_2O$
$M_r = 443.32$
Triclinic, $P\overline{1}$
$a = 10.912$ (4) Å
$b = 12.544$ (8) Å
$c = 15.583$ (7) Å
$\alpha = 89.23$ (4)°
$\beta = 79.49$ (3)°
$\gamma = 73.20$ (3)°
$V = 2006.0$ (18) Å^3
$Z = 4$
$F(000) = 904$
$D_x = 1.468$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9244 reflections
$\theta = 2.2$–$27.6°$
$\mu = 2.07$ mm^{-1}
$T = 100$ K
Plate, colourless
0.22 × 0.21 × 0.10 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)
$T_{min} = 0.737$, $T_{max} = 0.813$
96931 measured reflections
9244 independent reflections
6872 reflections with $I > 2\sigma(I)$
$R_{int} = 0.112$
$\theta_{max} = 27.6°$, $\theta_{min} = 2.2°$
$h = -14 \rightarrow 14$
$k = -16 \rightarrow 16$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.084$
$S = 1.02$
9244 reflections
523 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.025P)^2 + 2.2423P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.57$ 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}
N11	0.5097 (2)	0.73044 (17)	0.58025 (14)	0.0195 (5)
C11	0.5794 (2)	0.64664 (19)	0.59130 (15)	0.0131 (5)
C12	0.6656 (2)	0.53736 (18)	0.60712 (15)	0.0120 (5)
H12	0.7183	0.5464	0.6512	0.014*
C13	0.7577 (2)	0.48592 (19)	0.52103 (15)	0.0135 (5)
O13	0.71866 (16)	0.50668 (14)	0.45193 (11)	0.0195 (4)
C127	0.5832 (2)	0.45808 (19)	0.64171 (15)	0.0143 (5)
H12A	0.5311	0.4499	0.5978	0.017*
H12B	0.6427	0.3836	0.6488	0.017*
C121	0.4926 (2)	0.49869 (19)	0.72782 (15)	0.0142 (5)
C122	0.5251 (2)	0.4672 (2)	0.80876 (16)	0.0183 (5)
Br12	0.69435 (3)	0.37370 (2)	0.81581 (2)	0.02901 (8)
C123	0.4382 (3)	0.5025 (2)	0.88675 (17)	0.0260 (6)
H123	0.4620	0.4766	0.9407	0.031*
C124	0.3165 (3)	0.5757 (2)	0.88513 (18)	0.0265 (6)
H124	0.2569	0.6020	0.9382	0.032*
C125	0.2820 (2)	0.6105 (2)	0.80614 (17)	0.0222 (6)
H125	0.1989	0.6614	0.8049	0.027*
C126	0.3682 (2)	0.5712 (2)	0.72888 (16)	0.0157 (5)
H126	0.3421	0.5943	0.6750	0.019*
N131	1.05957 (19)	0.31755 (16)	0.58068 (13)	0.0148 (4)
C132	0.9391 (2)	0.39054 (19)	0.60079 (15)	0.0154 (5)
H132	0.8986	0.4230	0.6574	0.019*
C133	0.8832 (2)	0.41133 (19)	0.52668 (15)	0.0133 (5)
C13A	0.9782 (2)	0.34516 (19)	0.45581 (15)	0.0133 (5)
C134	0.9828 (2)	0.3297 (2)	0.36647 (16)	0.0170 (5)
H134	0.9101	0.3658	0.3405	0.020*
C135	1.0950 (2)	0.2608 (2)	0.31686 (17)	0.0217 (6)
H135	1.0997	0.2509	0.2559	0.026*
C136	1.2021 (2)	0.2051 (2)	0.35422 (17)	0.0224 (6)
H136	1.2780	0.1586	0.3181	0.027*
C137	1.1995 (2)	0.2166 (2)	0.44269 (16)	0.0174 (5)
H137	1.2712	0.1775	0.4686	0.021*
C17A	1.0870 (2)	0.28790 (19)	0.49215 (15)	0.0129 (5)
C131	1.1538 (2)	0.2766 (2)	0.63886 (16)	0.0218 (6)
H13A	1.2325	0.3007	0.6178	0.026*
H13B	1.1805	0.1941	0.6367	0.026*
C141	1.0984 (2)	0.3190 (2)	0.73197 (16)	0.0149 (5)
C142	1.1034 (2)	0.4222 (2)	0.75954 (17)	0.0202 (5)
H142	1.1446	0.4651	0.7199	0.024*
C143	1.0485 (2)	0.4633 (2)	0.84465 (17)	0.0214 (6)
H143	1.0501	0.5349	0.8626	0.026*
C144	0.9916 (2)	0.3996 (2)	0.90330 (16)	0.0179 (5)
H144	0.9546	0.4273	0.9616	0.022*
C145	0.9885 (2)	0.2956 (2)	0.87698 (16)	0.0184 (5)

H145	0.9507	0.2514	0.9174	0.022*
C146	1.0409 (2)	0.2560 (2)	0.79148 (17)	0.0182 (5)
H146	1.0373	0.1851	0.7734	0.022*
N21	−0.0140 (2)	0.75831 (17)	0.92059 (14)	0.0192 (5)
C21	−0.0827 (2)	0.84265 (19)	0.90939 (15)	0.0127 (5)
C22	−0.1675 (2)	0.95153 (18)	0.89134 (15)	0.0119 (5)
H22	−0.2217	0.9411	0.8486	0.014*
C23	−0.2577 (2)	1.00946 (18)	0.97633 (15)	0.0121 (5)
O23	−0.21598 (16)	0.99589 (14)	1.04483 (11)	0.0193 (4)
C227	−0.0836 (2)	1.02767 (19)	0.85264 (15)	0.0140 (5)
H22A	−0.1419	1.1008	0.8409	0.017*
H22B	−0.0334	1.0405	0.8964	0.017*
C221	0.0100 (2)	0.97895 (19)	0.76913 (15)	0.0134 (5)
C222	−0.0218 (2)	0.9938 (2)	0.68648 (16)	0.0192 (5)
Br22	−0.19115 (3)	1.08181 (2)	0.67357 (2)	0.03007 (8)
C223	0.0658 (3)	0.9457 (2)	0.61163 (17)	0.0278 (6)
H223	0.0407	0.9565	0.5562	0.033*
C224	0.1898 (3)	0.8817 (2)	0.61815 (17)	0.0234 (6)
H224	0.2508	0.8489	0.5671	0.028*
C225	0.2247 (2)	0.8658 (2)	0.69909 (16)	0.0180 (5)
H225	0.3099	0.8217	0.7038	0.022*
C226	0.1361 (2)	0.9140 (2)	0.77368 (16)	0.0151 (5)
H226	0.1617	0.9025	0.8290	0.018*
N231	−0.55993 (18)	1.17202 (16)	0.91369 (13)	0.0137 (4)
C232	−0.4425 (2)	1.09431 (19)	0.89647 (15)	0.0137 (5)
H232	−0.4059	1.0531	0.8426	0.016*
C233	−0.3829 (2)	1.08334 (19)	0.96888 (15)	0.0124 (5)
C23A	−0.4724 (2)	1.16068 (18)	1.03560 (15)	0.0111 (4)
C234	−0.4707 (2)	1.1906 (2)	1.12156 (15)	0.0159 (5)
H234	−0.3978	1.1566	1.1479	0.019*
C235	−0.5773 (2)	1.2708 (2)	1.16723 (16)	0.0191 (5)
H235	−0.5775	1.2910	1.2258	0.023*
C236	−0.6849 (2)	1.3230 (2)	1.12889 (16)	0.0198 (5)
H236	−0.7565	1.3779	1.1619	0.024*
C237	−0.6888 (2)	1.2960 (2)	1.04415 (16)	0.0166 (5)
H237	−0.7613	1.3316	1.0178	0.020*
C27A	−0.5817 (2)	1.21439 (19)	0.99891 (15)	0.0123 (5)
C231	−0.6569 (2)	1.2033 (2)	0.85626 (16)	0.0191 (5)
H23B	−0.6932	1.2854	0.8583	0.023*
H23A	−0.7294	1.1714	0.8779	0.023*
C241	−0.5981 (2)	1.16213 (19)	0.76315 (15)	0.0145 (5)
C242	−0.5973 (2)	1.0580 (2)	0.73402 (17)	0.0194 (5)
H242	−0.6364	1.0129	0.7727	0.023*
C243	−0.5399 (2)	1.0189 (2)	0.64900 (17)	0.0204 (5)
H243	−0.5384	0.9468	0.6300	0.025*
C244	−0.4846 (2)	1.0852 (2)	0.59180 (16)	0.0178 (5)
H244	−0.4451	1.0586	0.5336	0.021*
C245	−0.4871 (2)	1.1902 (2)	0.61982 (16)	0.0187 (5)

H245	−0.4506	1.2363	0.5805	0.022*
C246	−0.5431 (2)	1.22815 (19)	0.70545 (16)	0.0156 (5)
H246	−0.5437	1.2999	0.7246	0.019*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N11	0.0183 (11)	0.0179 (11)	0.0181 (11)	−0.0017 (9)	0.0017 (9)	0.0006 (9)
C11	0.0118 (11)	0.0158 (12)	0.0105 (11)	−0.0041 (9)	0.0015 (9)	−0.0027 (9)
C12	0.0126 (11)	0.0112 (11)	0.0105 (11)	−0.0006 (9)	−0.0023 (9)	−0.0011 (9)
C13	0.0137 (11)	0.0124 (12)	0.0145 (12)	−0.0041 (9)	−0.0025 (9)	−0.0017 (9)
O13	0.0193 (9)	0.0243 (10)	0.0112 (9)	0.0016 (7)	−0.0062 (7)	−0.0024 (7)
C127	0.0161 (12)	0.0106 (11)	0.0156 (12)	−0.0036 (9)	−0.0021 (9)	−0.0017 (9)
C121	0.0170 (12)	0.0143 (12)	0.0141 (12)	−0.0093 (9)	−0.0020 (9)	−0.0003 (9)
C122	0.0203 (13)	0.0197 (13)	0.0178 (13)	−0.0092 (10)	−0.0053 (10)	0.0017 (10)
Br12	0.02806 (16)	0.03083 (16)	0.02740 (16)	−0.00258 (12)	−0.01353 (12)	0.00779 (12)
C123	0.0300 (15)	0.0390 (17)	0.0155 (13)	−0.0199 (13)	−0.0049 (11)	0.0026 (12)
C124	0.0236 (14)	0.0372 (16)	0.0196 (14)	−0.0165 (12)	0.0068 (11)	−0.0091 (12)
C125	0.0154 (12)	0.0234 (14)	0.0268 (15)	−0.0077 (10)	0.0024 (11)	−0.0029 (11)
C126	0.0152 (12)	0.0183 (13)	0.0167 (12)	−0.0096 (10)	−0.0030 (10)	0.0013 (10)
N131	0.0139 (10)	0.0157 (10)	0.0128 (10)	0.0001 (8)	−0.0045 (8)	−0.0008 (8)
C132	0.0157 (12)	0.0165 (12)	0.0116 (12)	−0.0011 (9)	−0.0015 (9)	−0.0021 (9)
C133	0.0139 (11)	0.0140 (12)	0.0121 (12)	−0.0034 (9)	−0.0035 (9)	−0.0011 (9)
C13A	0.0141 (11)	0.0120 (11)	0.0136 (12)	−0.0040 (9)	−0.0014 (9)	−0.0009 (9)
C134	0.0183 (12)	0.0171 (12)	0.0157 (12)	−0.0046 (10)	−0.0046 (10)	0.0000 (10)
C135	0.0236 (13)	0.0268 (14)	0.0124 (12)	−0.0052 (11)	−0.0007 (10)	−0.0071 (10)
C136	0.0175 (13)	0.0242 (14)	0.0207 (14)	−0.0023 (10)	0.0035 (10)	−0.0080 (11)
C137	0.0125 (12)	0.0172 (13)	0.0197 (13)	−0.0015 (9)	−0.0004 (10)	−0.0019 (10)
C17A	0.0144 (11)	0.0107 (11)	0.0136 (12)	−0.0043 (9)	−0.0016 (9)	−0.0011 (9)
C131	0.0140 (12)	0.0283 (15)	0.0174 (13)	0.0042 (10)	−0.0056 (10)	0.0003 (11)
C141	0.0114 (11)	0.0170 (12)	0.0147 (12)	0.0007 (9)	−0.0063 (9)	0.0015 (9)
C142	0.0191 (13)	0.0200 (13)	0.0237 (14)	−0.0076 (10)	−0.0075 (11)	0.0099 (11)
C143	0.0244 (14)	0.0154 (13)	0.0259 (15)	−0.0064 (10)	−0.0074 (11)	−0.0007 (10)
C144	0.0182 (12)	0.0206 (13)	0.0152 (13)	−0.0043 (10)	−0.0056 (10)	−0.0014 (10)
C145	0.0215 (13)	0.0219 (13)	0.0154 (13)	−0.0104 (10)	−0.0067 (10)	0.0060 (10)
C146	0.0212 (13)	0.0141 (12)	0.0223 (14)	−0.0059 (10)	−0.0106 (10)	0.0011 (10)
N21	0.0190 (11)	0.0189 (11)	0.0163 (11)	−0.0018 (9)	−0.0010 (9)	0.0026 (9)
C21	0.0127 (11)	0.0154 (12)	0.0094 (11)	−0.0040 (9)	−0.0001 (9)	−0.0024 (9)
C22	0.0125 (11)	0.0125 (11)	0.0094 (11)	−0.0007 (9)	−0.0033 (9)	−0.0003 (9)
C23	0.0152 (11)	0.0117 (11)	0.0096 (11)	−0.0040 (9)	−0.0028 (9)	−0.0002 (9)
O23	0.0185 (9)	0.0236 (10)	0.0123 (9)	0.0013 (7)	−0.0062 (7)	−0.0025 (7)
C227	0.0144 (11)	0.0115 (11)	0.0148 (12)	−0.0017 (9)	−0.0029 (9)	−0.0005 (9)
C221	0.0145 (11)	0.0119 (11)	0.0146 (12)	−0.0064 (9)	−0.0010 (9)	0.0010 (9)
C222	0.0164 (12)	0.0184 (13)	0.0192 (13)	0.0010 (10)	−0.0044 (10)	0.0031 (10)
Br22	0.02352 (15)	0.03471 (17)	0.02276 (16)	0.00809 (11)	−0.00826 (11)	0.00540 (12)
C223	0.0295 (15)	0.0362 (16)	0.0118 (13)	−0.0010 (12)	−0.0029 (11)	0.0038 (11)
C224	0.0208 (13)	0.0263 (14)	0.0171 (13)	−0.0025 (11)	0.0048 (10)	−0.0002 (11)
C225	0.0122 (11)	0.0198 (13)	0.0209 (13)	−0.0043 (9)	−0.0008 (10)	0.0015 (10)

C226	0.0147 (12)	0.0179 (12)	0.0149 (12)	−0.0078 (9)	−0.0034 (9)	0.0019 (9)
N231	0.0111 (9)	0.0161 (10)	0.0120 (10)	−0.0002 (8)	−0.0030 (8)	−0.0018 (8)
C232	0.0132 (11)	0.0141 (12)	0.0117 (12)	−0.0006 (9)	−0.0026 (9)	−0.0015 (9)
C233	0.0146 (11)	0.0112 (11)	0.0107 (11)	−0.0027 (9)	−0.0023 (9)	−0.0008 (9)
C23A	0.0128 (11)	0.0100 (11)	0.0101 (11)	−0.0037 (9)	−0.0007 (9)	0.0000 (9)
C234	0.0155 (12)	0.0175 (12)	0.0147 (12)	−0.0042 (9)	−0.0038 (9)	0.0001 (10)
C235	0.0233 (13)	0.0222 (13)	0.0115 (12)	−0.0079 (10)	−0.0002 (10)	−0.0059 (10)
C236	0.0178 (12)	0.0191 (13)	0.0187 (13)	−0.0027 (10)	0.0023 (10)	−0.0043 (10)
C237	0.0123 (11)	0.0162 (12)	0.0200 (13)	−0.0032 (9)	−0.0015 (10)	0.0007 (10)
C27A	0.0136 (11)	0.0124 (11)	0.0108 (11)	−0.0050 (9)	0.0000 (9)	0.0008 (9)
C231	0.0156 (12)	0.0252 (14)	0.0145 (13)	0.0000 (10)	−0.0073 (10)	0.0006 (10)
C241	0.0135 (12)	0.0164 (12)	0.0133 (12)	−0.0011 (9)	−0.0070 (9)	0.0022 (9)
C242	0.0223 (13)	0.0186 (13)	0.0195 (13)	−0.0067 (10)	−0.0085 (11)	0.0076 (10)
C243	0.0253 (14)	0.0148 (12)	0.0247 (14)	−0.0073 (10)	−0.0107 (11)	−0.0007 (10)
C244	0.0188 (12)	0.0197 (13)	0.0150 (12)	−0.0042 (10)	−0.0058 (10)	−0.0023 (10)
C245	0.0225 (13)	0.0194 (13)	0.0174 (13)	−0.0096 (10)	−0.0066 (10)	0.0028 (10)
C246	0.0213 (12)	0.0109 (11)	0.0166 (13)	−0.0043 (9)	−0.0091 (10)	−0.0008 (9)

Geometric parameters (Å, º)

N11—C11	1.137 (3)	N21—C21	1.139 (3)
C11—C12	1.468 (3)	C21—C22	1.468 (3)
C12—C13	1.544 (3)	C22—C23	1.545 (3)
C12—C127	1.554 (3)	C22—C227	1.551 (3)
C12—H12	1.0000	C22—H22	1.0000
C13—O13	1.227 (3)	C23—O23	1.226 (3)
C13—C133	1.435 (3)	C23—C233	1.436 (3)
C127—C121	1.510 (3)	C227—C221	1.511 (3)
C127—H12A	0.9900	C227—H22A	0.9900
C127—H12B	0.9900	C227—H22B	0.9900
C121—C122	1.393 (3)	C221—C222	1.391 (3)
C121—C126	1.396 (3)	C221—C226	1.396 (3)
C122—C123	1.387 (4)	C222—C223	1.385 (4)
C122—Br12	1.902 (3)	C222—Br22	1.903 (2)
C123—C124	1.384 (4)	C223—C224	1.381 (4)
C123—H123	0.9500	C223—H223	0.9500
C124—C125	1.381 (4)	C224—C225	1.378 (4)
C124—H124	0.9500	C224—H224	0.9500
C125—C126	1.383 (3)	C225—C226	1.387 (3)
C125—H125	0.9500	C225—H225	0.9500
C126—H126	0.9500	C226—H226	0.9500
N131—C132	1.351 (3)	N231—C232	1.351 (3)
N131—C17A	1.389 (3)	N231—C27A	1.390 (3)
N131—C131	1.472 (3)	N231—C231	1.473 (3)
C132—C133	1.390 (3)	C232—C233	1.388 (3)
C132—H132	0.9500	C232—H232	0.9500
C133—C13A	1.447 (3)	C233—C23A	1.444 (3)
C13A—C134	1.398 (3)	C23A—C234	1.401 (3)

C13A—C17A	1.409 (3)	C23A—C27A	1.406 (3)
C134—C135	1.381 (3)	C234—C235	1.382 (3)
C134—H134	0.9500	C234—H234	0.9500
C135—C136	1.398 (4)	C235—C236	1.402 (4)
C135—H135	0.9500	C235—H235	0.9500
C136—C137	1.382 (4)	C236—C237	1.379 (3)
C136—H136	0.9500	C236—H236	0.9500
C137—C17A	1.393 (3)	C237—C27A	1.392 (3)
C137—H137	0.9500	C237—H237	0.9500
C131—C141	1.506 (3)	C231—C241	1.507 (3)
C131—H13A	0.9900	C231—H23B	0.9900
C131—H13B	0.9900	C231—H23A	0.9900
C141—C142	1.389 (4)	C241—C242	1.385 (3)
C141—C146	1.390 (4)	C241—C246	1.388 (3)
C142—C143	1.391 (4)	C242—C243	1.388 (4)
C142—H142	0.9500	C242—H242	0.9500
C143—C144	1.385 (4)	C243—C244	1.387 (4)
C143—H143	0.9500	C243—H243	0.9500
C144—C145	1.384 (4)	C244—C245	1.384 (3)
C144—H144	0.9500	C244—H244	0.9500
C145—C146	1.387 (4)	C245—C246	1.390 (3)
C145—H145	0.9500	C245—H245	0.9500
C146—H146	0.9500	C246—H246	0.9500

N11—C11—C12	178.1 (3)	N21—C21—C22	177.5 (3)
C11—C12—C13	109.69 (19)	C21—C22—C23	110.56 (19)
C11—C12—C127	109.87 (19)	C21—C22—C227	109.81 (19)
C13—C12—C127	108.84 (18)	C23—C22—C227	108.10 (19)
C11—C12—H12	109.5	C21—C22—H22	109.4
C13—C12—H12	109.5	C23—C22—H22	109.4
C127—C12—H12	109.5	C227—C22—H22	109.4
O13—C13—C133	123.3 (2)	O23—C23—C233	123.7 (2)
O13—C13—C12	118.9 (2)	O23—C23—C22	118.8 (2)
C133—C13—C12	117.8 (2)	C233—C23—C22	117.35 (19)
C121—C127—C12	113.25 (19)	C221—C227—C22	112.96 (19)
C121—C127—H12A	108.9	C221—C227—H22A	109.0
C12—C127—H12A	108.9	C22—C227—H22A	109.0
C121—C127—H12B	108.9	C221—C227—H22B	109.0
C12—C127—H12B	108.9	C22—C227—H22B	109.0
H12A—C127—H12B	107.7	H22A—C227—H22B	107.8
C122—C121—C126	116.5 (2)	C222—C221—C226	116.9 (2)
C122—C121—C127	123.7 (2)	C222—C221—C227	124.0 (2)
C126—C121—C127	119.8 (2)	C226—C221—C227	119.1 (2)
C123—C122—C121	122.5 (2)	C223—C222—C221	122.2 (2)
C123—C122—Br12	117.2 (2)	C223—C222—Br22	117.78 (19)
C121—C122—Br12	120.30 (19)	C221—C222—Br22	120.00 (18)
C124—C123—C122	119.3 (3)	C224—C223—C222	119.6 (2)
C124—C123—H123	120.4	C224—C223—H223	120.2

C122—C123—H123	120.4	C222—C223—H223	120.2
C125—C124—C123	119.8 (2)	C225—C224—C223	119.7 (2)
C125—C124—H124	120.1	C225—C224—H224	120.2
C123—C124—H124	120.1	C223—C224—H224	120.2
C124—C125—C126	120.1 (2)	C224—C225—C226	120.3 (2)
C124—C125—H125	119.9	C224—C225—H225	119.8
C126—C125—H125	119.9	C226—C225—H225	119.8
C125—C126—C121	121.8 (2)	C225—C226—C221	121.3 (2)
C125—C126—H126	119.1	C225—C226—H226	119.3
C121—C126—H126	119.1	C221—C226—H226	119.3
C132—N131—C17A	109.16 (19)	C232—N231—C27A	109.04 (19)
C132—N131—C131	127.8 (2)	C232—N231—C231	127.2 (2)
C17A—N131—C131	122.94 (19)	C27A—N231—C231	123.64 (19)
N131—C132—C133	110.2 (2)	N231—C232—C233	110.2 (2)
N131—C132—H132	124.9	N231—C232—H232	124.9
C133—C132—H132	124.9	C233—C232—H232	124.9
C132—C133—C13	126.9 (2)	C232—C233—C23	127.0 (2)
C132—C133—C13A	106.3 (2)	C232—C233—C23A	106.3 (2)
C13—C133—C13A	126.8 (2)	C23—C233—C23A	126.8 (2)
C134—C13A—C17A	118.9 (2)	C234—C23A—C27A	118.7 (2)
C134—C13A—C133	134.8 (2)	C234—C23A—C233	134.8 (2)
C17A—C13A—C133	106.4 (2)	C27A—C23A—C233	106.5 (2)
C135—C134—C13A	118.6 (2)	C235—C234—C23A	118.7 (2)
C135—C134—H134	120.7	C235—C234—H234	120.7
C13A—C134—H134	120.7	C23A—C234—H234	120.7
C134—C135—C136	121.6 (2)	C234—C235—C236	121.4 (2)
C134—C135—H135	119.2	C234—C235—H235	119.3
C136—C135—H135	119.2	C236—C235—H235	119.3
C137—C136—C135	121.2 (2)	C237—C236—C235	121.3 (2)
C137—C136—H136	119.4	C237—C236—H236	119.4
C135—C136—H136	119.4	C235—C236—H236	119.4
C136—C137—C17A	116.9 (2)	C236—C237—C27A	117.0 (2)
C136—C137—H137	121.5	C236—C237—H237	121.5
C17A—C137—H137	121.5	C27A—C237—H237	121.5
N131—C17A—C137	129.2 (2)	N231—C27A—C237	129.0 (2)
N131—C17A—C13A	108.0 (2)	N231—C27A—C23A	107.97 (19)
C137—C17A—C13A	122.8 (2)	C237—C27A—C23A	123.0 (2)
N131—C131—C141	112.18 (19)	N231—C231—C241	111.72 (19)
N131—C131—H13A	109.2	N231—C231—H23B	109.3
C141—C131—H13A	109.2	C241—C231—H23B	109.3
N131—C131—H13B	109.2	N231—C231—H23A	109.3
C141—C131—H13B	109.2	C241—C231—H23A	109.3
H13A—C131—H13B	107.9	H23B—C231—H23A	107.9
C142—C141—C146	118.9 (2)	C242—C241—C246	119.0 (2)
C142—C141—C131	120.3 (2)	C242—C241—C231	120.8 (2)
C146—C141—C131	120.7 (2)	C246—C241—C231	120.2 (2)
C141—C142—C143	120.5 (2)	C241—C242—C243	120.7 (2)
C141—C142—H142	119.7	C241—C242—H242	119.6

C143—C142—H142	119.7	C243—C242—H242	119.6
C144—C143—C142	119.9 (2)	C244—C243—C242	119.9 (2)
C144—C143—H143	120.1	C244—C243—H243	120.1
C142—C143—H143	120.1	C242—C243—H243	120.1
C145—C144—C143	120.0 (2)	C245—C244—C243	119.8 (2)
C145—C144—H144	120.0	C245—C244—H244	120.1
C143—C144—H144	120.0	C243—C244—H244	120.1
C144—C145—C146	119.9 (2)	C244—C245—C246	120.0 (2)
C144—C145—H145	120.1	C244—C245—H245	120.0
C146—C145—H145	120.1	C246—C245—H245	120.0
C145—C146—C141	120.7 (2)	C241—C246—C245	120.5 (2)
C145—C146—H146	119.6	C241—C246—H246	119.7
C141—C146—H146	119.6	C245—C246—H246	119.7

C11—C12—C13—O13	−31.9 (3)	C21—C22—C23—O23	34.5 (3)
C127—C12—C13—O13	88.3 (3)	C227—C22—C23—O23	−85.7 (3)
C11—C12—C13—C133	151.1 (2)	C21—C22—C23—C233	−150.1 (2)
C127—C12—C13—C133	−88.6 (2)	C227—C22—C23—C233	89.7 (2)
C11—C12—C127—C121	−61.9 (3)	C21—C22—C227—C221	58.8 (3)
C13—C12—C127—C121	177.96 (19)	C23—C22—C227—C221	179.53 (18)
C12—C127—C121—C122	−93.1 (3)	C22—C227—C221—C222	87.4 (3)
C12—C127—C121—C126	87.4 (3)	C22—C227—C221—C226	−92.0 (3)
C126—C121—C122—C123	2.0 (4)	C226—C221—C222—C223	0.9 (4)
C127—C121—C122—C123	−177.5 (2)	C227—C221—C222—C223	−178.5 (2)
C126—C121—C122—Br12	−177.34 (17)	C226—C221—C222—Br22	−179.65 (18)
C127—C121—C122—Br12	3.1 (3)	C227—C221—C222—Br22	0.9 (3)
C121—C122—C123—C124	−3.0 (4)	C221—C222—C223—C224	−0.9 (4)
Br12—C122—C123—C124	176.3 (2)	Br22—C222—C223—C224	179.7 (2)
C122—C123—C124—C125	1.6 (4)	C222—C223—C224—C225	0.5 (4)
C123—C124—C125—C126	0.7 (4)	C223—C224—C225—C226	−0.2 (4)
C124—C125—C126—C121	−1.8 (4)	C224—C225—C226—C221	0.2 (4)
C122—C121—C126—C125	0.4 (3)	C222—C221—C226—C225	−0.6 (3)
C127—C121—C126—C125	179.9 (2)	C227—C221—C226—C225	178.9 (2)
C17A—N131—C132—C133	0.1 (3)	C27A—N231—C232—C233	−0.4 (3)
C131—N131—C132—C133	177.4 (2)	C231—N231—C232—C233	−175.8 (2)
N131—C132—C133—C13	−178.4 (2)	N231—C232—C233—C23	180.0 (2)
N131—C132—C133—C13A	−0.1 (3)	N231—C232—C233—C23A	0.3 (3)
O13—C13—C133—C132	174.1 (2)	O23—C23—C233—C232	−172.6 (2)
C12—C13—C133—C132	−9.1 (4)	C22—C23—C233—C232	12.3 (4)
O13—C13—C133—C13A	−3.9 (4)	O23—C23—C233—C23A	7.0 (4)
C12—C13—C133—C13A	172.9 (2)	C22—C23—C233—C23A	−168.1 (2)
C132—C133—C13A—C134	−179.2 (3)	C232—C233—C23A—C234	−179.3 (3)
C13—C133—C13A—C134	−0.9 (4)	C23—C233—C23A—C234	1.1 (4)
C132—C133—C13A—C17A	0.0 (3)	C232—C233—C23A—C27A	−0.1 (3)
C13—C133—C13A—C17A	178.4 (2)	C23—C233—C23A—C27A	−179.8 (2)
C17A—C13A—C134—C135	−1.5 (4)	C27A—C23A—C234—C235	0.8 (3)
C133—C13A—C134—C135	177.7 (3)	C233—C23A—C234—C235	179.9 (3)
C13A—C134—C135—C136	1.3 (4)	C23A—C234—C235—C236	−0.8 (4)

C134—C135—C136—C137	0.3 (4)	C234—C235—C236—C237	0.2 (4)
C135—C136—C137—C17A	−1.5 (4)	C235—C236—C237—C27A	0.5 (4)
C132—N131—C17A—C137	179.0 (2)	C232—N231—C27A—C237	179.6 (2)
C131—N131—C17A—C137	1.5 (4)	C231—N231—C27A—C237	−4.8 (4)
C132—N131—C17A—C13A	−0.1 (3)	C232—N231—C27A—C23A	0.3 (3)
C131—N131—C17A—C13A	−177.6 (2)	C231—N231—C27A—C23A	176.0 (2)
C136—C137—C17A—N131	−177.7 (2)	C236—C237—C27A—N231	−179.7 (2)
C136—C137—C17A—C13A	1.2 (4)	C236—C237—C27A—C23A	−0.5 (4)
C134—C13A—C17A—N131	179.4 (2)	C234—C23A—C27A—N231	179.2 (2)
C133—C13A—C17A—N131	0.1 (3)	C233—C23A—C27A—N231	−0.1 (3)
C134—C13A—C17A—C137	0.3 (4)	C234—C23A—C27A—C237	−0.1 (4)
C133—C13A—C17A—C137	−179.1 (2)	C233—C23A—C27A—C237	−179.4 (2)
C132—N131—C131—C141	4.5 (4)	C232—N231—C231—C241	−16.2 (3)
C17A—N131—C131—C141	−178.5 (2)	C27A—N231—C231—C241	169.0 (2)
N131—C131—C141—C142	−85.5 (3)	N231—C231—C241—C242	89.9 (3)
N131—C131—C141—C146	94.2 (3)	N231—C231—C241—C246	−89.6 (3)
C146—C141—C142—C143	−1.8 (3)	C246—C241—C242—C243	1.4 (3)
C131—C141—C142—C143	177.9 (2)	C231—C241—C242—C243	−178.2 (2)
C141—C142—C143—C144	1.8 (4)	C241—C242—C243—C244	−1.1 (4)
C142—C143—C144—C145	−0.4 (4)	C242—C243—C244—C245	−0.1 (4)
C143—C144—C145—C146	−1.0 (4)	C243—C244—C245—C246	1.0 (4)
C144—C145—C146—C141	1.0 (4)	C242—C241—C246—C245	−0.4 (3)
C142—C141—C146—C145	0.4 (3)	C231—C241—C246—C245	179.1 (2)
C131—C141—C146—C145	−179.3 (2)	C244—C245—C246—C241	−0.8 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C225—H225···N11	0.95	2.62	3.304 (4)	129
C231—H23*A*···O23^{i}	0.99	2.57	3.415 (4)	143

Symmetry code: (i) −*x*−1, −*y*+2, −*z*+2.

(*E*)-1-(1-Acetyl-1*H*-indol-3-yl)-3-(2-bromophenyl)-2-cyanoprop-1-en-1-yl acetate (III)

Crystal data

$C_{22}H_{17}BrN_2O_3$
M_r = 437.28
Monoclinic, *C*2/*c*
a = 19.406 (9) Å
b = 8.773 (4) Å
c = 23.028 (9) Å
β = 103.318 (15)°
V = 3815 (3) Å^3
Z = 8

F(000) = 1776
D_x = 1.523 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 4412 reflections
θ = 2.5–27.6°
μ = 2.18 mm^{-1}
T = 173 K
Block, colourless
0.42 × 0.32 × 0.24 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} = 0.420, T_{max} = 0.594
49228 measured reflections
4379 independent reflections
3435 reflections with $I > 2\sigma(I)$
R_{int} = 0.057
θ_{max} = 27.5°, θ_{min} = 2.5°
h = −25→25
k = −11→11
l = −29→29

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.136
S = 1.08
4379 reflections
255 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0427P)^2 + 18.903P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.80 e Å^{-3}
$\Delta\rho_{min}$ = −1.89 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}
C1	0.62414 (17)	0.3935 (4)	0.29701 (16)	0.0337 (8)
C2	0.62775 (17)	0.4887 (4)	0.34346 (16)	0.0379 (8)
C3	0.65936 (19)	0.4495 (5)	0.40750 (16)	0.0428 (10)
H3A	0.6666	0.3378	0.4107	0.051*
H3B	0.6250	0.4771	0.4316	0.051*
O11	0.64160 (14)	0.2417 (3)	0.31041 (10)	0.0376 (6)
C11	0.6943 (2)	0.1770 (4)	0.28804 (15)	0.0372 (8)
O12	0.73268 (15)	0.2507 (3)	0.26607 (14)	0.0533 (8)
C111	0.6960 (3)	0.0090 (4)	0.29636 (17)	0.0619 (14)
H11A	0.7145	−0.0392	0.2647	0.093*
H11B	0.6480	−0.0284	0.2946	0.093*
H11C	0.7266	−0.0163	0.3353	0.093*
N11	0.57308 (15)	0.5602 (3)	0.14756 (14)	0.0386 (7)
C12	0.60600 (18)	0.5633 (4)	0.20784 (16)	0.0374 (8)
H12	0.6310	0.6483	0.2280	0.045*
C13	0.59756 (16)	0.4290 (4)	0.23388 (16)	0.0325 (7)
C13A	0.55771 (16)	0.3298 (4)	0.18758 (16)	0.0359 (8)
C14	0.53385 (19)	0.1801 (5)	0.18688 (18)	0.0452 (10)
H14	0.5439	0.1197	0.2221	0.054*
C15	0.4948 (2)	0.1214 (5)	0.1331 (2)	0.0531 (12)
H15	0.4766	0.0206	0.1321	0.064*

C16	0.48178 (18)	0.2070 (5)	0.08106 (19)	0.0474 (11)
H16	0.4562	0.1621	0.0449	0.057*
C17	0.50493 (16)	0.3554 (5)	0.08029 (18)	0.0408 (9)
H17	0.4958	0.4140	0.0446	0.049*
C17A	0.54247 (16)	0.4151 (4)	0.13455 (17)	0.0373 (8)
C18	0.5663 (2)	0.6840 (4)	0.10827 (19)	0.0518 (11)
O18	0.52769 (17)	0.6757 (3)	0.05891 (13)	0.0612 (9)
C19	0.6093 (3)	0.8218 (5)	0.1310 (2)	0.083 (2)
H19A	0.6047	0.8970	0.0988	0.125*
H19B	0.6592	0.7927	0.1449	0.125*
H19C	0.5923	0.8662	0.1642	0.125*
C21	0.59703 (18)	0.6371 (5)	0.33325 (19)	0.0472 (10)
N21	0.57241 (18)	0.7561 (5)	0.32946 (19)	0.0635 (12)
C31	0.72899 (17)	0.5274 (4)	0.43386 (15)	0.0318 (7)
C32	0.75899 (19)	0.5183 (4)	0.49450 (16)	0.0363 (8)
Br32	0.71124 (3)	0.40569 (5)	0.54420 (2)	0.06003 (18)
C33	0.82242 (19)	0.5875 (4)	0.52081 (19)	0.0450 (9)
H33	0.8413	0.5791	0.5626	0.054*
C34	0.85800 (19)	0.6691 (5)	0.4856 (2)	0.0520 (12)
H34	0.9016	0.7177	0.5030	0.062*
C35	0.82997 (19)	0.6798 (5)	0.4250 (2)	0.0478 (11)
H35	0.8547	0.7353	0.4007	0.057*
C36	0.76622 (17)	0.6105 (4)	0.39925 (17)	0.0357 (8)
H36	0.7475	0.6194	0.3575	0.043*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0289 (16)	0.0368 (18)	0.0431 (18)	−0.0134 (14)	0.0244 (14)	−0.0193 (15)
C2	0.0262 (16)	0.046 (2)	0.049 (2)	−0.0179 (15)	0.0247 (15)	−0.0261 (17)
C3	0.042 (2)	0.054 (2)	0.0413 (19)	−0.0323 (17)	0.0287 (16)	−0.0278 (17)
O11	0.0553 (15)	0.0328 (12)	0.0344 (12)	−0.0188 (11)	0.0304 (11)	−0.0129 (10)
C11	0.063 (2)	0.0254 (16)	0.0305 (16)	−0.0017 (15)	0.0254 (16)	0.0015 (13)
O12	0.0598 (17)	0.0359 (14)	0.080 (2)	0.0198 (13)	0.0487 (16)	0.0268 (14)
C111	0.135 (5)	0.0218 (17)	0.0329 (19)	−0.010 (2)	0.028 (2)	−0.0060 (14)
N11	0.0341 (15)	0.0296 (14)	0.0467 (17)	0.0119 (12)	−0.0017 (13)	−0.0189 (13)
C12	0.0333 (17)	0.0328 (17)	0.0437 (19)	0.0079 (14)	0.0034 (15)	−0.0211 (15)
C13	0.0243 (15)	0.0335 (17)	0.0435 (18)	−0.0025 (13)	0.0158 (14)	−0.0209 (14)
C13A	0.0199 (15)	0.0441 (19)	0.049 (2)	−0.0058 (13)	0.0203 (14)	−0.0261 (16)
C14	0.0328 (18)	0.056 (2)	0.056 (2)	−0.0243 (17)	0.0307 (17)	−0.0271 (19)
C15	0.038 (2)	0.064 (3)	0.067 (3)	−0.0304 (19)	0.033 (2)	−0.036 (2)
C16	0.0216 (16)	0.065 (3)	0.060 (2)	−0.0111 (16)	0.0176 (16)	−0.036 (2)
C17	0.0166 (14)	0.050 (2)	0.055 (2)	0.0042 (14)	0.0073 (14)	−0.0285 (18)
C17A	0.0171 (14)	0.0426 (19)	0.053 (2)	0.0052 (13)	0.0102 (14)	−0.0257 (17)
C18	0.061 (3)	0.0333 (19)	0.050 (2)	0.0215 (18)	−0.010 (2)	−0.0204 (17)
O18	0.070 (2)	0.0474 (17)	0.0525 (17)	0.0266 (15)	−0.0145 (15)	−0.0224 (14)
C19	0.134 (5)	0.0234 (19)	0.064 (3)	0.011 (2)	−0.035 (3)	−0.0124 (19)
C21	0.0237 (16)	0.061 (2)	0.061 (2)	−0.0117 (17)	0.0190 (16)	−0.042 (2)

N21	0.0372 (18)	0.066 (2)	0.090 (3)	0.0017 (17)	0.0200 (18)	−0.051 (2)
C31	0.0292 (16)	0.0276 (15)	0.0457 (19)	−0.0128 (13)	0.0235 (14)	−0.0206 (14)
C32	0.0383 (18)	0.0296 (17)	0.047 (2)	−0.0078 (14)	0.0212 (16)	−0.0194 (15)
Br32	0.0965 (4)	0.0472 (3)	0.0470 (2)	−0.0271 (2)	0.0384 (2)	−0.01872 (18)
C33	0.0353 (19)	0.046 (2)	0.055 (2)	0.0015 (16)	0.0134 (17)	−0.0282 (18)
C34	0.0247 (17)	0.060 (3)	0.076 (3)	−0.0150 (17)	0.0211 (18)	−0.042 (2)
C35	0.0351 (19)	0.048 (2)	0.072 (3)	−0.0234 (16)	0.0369 (19)	−0.031 (2)
C36	0.0312 (17)	0.0341 (17)	0.050 (2)	−0.0160 (14)	0.0267 (15)	−0.0218 (15)

Geometric parameters (Å, º)

C1—C2	1.346 (4)	C15—C16	1.388 (7)
C1—O11	1.391 (4)	C15—H15	0.9500
C1—C13	1.460 (5)	C16—C17	1.378 (6)
C2—C21	1.429 (6)	C16—H16	0.9500
C2—C3	1.501 (6)	C17—C17A	1.395 (5)
C3—C31	1.511 (4)	C17—H17	0.9500
C3—H3A	0.9900	C18—O18	1.211 (5)
C3—H3B	0.9900	C18—C19	1.494 (6)
O11—C11	1.369 (4)	C19—H19A	0.9800
C11—O12	1.185 (4)	C19—H19B	0.9800
C11—C111	1.486 (5)	C19—H19C	0.9800
C111—H11A	0.9800	C21—N21	1.143 (6)
C111—H11B	0.9800	C31—C32	1.386 (5)
C111—H11C	0.9800	C31—C36	1.398 (4)
N11—C12	1.388 (5)	C32—C33	1.381 (5)
N11—C18	1.400 (5)	C32—Br32	1.906 (4)
N11—C17A	1.407 (4)	C33—C34	1.381 (6)
C12—C13	1.349 (5)	C33—H33	0.9500
C12—H12	0.9500	C34—C35	1.378 (6)
C13—C13A	1.453 (4)	C34—H34	0.9500
C13A—C14	1.392 (5)	C35—C36	1.383 (5)
C13A—C17A	1.404 (6)	C35—H35	0.9500
C14—C15	1.393 (5)	C36—H36	0.9500
C14—H14	0.9500		

C2—C1—O11	116.9 (3)	C14—C15—H15	119.3
C2—C1—C13	126.9 (3)	C17—C16—C15	121.8 (4)
O11—C1—C13	115.9 (3)	C17—C16—H16	119.1
C1—C2—C21	119.4 (4)	C15—C16—H16	119.1
C1—C2—C3	125.0 (4)	C16—C17—C17A	116.6 (4)
C21—C2—C3	115.5 (3)	C16—C17—H17	121.7
C2—C3—C31	114.6 (3)	C17A—C17—H17	121.7
C2—C3—H3A	108.6	C17—C17A—C13A	122.7 (4)
C31—C3—H3A	108.6	C17—C17A—N11	129.6 (4)
C2—C3—H3B	108.6	C13A—C17A—N11	107.6 (3)
C31—C3—H3B	108.6	O18—C18—N11	120.4 (4)
H3A—C3—H3B	107.6	O18—C18—C19	123.5 (4)

C11—O11—C1	118.6 (2)	N11—C18—C19	116.1 (3)
O12—C11—O11	122.1 (3)	C18—C19—H19A	109.5
O12—C11—C111	126.8 (4)	C18—C19—H19B	109.5
O11—C11—C111	111.1 (3)	H19A—C19—H19B	109.5
C11—C111—H11A	109.5	C18—C19—H19C	109.5
C11—C111—H11B	109.5	H19A—C19—H19C	109.5
H11A—C111—H11B	109.5	H19B—C19—H19C	109.5
C11—C111—H11C	109.5	N21—C21—C2	174.9 (5)
H11A—C111—H11C	109.5	C32—C31—C36	116.9 (3)
H11B—C111—H11C	109.5	C32—C31—C3	120.5 (3)
C12—N11—C18	125.7 (3)	C36—C31—C3	122.6 (3)
C12—N11—C17A	107.8 (3)	C33—C32—C31	122.8 (3)
C18—N11—C17A	126.3 (3)	C33—C32—Br32	118.1 (3)
C13—C12—N11	110.6 (3)	C31—C32—Br32	119.0 (2)
C13—C12—H12	124.7	C32—C33—C34	119.1 (4)
N11—C12—H12	124.7	C32—C33—H33	120.5
C12—C13—C13A	107.2 (3)	C34—C33—H33	120.5
C12—C13—C1	125.3 (3)	C35—C34—C33	119.7 (3)
C13A—C13—C1	127.5 (3)	C35—C34—H34	120.1
C14—C13A—C17A	119.3 (3)	C33—C34—H34	120.1
C14—C13A—C13	133.9 (4)	C34—C35—C36	120.6 (4)
C17A—C13A—C13	106.8 (3)	C34—C35—H35	119.7
C13A—C14—C15	118.1 (4)	C36—C35—H35	119.7
C13A—C14—H14	121.0	C35—C36—C31	120.9 (4)
C15—C14—H14	121.0	C35—C36—H36	119.6
C16—C15—C14	121.4 (4)	C31—C36—H36	119.6
C16—C15—H15	119.3		

O11—C1—C2—C21	−167.3 (3)	C16—C17—C17A—C13A	1.2 (4)
C13—C1—C2—C21	6.6 (5)	C16—C17—C17A—N11	179.2 (3)
O11—C1—C2—C3	9.8 (5)	C14—C13A—C17A—C17	−1.0 (5)
C13—C1—C2—C3	−176.3 (3)	C13—C13A—C17A—C17	179.5 (3)
C1—C2—C3—C31	108.7 (4)	C14—C13A—C17A—N11	−179.4 (3)
C21—C2—C3—C31	−74.1 (4)	C13—C13A—C17A—N11	1.2 (3)
C2—C1—O11—C11	−123.6 (3)	C12—N11—C17A—C17	−178.7 (3)
C13—C1—O11—C11	61.9 (4)	C18—N11—C17A—C17	6.6 (6)
C1—O11—C11—O12	12.3 (5)	C12—N11—C17A—C13A	−0.5 (3)
C1—O11—C11—C111	−168.6 (3)	C18—N11—C17A—C13A	−175.2 (3)
C18—N11—C12—C13	174.3 (3)	C12—N11—C18—O18	−168.9 (4)
C17A—N11—C12—C13	−0.5 (4)	C17A—N11—C18—O18	4.9 (6)
N11—C12—C13—C13A	1.2 (4)	C12—N11—C18—C19	11.6 (6)
N11—C12—C13—C1	−178.8 (3)	C17A—N11—C18—C19	−174.6 (4)
C2—C1—C13—C12	36.4 (5)	C2—C3—C31—C32	171.0 (3)
O11—C1—C13—C12	−149.7 (3)	C2—C3—C31—C36	−9.3 (5)
C2—C1—C13—C13A	−143.5 (3)	C36—C31—C32—C33	0.3 (5)
O11—C1—C13—C13A	30.3 (4)	C3—C31—C32—C33	−179.9 (3)
C12—C13—C13A—C14	179.2 (3)	C36—C31—C32—Br32	−179.6 (2)
C1—C13—C13A—C14	−0.8 (6)	C3—C31—C32—Br32	0.2 (4)

C12—C13—C13A—C17A	−1.4 (3)	C31—C32—C33—C34	−0.2 (5)
C1—C13—C13A—C17A	178.5 (3)	Br32—C32—C33—C34	179.7 (3)
C17A—C13A—C14—C15	−0.7 (5)	C32—C33—C34—C35	−0.2 (6)
C13—C13A—C14—C15	178.6 (3)	C33—C34—C35—C36	0.5 (6)
C13A—C14—C15—C16	2.2 (5)	C34—C35—C36—C31	−0.4 (6)
C14—C15—C16—C17	−2.0 (6)	C32—C31—C36—C35	0.0 (5)
C15—C16—C17—C17A	0.3 (5)	C3—C31—C36—C35	−179.8 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C17—H17···O18	0.95	2.38	2.904 (5)	114
C17—H17···O18^{i}	0.95	2.45	3.133 (5)	129
C35—H35···*Cg*2^{ii}	0.95	2.76	3.582 (5)	146

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

(*E*)-1-(1-Benzyl-1*H*-indol-3-yl)-3-(2-bromophenyl)-2-cyanoprop-1-en-1-yl benzoate (IV)

Crystal data

$C_{32}H_{23}BrN_2O_2$
M_r = 547.42
Monoclinic, $P2_1/c$
a = 14.886 (4) Å
b = 8.129 (3) Å
c = 21.432 (6) Å
β = 104.638 (12)°
V = 2509.3 (14) Å^3
Z = 4
$F(000)$ = 1120
D_x = 1.449 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 7902 reflections
θ = 2.7–31.7°
μ = 1.67 mm^{-1}
T = 100 K
Block, colourless
0.21 × 0.12 × 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} = 0.676, T_{max} = 0.818
87392 measured reflections
6999 independent reflections
5828 reflections with $I > 2\sigma(I)$
R_{int} = 0.074
θ_{max} = 29.5°, θ_{min} = 2.7°
h = −20→20
k = −10→11
l = −29→29

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.086
S = 1.05
6999 reflections
334 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0363P)^2 + 2.1078P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.94 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}
C1	0.25289 (10)	0.82801 (18)	0.47696 (7)	0.0105 (3)
C2	0.27542 (10)	0.93568 (19)	0.52616 (7)	0.0106 (3)
C3	0.20854 (11)	1.04297 (19)	0.55102 (7)	0.0124 (3)
H3A	0.2265	1.1599	0.5494	0.015*
H3B	0.1450	1.0294	0.5229	0.015*
O11	0.15866 (7)	0.80355 (14)	0.44880 (5)	0.0130 (2)
C11	0.13009 (11)	0.83794 (18)	0.38406 (7)	0.0127 (3)
O12	0.17813 (8)	0.91169 (15)	0.35614 (6)	0.0191 (2)
C111	0.03599 (11)	0.7745 (2)	0.35434 (8)	0.0142 (3)
C112	−0.00318 (12)	0.8198 (2)	0.29061 (8)	0.0183 (3)
H112	0.0285	0.8939	0.2693	0.022*
C113	−0.08877 (12)	0.7562 (2)	0.25838 (9)	0.0239 (4)
H113	−0.1155	0.7856	0.2147	0.029*
C114	−0.13509 (12)	0.6503 (2)	0.28971 (10)	0.0272 (4)
H114	−0.1934	0.6061	0.2673	0.033*
C115	−0.09723 (12)	0.6078 (2)	0.35362 (10)	0.0258 (4)
H115	−0.1300	0.5360	0.3750	0.031*
C116	−0.01135 (11)	0.6703 (2)	0.38637 (9)	0.0190 (3)
H116	0.0147	0.6420	0.4302	0.023*
N11	0.44244 (9)	0.61866 (17)	0.42862 (6)	0.0104 (2)
C12	0.40480 (10)	0.75381 (19)	0.45008 (7)	0.0114 (3)
H12	0.4357	0.8560	0.4610	0.014*
C13	0.31527 (10)	0.72040 (19)	0.45362 (7)	0.0105 (3)
C13A	0.29651 (10)	0.55208 (19)	0.43271 (7)	0.0098 (3)
C14	0.22226 (10)	0.4437 (2)	0.42943 (7)	0.0127 (3)
H14	0.1665	0.4805	0.4388	0.015*
C15	0.23190 (11)	0.2821 (2)	0.41223 (8)	0.0147 (3)
H15	0.1820	0.2076	0.4099	0.018*
C16	0.31388 (11)	0.2253 (2)	0.39810 (7)	0.0147 (3)
H16	0.3183	0.1134	0.3865	0.018*
C17	0.38813 (11)	0.32977 (19)	0.40090 (7)	0.0128 (3)
H17	0.4437	0.2921	0.3916	0.015*
C17A	0.37781 (10)	0.49289 (19)	0.41803 (7)	0.0100 (3)
C21	0.37140 (11)	0.95045 (19)	0.55964 (7)	0.0116 (3)
N21	0.44759 (9)	0.96982 (18)	0.58749 (7)	0.0165 (3)
C31	0.20907 (11)	0.99745 (19)	0.61958 (7)	0.0127 (3)
C32	0.14347 (11)	0.8950 (2)	0.63576 (8)	0.0159 (3)
Br32	0.03898 (2)	0.81786 (2)	0.57128 (2)	0.02400 (6)
C33	0.14907 (13)	0.8471 (2)	0.69864 (9)	0.0223 (4)

H33	0.1034	0.7763	0.7080	0.027*
C34	0.22217 (14)	0.9037 (2)	0.74773 (8)	0.0253 (4)
H34	0.2275	0.8703	0.7910	0.030*
C35	0.28693 (13)	1.0083 (2)	0.73362 (8)	0.0225 (4)
H35	0.3364	1.0485	0.7673	0.027*
C36	0.28043 (11)	1.0552 (2)	0.67051 (8)	0.0179 (3)
H36	0.3254	1.1283	0.6616	0.022*
C18	0.54006 (10)	0.5994 (2)	0.42926 (7)	0.0119 (3)
H18A	0.5652	0.5035	0.4565	0.014*
H18B	0.5743	0.6980	0.4496	0.014*
C181	0.55868 (10)	0.57590 (19)	0.36390 (7)	0.0105 (3)
C182	0.63057 (11)	0.4722 (2)	0.35846 (8)	0.0160 (3)
H182	0.6651	0.4142	0.3952	0.019*
C183	0.65196 (12)	0.4533 (2)	0.29938 (9)	0.0217 (4)
H183	0.7011	0.3822	0.2958	0.026*
C184	0.60197 (13)	0.5376 (2)	0.24594 (8)	0.0216 (4)
H184	0.6169	0.5249	0.2056	0.026*
C185	0.53023 (12)	0.6405 (2)	0.25104 (8)	0.0207 (3)
H185	0.4957	0.6980	0.2142	0.025*
C186	0.50856 (11)	0.6599 (2)	0.30995 (8)	0.0154 (3)
H186	0.4593	0.7309	0.3133	0.018*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0080 (6)	0.0117 (7)	0.0113 (6)	−0.0008 (5)	0.0017 (5)	0.0019 (5)
C2	0.0106 (6)	0.0112 (7)	0.0101 (6)	0.0008 (5)	0.0028 (5)	0.0012 (5)
C3	0.0134 (7)	0.0125 (7)	0.0116 (7)	0.0021 (6)	0.0036 (5)	0.0000 (5)
O11	0.0076 (5)	0.0175 (6)	0.0133 (5)	−0.0005 (4)	0.0012 (4)	−0.0021 (4)
C11	0.0131 (7)	0.0091 (7)	0.0139 (7)	0.0009 (5)	−0.0005 (5)	−0.0016 (5)
O12	0.0185 (6)	0.0183 (6)	0.0176 (6)	−0.0046 (5)	−0.0007 (5)	0.0040 (5)
C111	0.0104 (7)	0.0122 (7)	0.0180 (7)	0.0022 (5)	−0.0004 (6)	−0.0049 (6)
C112	0.0153 (7)	0.0199 (8)	0.0175 (8)	0.0036 (6)	−0.0002 (6)	−0.0054 (6)
C113	0.0159 (8)	0.0299 (10)	0.0214 (8)	0.0049 (7)	−0.0037 (6)	−0.0098 (7)
C114	0.0110 (8)	0.0288 (10)	0.0365 (11)	0.0003 (7)	−0.0034 (7)	−0.0127 (8)
C115	0.0132 (8)	0.0226 (9)	0.0398 (11)	−0.0030 (7)	0.0032 (7)	−0.0025 (8)
C116	0.0119 (7)	0.0182 (8)	0.0253 (8)	0.0008 (6)	0.0019 (6)	−0.0012 (7)
N11	0.0086 (6)	0.0133 (6)	0.0100 (6)	−0.0008 (5)	0.0034 (4)	−0.0013 (5)
C12	0.0125 (7)	0.0117 (7)	0.0108 (6)	−0.0008 (5)	0.0043 (5)	−0.0004 (5)
C13	0.0095 (6)	0.0124 (7)	0.0095 (6)	−0.0010 (5)	0.0020 (5)	−0.0003 (5)
C13A	0.0111 (6)	0.0116 (7)	0.0061 (6)	0.0005 (5)	0.0009 (5)	0.0003 (5)
C14	0.0109 (7)	0.0157 (7)	0.0113 (6)	−0.0009 (6)	0.0027 (5)	0.0000 (6)
C15	0.0160 (7)	0.0139 (7)	0.0142 (7)	−0.0038 (6)	0.0037 (6)	−0.0003 (6)
C16	0.0200 (8)	0.0114 (7)	0.0129 (7)	−0.0010 (6)	0.0043 (6)	−0.0012 (6)
C17	0.0151 (7)	0.0135 (7)	0.0103 (6)	0.0024 (6)	0.0040 (5)	−0.0005 (5)
C17A	0.0116 (6)	0.0126 (7)	0.0056 (6)	−0.0003 (5)	0.0019 (5)	0.0007 (5)
C21	0.0144 (7)	0.0098 (7)	0.0116 (7)	−0.0004 (5)	0.0053 (5)	−0.0012 (5)
N21	0.0135 (6)	0.0194 (7)	0.0164 (6)	−0.0005 (5)	0.0031 (5)	−0.0039 (5)

C31	0.0124 (7)	0.0136 (7)	0.0126 (7)	0.0041 (6)	0.0041 (5)	−0.0010 (6)
C32	0.0151 (7)	0.0165 (8)	0.0172 (7)	0.0029 (6)	0.0059 (6)	−0.0003 (6)
Br32	0.01663 (9)	0.02752 (10)	0.02782 (10)	−0.00654 (7)	0.00557 (7)	−0.00047 (7)
C33	0.0272 (9)	0.0222 (9)	0.0222 (8)	0.0060 (7)	0.0152 (7)	0.0040 (7)
C34	0.0354 (10)	0.0293 (10)	0.0134 (8)	0.0150 (8)	0.0105 (7)	0.0036 (7)
C35	0.0233 (9)	0.0296 (10)	0.0132 (7)	0.0114 (7)	0.0019 (6)	−0.0042 (7)
C36	0.0145 (7)	0.0227 (8)	0.0165 (7)	0.0037 (6)	0.0035 (6)	−0.0051 (6)
C18	0.0083 (6)	0.0179 (8)	0.0094 (6)	0.0009 (5)	0.0020 (5)	−0.0005 (5)
C181	0.0090 (6)	0.0123 (7)	0.0104 (6)	−0.0024 (5)	0.0029 (5)	−0.0014 (5)
C182	0.0136 (7)	0.0167 (8)	0.0187 (7)	0.0022 (6)	0.0061 (6)	0.0018 (6)
C183	0.0200 (8)	0.0218 (9)	0.0283 (9)	0.0004 (7)	0.0154 (7)	−0.0041 (7)
C184	0.0249 (9)	0.0283 (9)	0.0158 (7)	−0.0097 (7)	0.0129 (7)	−0.0066 (7)
C185	0.0201 (8)	0.0305 (9)	0.0116 (7)	−0.0042 (7)	0.0041 (6)	0.0027 (7)
C186	0.0122 (7)	0.0212 (8)	0.0128 (7)	0.0016 (6)	0.0032 (6)	0.0018 (6)

Geometric parameters (Å, º)

C1—C2	1.346 (2)	C15—C16	1.408 (2)
C1—O11	1.3942 (18)	C15—H15	0.9500
C1—C13	1.453 (2)	C16—C17	1.383 (2)
C2—C21	1.433 (2)	C16—H16	0.9500
C2—C3	1.518 (2)	C17—C17A	1.395 (2)
C3—C31	1.513 (2)	C17—H17	0.9500
C3—H3A	0.9900	C21—N21	1.151 (2)
C3—H3B	0.9900	C31—C32	1.392 (2)
O11—C11	1.3734 (19)	C31—C36	1.398 (2)
C11—O12	1.202 (2)	C32—C33	1.385 (2)
C11—C111	1.477 (2)	C32—Br32	1.9068 (17)
C111—C116	1.389 (2)	C33—C34	1.387 (3)
C111—C112	1.393 (2)	C33—H33	0.9500
C112—C113	1.387 (2)	C34—C35	1.375 (3)
C112—H112	0.9500	C34—H34	0.9500
C113—C114	1.379 (3)	C35—C36	1.385 (2)
C113—H113	0.9500	C35—H35	0.9500
C114—C115	1.387 (3)	C36—H36	0.9500
C114—H114	0.9500	C18—C181	1.507 (2)
C115—C116	1.390 (2)	C18—H18A	0.9900
C115—H115	0.9500	C18—H18B	0.9900
C116—H116	0.9500	C181—C186	1.387 (2)
N11—C12	1.365 (2)	C181—C182	1.390 (2)
N11—C17A	1.383 (2)	C182—C183	1.390 (2)
N11—C18	1.4581 (19)	C182—H182	0.9500
C12—C13	1.381 (2)	C183—C184	1.380 (3)
C12—H12	0.9500	C183—H183	0.9500
C13—C13A	1.445 (2)	C184—C185	1.382 (3)
C13A—C14	1.401 (2)	C184—H184	0.9500
C13A—C17A	1.410 (2)	C185—C186	1.389 (2)
C14—C15	1.381 (2)	C185—H185	0.9500

C14—H14	0.9500	C186—H186	0.9500
C2—C1—O11	117.13 (13)	C17—C16—C15	121.04 (15)
C2—C1—C13	127.35 (14)	C17—C16—H16	119.5
O11—C1—C13	115.19 (13)	C15—C16—H16	119.5
C1—C2—C21	117.79 (14)	C16—C17—C17A	117.09 (14)
C1—C2—C3	126.37 (14)	C16—C17—H17	121.5
C21—C2—C3	115.83 (13)	C17A—C17—H17	121.5
C31—C3—C2	110.94 (12)	N11—C17A—C17	128.89 (14)
C31—C3—H3A	109.5	N11—C17A—C13A	108.18 (13)
C2—C3—H3A	109.5	C17—C17A—C13A	122.75 (14)
C31—C3—H3B	109.5	N21—C21—C2	176.67 (17)
C2—C3—H3B	109.5	C32—C31—C36	116.67 (15)
H3A—C3—H3B	108.0	C32—C31—C3	123.79 (14)
C11—O11—C1	115.40 (12)	C36—C31—C3	119.50 (15)
O12—C11—O11	122.22 (14)	C33—C32—C31	122.49 (16)
O12—C11—C111	125.31 (15)	C33—C32—Br32	116.75 (13)
O11—C11—C111	112.47 (14)	C31—C32—Br32	120.75 (12)
C116—C111—C112	120.47 (15)	C32—C33—C34	119.17 (17)
C116—C111—C11	122.96 (15)	C32—C33—H33	120.4
C112—C111—C11	116.54 (15)	C34—C33—H33	120.4
C113—C112—C111	119.61 (18)	C35—C34—C33	119.83 (16)
C113—C112—H112	120.2	C35—C34—H34	120.1
C111—C112—H112	120.2	C33—C34—H34	120.1
C114—C113—C112	119.99 (18)	C34—C35—C36	120.35 (17)
C114—C113—H113	120.0	C34—C35—H35	119.8
C112—C113—H113	120.0	C36—C35—H35	119.8
C113—C114—C115	120.54 (17)	C35—C36—C31	121.45 (17)
C113—C114—H114	119.7	C35—C36—H36	119.3
C115—C114—H114	119.7	C31—C36—H36	119.3
C114—C115—C116	120.01 (18)	N11—C18—C181	114.95 (12)
C114—C115—H115	120.0	N11—C18—H18A	108.5
C116—C115—H115	120.0	C181—C18—H18A	108.5
C111—C116—C115	119.35 (17)	N11—C18—H18B	108.5
C111—C116—H116	120.3	C181—C18—H18B	108.5
C115—C116—H116	120.3	H18A—C18—H18B	107.5
C12—N11—C17A	108.82 (12)	C186—C181—C182	119.47 (14)
C12—N11—C18	125.19 (13)	C186—C181—C18	121.72 (14)
C17A—N11—C18	124.93 (13)	C182—C181—C18	118.76 (14)
N11—C12—C13	110.14 (14)	C181—C182—C183	120.12 (15)
N11—C12—H12	124.9	C181—C182—H182	119.9
C13—C12—H12	124.9	C183—C182—H182	119.9
C12—C13—C13A	106.43 (13)	C184—C183—C182	120.09 (16)
C12—C13—C1	127.59 (14)	C184—C183—H183	120.0
C13A—C13—C1	125.90 (13)	C182—C183—H183	120.0
C14—C13A—C17A	119.00 (14)	C183—C184—C185	120.01 (15)
C14—C13A—C13	134.36 (14)	C183—C184—H184	120.0
C17A—C13A—C13	106.43 (13)	C185—C184—H184	120.0

C15—C14—C13A	118.52 (14)	C184—C185—C186	120.15 (16)
C15—C14—H14	120.7	C184—C185—H185	119.9
C13A—C14—H14	120.7	C186—C185—H185	119.9
C14—C15—C16	121.59 (15)	C181—C186—C185	120.15 (15)
C14—C15—H15	119.2	C181—C186—H186	119.9
C16—C15—H15	119.2	C185—C186—H186	119.9
O11—C1—C2—C21	174.10 (13)	C14—C15—C16—C17	−0.1 (2)
C13—C1—C2—C21	1.0 (2)	C15—C16—C17—C17A	−0.2 (2)
O11—C1—C2—C3	−4.8 (2)	C12—N11—C17A—C17	−174.54 (14)
C13—C1—C2—C3	−177.92 (14)	C18—N11—C17A—C17	−5.8 (2)
C1—C2—C3—C31	115.94 (17)	C12—N11—C17A—C13A	0.67 (16)
C21—C2—C3—C31	−63.03 (17)	C18—N11—C17A—C13A	169.37 (13)
C2—C1—O11—C11	121.15 (15)	C16—C17—C17A—N11	175.16 (15)
C13—C1—O11—C11	−64.94 (17)	C16—C17—C17A—C13A	0.6 (2)
C1—O11—C11—O12	−13.0 (2)	C14—C13A—C17A—N11	−176.22 (13)
C1—O11—C11—C111	166.92 (12)	C13—C13A—C17A—N11	−0.69 (16)
O12—C11—C111—C116	170.58 (16)	C14—C13A—C17A—C17	−0.7 (2)
O11—C11—C111—C116	−9.4 (2)	C13—C13A—C17A—C17	174.88 (14)
O12—C11—C111—C112	−7.3 (2)	C2—C3—C31—C32	−98.04 (18)
O11—C11—C111—C112	172.79 (14)	C2—C3—C31—C36	79.71 (18)
C116—C111—C112—C113	−2.0 (2)	C36—C31—C32—C33	−2.0 (2)
C11—C111—C112—C113	175.86 (15)	C3—C31—C32—C33	175.78 (15)
C111—C112—C113—C114	0.8 (3)	C36—C31—C32—Br32	176.51 (12)
C112—C113—C114—C115	0.6 (3)	C3—C31—C32—Br32	−5.7 (2)
C113—C114—C115—C116	−0.9 (3)	C31—C32—C33—C34	0.5 (3)
C112—C111—C116—C115	1.8 (2)	Br32—C32—C33—C34	−178.09 (13)
C11—C111—C116—C115	−175.96 (16)	C32—C33—C34—C35	1.1 (3)
C114—C115—C116—C111	−0.4 (3)	C33—C34—C35—C36	−1.1 (3)
C17A—N11—C12—C13	−0.38 (17)	C34—C35—C36—C31	−0.5 (3)
C18—N11—C12—C13	−169.05 (13)	C32—C31—C36—C35	2.0 (2)
N11—C12—C13—C13A	−0.05 (17)	C3—C31—C36—C35	−175.87 (15)
N11—C12—C13—C1	176.84 (14)	C12—N11—C18—C181	−119.05 (16)
C2—C1—C13—C12	−36.3 (3)	C17A—N11—C18—C181	74.06 (19)
O11—C1—C13—C12	150.51 (15)	N11—C18—C181—C186	38.1 (2)
C2—C1—C13—C13A	140.01 (16)	N11—C18—C181—C182	−144.58 (15)
O11—C1—C13—C13A	−33.2 (2)	C186—C181—C182—C183	0.1 (2)
C12—C13—C13A—C14	174.99 (16)	C18—C181—C182—C183	−177.33 (15)
C1—C13—C13A—C14	−2.0 (3)	C181—C182—C183—C184	0.1 (3)
C12—C13—C13A—C17A	0.45 (16)	C182—C183—C184—C185	−0.3 (3)
C1—C13—C13A—C17A	−176.50 (14)	C183—C184—C185—C186	0.3 (3)
C17A—C13A—C14—C15	0.4 (2)	C182—C181—C186—C185	−0.1 (2)
C13—C13A—C14—C15	−173.65 (16)	C18—C181—C186—C185	177.27 (15)
C13A—C14—C15—C16	0.0 (2)	C184—C185—C186—C181	−0.1 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C36—H36···*Cg*3^{i}	0.95	2.92	3.739 (2)	145
C183—H183···*Cg*1^{ii}	0.95	2.97	3.807 (2)	148

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

2-Benzoyl-3-(2-bromophenyl)acrylonitrile (V)

Crystal data

$C_{16}H_{10}BrNO$
M_r = 312.15
Monoclinic, $P2_1/n$
a = 10.986 (4) Å
b = 8.977 (4) Å
c = 13.209 (5) Å
β = 97.140 (17)°
V = 1292.6 (9) Å^3
Z = 4
$F(000)$ = 624
D_x = 1.604 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2986 reflections
θ = 2.3–27.6°
μ = 3.17 mm^{-1}
T = 100 K
Block, orange
0.27 × 0.26 × 0.23 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} = 0.366, T_{max} = 0.482
30048 measured reflections
2985 independent reflections
2754 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 27.6°, θ_{min} = 2.3°
h = −14→14
k = −11→11
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.053
S = 1.11
2985 reflections
172 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.023P)^2 + 0.9733P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 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}
C1	0.52114 (14)	0.09372 (17)	0.66805 (12)	0.0114 (3)
N1	0.46836 (14)	0.03411 (16)	0.72541 (11)	0.0179 (3)
C2	0.59444 (13)	0.16873 (17)	0.60003 (11)	0.0092 (3)

C3	0.58208 (13)	0.31481 (17)	0.57997 (11)	0.0087 (3)
H3	0.6362	0.3569	0.5369	0.010*
C27	0.67937 (14)	0.06881 (17)	0.55029 (11)	0.0096 (3)
O27	0.65240 (11)	−0.06180 (13)	0.53670 (9)	0.0158 (2)
C21	0.79280 (14)	0.13384 (16)	0.51742 (11)	0.0098 (3)
C22	0.83791 (15)	0.07181 (18)	0.43254 (12)	0.0132 (3)
H22	0.7956	−0.0083	0.3967	0.016*
C23	0.94443 (16)	0.1274 (2)	0.40076 (13)	0.0176 (3)
H23	0.9746	0.0862	0.3425	0.021*
C24	1.00734 (15)	0.2433 (2)	0.45368 (13)	0.0190 (3)
H24	1.0804	0.2811	0.4316	0.023*
C25	0.96375 (15)	0.30402 (19)	0.53877 (13)	0.0158 (3)
H25	1.0072	0.3828	0.5751	0.019*
C26	0.85669 (14)	0.24971 (17)	0.57080 (11)	0.0115 (3)
H26	0.8268	0.2914	0.6291	0.014*
C31	0.49311 (14)	0.41605 (16)	0.61805 (11)	0.0091 (3)
C32	0.53014 (14)	0.55567 (17)	0.65733 (11)	0.0102 (3)
Br32	0.69900 (2)	0.60654 (2)	0.67042 (2)	0.01496 (6)
C33	0.44831 (15)	0.65635 (18)	0.69096 (12)	0.0140 (3)
H33	0.4756	0.7510	0.7168	0.017*
C34	0.32568 (15)	0.61617 (19)	0.68605 (12)	0.0155 (3)
H34	0.2684	0.6836	0.7091	0.019*
C35	0.28618 (14)	0.47790 (19)	0.64766 (12)	0.0152 (3)
H35	0.2022	0.4510	0.6450	0.018*
C36	0.36892 (14)	0.37893 (17)	0.61324 (12)	0.0122 (3)
H36	0.3410	0.2852	0.5862	0.015*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0104 (7)	0.0090 (7)	0.0152 (7)	0.0003 (5)	0.0025 (6)	−0.0013 (6)
N1	0.0202 (7)	0.0143 (7)	0.0210 (7)	−0.0010 (6)	0.0093 (6)	0.0022 (6)
C2	0.0079 (6)	0.0104 (7)	0.0095 (6)	−0.0018 (5)	0.0017 (5)	−0.0012 (5)
C3	0.0077 (6)	0.0113 (7)	0.0071 (6)	−0.0017 (5)	0.0014 (5)	−0.0019 (5)
C27	0.0108 (7)	0.0103 (7)	0.0076 (6)	0.0010 (5)	−0.0001 (5)	0.0002 (5)
O27	0.0186 (6)	0.0097 (5)	0.0197 (6)	−0.0017 (4)	0.0047 (5)	−0.0031 (4)
C21	0.0091 (7)	0.0105 (7)	0.0097 (7)	0.0024 (5)	0.0015 (5)	0.0017 (5)
C22	0.0152 (7)	0.0135 (7)	0.0111 (7)	0.0037 (6)	0.0024 (6)	−0.0001 (6)
C23	0.0183 (8)	0.0240 (9)	0.0121 (7)	0.0060 (7)	0.0073 (6)	0.0017 (6)
C24	0.0121 (7)	0.0270 (9)	0.0192 (8)	−0.0003 (7)	0.0068 (6)	0.0063 (7)
C25	0.0118 (7)	0.0181 (8)	0.0174 (8)	−0.0028 (6)	0.0012 (6)	0.0005 (6)
C26	0.0115 (7)	0.0128 (7)	0.0103 (7)	0.0011 (6)	0.0019 (5)	0.0005 (6)
C31	0.0106 (7)	0.0099 (7)	0.0069 (6)	0.0013 (5)	0.0019 (5)	0.0013 (5)
C32	0.0111 (7)	0.0115 (7)	0.0081 (6)	−0.0004 (5)	0.0010 (5)	0.0014 (5)
Br32	0.01375 (9)	0.01610 (9)	0.01532 (9)	−0.00647 (6)	0.00294 (6)	−0.00398 (6)
C33	0.0215 (8)	0.0103 (7)	0.0105 (7)	0.0024 (6)	0.0028 (6)	−0.0003 (6)
C34	0.0172 (8)	0.0185 (8)	0.0113 (7)	0.0102 (6)	0.0038 (6)	0.0021 (6)
C35	0.0097 (7)	0.0224 (8)	0.0139 (7)	0.0025 (6)	0.0023 (6)	0.0018 (6)

C36	0.0113 (7)	0.0141 (7)	0.0113 (7)	−0.0007 (6)	0.0016 (6)	0.0004 (6)

Geometric parameters (Å, º)

C1—N1	1.143 (2)	C24—H24	0.9500
C1—C2	1.445 (2)	C25—C26	1.387 (2)
C2—C3	1.341 (2)	C25—H25	0.9500
C2—C27	1.503 (2)	C26—H26	0.9500
C3—C31	1.469 (2)	C31—C32	1.398 (2)
C3—H3	0.9500	C31—C36	1.398 (2)
C27—O27	1.217 (2)	C32—C33	1.386 (2)
C27—C21	1.489 (2)	C32—Br32	1.8973 (17)
C21—C26	1.396 (2)	C33—C34	1.388 (2)
C21—C22	1.397 (2)	C33—H33	0.9500
C22—C23	1.385 (2)	C34—C35	1.390 (2)
C22—H22	0.9500	C34—H34	0.9500
C23—C24	1.389 (3)	C35—C36	1.387 (2)
C23—H23	0.9500	C35—H35	0.9500
C24—C25	1.387 (2)	C36—H36	0.9500
N1—C1—C2	176.52 (17)	C24—C25—H25	120.0
C3—C2—C1	121.85 (14)	C26—C25—H25	120.0
C3—C2—C27	123.38 (13)	C25—C26—C21	120.01 (15)
C1—C2—C27	114.70 (13)	C25—C26—H26	120.0
C2—C3—C31	126.35 (14)	C21—C26—H26	120.0
C2—C3—H3	116.8	C32—C31—C36	117.91 (14)
C31—C3—H3	116.8	C32—C31—C3	120.45 (14)
O27—C27—C21	122.14 (14)	C36—C31—C3	121.61 (14)
O27—C27—C2	119.10 (14)	C33—C32—C31	122.22 (15)
C21—C27—C2	118.75 (13)	C33—C32—Br32	118.65 (12)
C26—C21—C22	119.75 (14)	C31—C32—Br32	119.07 (11)
C26—C21—C27	122.10 (14)	C32—C33—C34	118.67 (15)
C22—C21—C27	118.10 (14)	C32—C33—H33	120.7
C23—C22—C21	119.81 (15)	C34—C33—H33	120.7
C23—C22—H22	120.1	C33—C34—C35	120.42 (15)
C21—C22—H22	120.1	C33—C34—H34	119.8
C22—C23—C24	120.27 (15)	C35—C34—H34	119.8
C22—C23—H23	119.9	C36—C35—C34	120.26 (15)
C24—C23—H23	119.9	C36—C35—H35	119.9
C25—C24—C23	120.14 (15)	C34—C35—H35	119.9
C25—C24—H24	119.9	C35—C36—C31	120.52 (15)
C23—C24—H24	119.9	C35—C36—H36	119.7
C24—C25—C26	120.02 (16)	C31—C36—H36	119.7
C1—C2—C3—C31	−2.8 (2)	C22—C21—C26—C25	0.8 (2)
C27—C2—C3—C31	173.86 (14)	C27—C21—C26—C25	178.30 (14)
C3—C2—C27—O27	−149.43 (15)	C2—C3—C31—C32	134.36 (16)
C1—C2—C27—O27	27.4 (2)	C2—C3—C31—C36	−47.8 (2)

C3—C2—C27—C21	29.4 (2)	C36—C31—C32—C33	0.0 (2)
C1—C2—C27—C21	−153.71 (14)	C3—C31—C32—C33	177.87 (14)
O27—C27—C21—C26	−147.87 (15)	C36—C31—C32—Br32	177.34 (11)
C2—C27—C21—C26	33.3 (2)	C3—C31—C32—Br32	−4.77 (19)
O27—C27—C21—C22	29.7 (2)	C31—C32—C33—C34	0.5 (2)
C2—C27—C21—C22	−149.12 (14)	Br32—C32—C33—C34	−176.85 (11)
C26—C21—C22—C23	−1.2 (2)	C32—C33—C34—C35	−0.3 (2)
C27—C21—C22—C23	−178.82 (14)	C33—C34—C35—C36	−0.4 (2)
C21—C22—C23—C24	0.8 (2)	C34—C35—C36—C31	0.9 (2)
C22—C23—C24—C25	0.0 (3)	C32—C31—C36—C35	−0.7 (2)
C23—C24—C25—C26	−0.4 (3)	C3—C31—C36—C35	−178.56 (14)
C24—C25—C26—C21	0.0 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C33—H33···N1^{i}	0.95	2.55	3.425 (3)	154
C36—H36···O27^{ii}	0.95	2.59	3.459 (2)	153
C3—H3···*Cg*1^{iii}	0.95	2.73	3.431 (2)	131
C23—H23···*Cg*1^{iv}	0.95	2.69	3.506 (2)	144

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