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Synthesis of *N*-substituted 3-(2-aryl-2-oxoethyl)-3-hydroxyindolin-2-ones and their conversion to *N*-substituted (*E*)-3-(2-aryl-2-oxoethylidene)-indolin-2-ones: synthetic sequence, spectroscopic characterization and structures of four 3-hydroxy compounds and five oxoethylidene products

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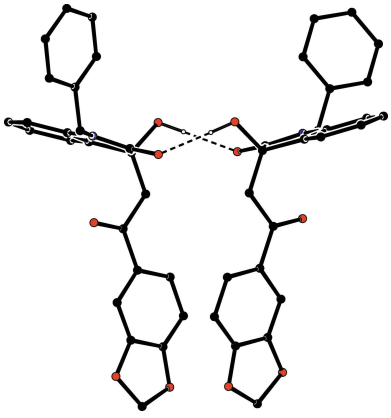
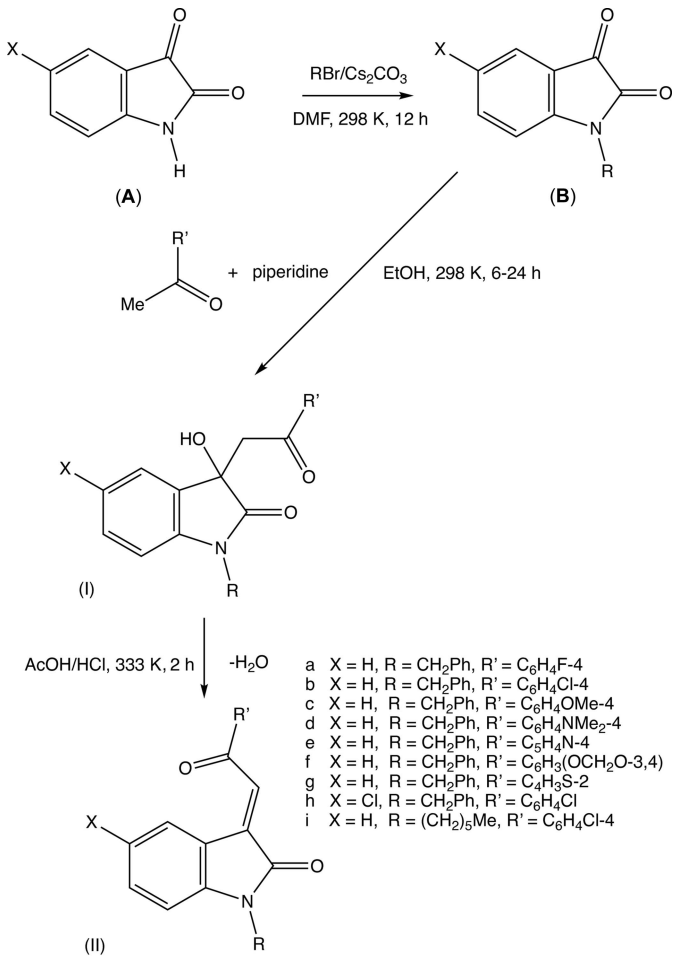
An operationally simple and time-efficient approach has been developed for the synthesis of racemic *N*-substituted 3-(2-aryl-2-oxoethyl)-3-hydroxyindolin-2-ones by a piperidine-catalysed aldol reaction between aryl methyl ketones and *N*-alkylisatins. These aldol products were used successfully as strategic intermediates for the preparation of *N*-substituted (*E*)-3-(2-hetaryl-2-oxoethylidene)indolin-2-ones by a stereoselective dehydration reaction under acidic conditions. The products have all been fully characterized by ^{1}H and ^{13}C NMR spectroscopy, by mass spectrometry and, for a representative selection, by crystal structure analysis. In each of (*RS*)-1-benzyl-3-hydroxy-3-[2-(4-methoxyphenyl)-2-oxoethyl]indolin-2-one, $C_{24}H_{21}NO_4$, (I*c*), and (*RS*)-1-benzyl-3-{2-[4-(dimethylamino)phenyl]-2-oxoethyl}-3-hydroxyindolin-2-one, $C_{25}H_{24}N_2O_3$, (I*d*), inversion-related pairs of molecules are linked by O—H···O hydrogen bonds to form $R_2^2(10)$ rings, which are further linked into chains of rings by a combination of C—H···O and C—H···π(arene) hydrogen bonds in (I*c*) and by C—H···π(arene) hydrogen bonds in (I*d*). The molecules of (*RS*)-1-benzyl-3-hydroxy-3-[2-oxo-2-(pyridin-4-yl)ethyl]indolin-2-one, $C_{22}H_{18}N_2O_3$, (I*e*), are linked into a three-dimensional framework structure by a combination of O—H···N, C—H···O and C—H···π(arene) hydrogen bonds. (*RS*)-3-[2-(Benzo[*d*][1,3]dioxol-5-yl)-2-oxoethyl]-1-benzyl-3-hydroxyindolin-2-one, $C_{24}H_{19}NO_5$, (I*f*), crystallizes with Z' = 2 in the space group $P\overline{1}$ and the molecules are linked into complex sheets by a combination of O—H···O, C—H···O and C—H···π(arene) hydrogen bonds. In each of (*E*)-1-benzyl-3-[2-(4-fluorophenyl)-2-oxoethylidene]indolin-2-one, $C_{23}H_{16}FNO_2$, (II*a*), and (*E*)-1-benzyl-3-[2-oxo-2-(thiophen-2-yl)ethylidene]-indolin-2-one, $C_{21}H_{15}NO_2S$, (II*g*), the molecules are linked into simple chains by a single C—H···O hydrogen bond, while those of (*E*)-1-benzyl-3-[2-oxo-2-(pyridin-4-yl)ethylidene]indolin-2-one, $C_{22}H_{16}N_2O_2$, (II*e*), are linked by three C—H···O hydrogen bonds to form sheets which are further linked into a three-dimensional structure by C—H···π(arene) hydrogen bonds. There are no hydrogen bonds in the structures of either (*E*)-1-benzyl-3-[2-(4-methoxyphenyl)-2-oxoethylidene]-indolin-2-one, $C_{24}H_{19}NO_3$, (II*c*), or (*E*)-1-benzyl-5-chloro-3-[2-(4-chlorophenyl)-2-oxoethylidene]indolin-2-one, $C_{23}H_{15}Cl_2NO_2$, (II*h*), but the molecules of (II*h*) are linked into chains of π-stacked dimers by a combination of C—Cl···π(arene) and aromatic π–π stacking interactions.



1. Introduction

Almost 60% of drugs based on small organic molecules which are in use for medicinal purposes contain at least one

N-heterocyclic ring (Vitaku *et al.*, 2014). Amongst these, isatin (1*H*-indole-2,3-dione) and its derivatives have attracted



Scheme 1

particular interest because of their broad range of biological and pharmacological activities (Singh & Desta, 2012; Pakravan *et al.*, 2013). Isatin derivatives have also been found to be useful synthetic intermediates for the production of both dyestuffs and organic electronic materials (Stalder *et al.*, 2014; Deng & Zhang, 2014). These wide-ranging applications have prompted the development of a large range of synthetic approaches to functionalized isatin derivatives (Moradi *et al.*, 2017; Bogdanov & Mironov, 2018; Varun *et al.*, 2019). Amongst these, the addition of nucleophilic units to the prochiral carbonyl group at atom C3 permits the construction of chiral 3-substituted-3-hydroxyindolin-2-ones containing a stereogenic centre at the 3-position (Peddibhotla, 2009; Mohammadi *et al.*, 2013). Such species are desirable targets, because many related structural motifs are found in natural products and pharmaceutically active compounds; for example, convolutamydine A is a bioactive alkaloid with significant activity against HL-60 human plomyelocytic leukemia cells (Kamano *et al.*, 1995), SM-130686 is a novel orally active growth hormone secretagogue (Nagamine *et al.*, 2001), donaxaridine has shown effective anticancer properties (Kimura *et al.*, 2016) and maremycins A and B exhibit antibacterial, antifungal and antitumour properties (Duan *et al.*, 2018).

Several years ago, we reported the synthesis and structures of a range of 3-alkyl-3-hydroxyindolin-2-ones by reaction of isatin itself with a variety of methyl ketones in the presence of piperidine. Although the procedures were straightforward, the yields were consistently rather disappointing, in the range 40–60% (Becerra *et al.*, 2010). Within the isatin molecule, both simple amide and vinylogous amide fragments can be identified, so that in the conjugate base of isatin the negative charge can be delocalized into both carbonyl groups. Any consequent partial proton transfer from isatin to piperidine is thus likely to be a factor in depressing the overall yields. We therefore reasoned that incorporation of a substituent at the N atom of isatin should prevent any such ionization and thus increase the product yields significantly.

Accordingly, we have now studied the synthesis and structures of a range of 3-(2-aryl-2-oxoethyl)-3-hydroxyindolin-2-ones, (I), and their dehydration to the corresponding chalcones, (II) (see Scheme 1), and we report here the synthesis and spectroscopic characterization of nine compounds of type (I), and eight of type (II), along with the molecular and supramolecular structures of four representative type (I) compounds [(I*c*), (I*d*), (I*e*) and (I*f*)] and of five representative type (II) compounds [(II*a*), (II*c*), (II*e*), (II*g*) and (II*h*)].

2. Experimental

2.1. Synthesis and crystallization

Isatins (**A**) (see Scheme 1), where *X* = H or Cl, were converted to the corresponding *N*-alkyl analogues (**B**) by reaction with the appropriate alkyl bromide in dimethylformamide solution in the presence of solid caesium carbonate acting as a weak base, giving yields in excess of 90% after a reaction time of 12 h at 298 K. For the synthesis of the 3-hydroxyindolin-2-ones, (I) (see Scheme 1), a mixture of an *N*-alkylisatin, (**B**) (1.0 mmol), the appropriate aryl methyl ketone (1.0 mmol) and piperidine (0.2 mmol) in ethanol (10 ml) was stirred at 298 K for 6 h [24 h in the case of compound (I*d*)], after which time the starting materials were no longer detectable using thin-layer chromatography (TLC). The resulting solid products were collected by filtration, washed with cold ethanol (2 ml) and dried in air to give the products of type (I). Analytical data for compound (I*a*): yield 93%, m.p. 437–438 K (literature 437–441 K; Tripathi *et al.*, 2016); compound (I*b*): yield 91%, m.p. 430–431 K (literature 431–433 K; Duan *et al.*, 2013); compound (I*c*): yield 85%, m.p. 430 K (literature 429–431 K; Duan *et al.*, 2013); compound (I*d*): yield 36%, m.p. 450 K; compound (I*e*): yield 92%, m.p. 479–481 K; compound (I*f*): yield 87%, m.p. 452–453 K; compound (I*g*): yield 82%, m.p. 421–422 K (literature 421–423 K; Satish *et al.*, 2015); compound (I*h*): yield 89%, m.p. 423 K; compound (I*i*): yield 83%, m.p. 398 K. Colourless crystals of compounds (I*c*), (I*d*), (I*e*) and (I*f*) suitable for single-crystal X-ray diffraction analysis were grown by slow evaporation, at ambient temperature and in the presence of air, of solutions in ethanol–dimethylformamide (6:1 *v*/*v*).

For the conversion of the 3-hydroxy compounds (I) into the ethylidene products (II), a solution of the appropriate 3-hy-

Table 1
Experimental details.

Experiments were carried out at 100 K with Mo $K\alpha$ radiation using a Bruker D8 Venture diffractometer. Absorption was corrected for by multi-scan methods (*SADABS*; Bruker, 2016), except for (II*a*) and (II*e*), where *TWINABS* (Bruker, 2012) was used.

	(Ic)	(Id)	(Ie)
Crystal data			
Chemical formula	$C_{24}H_{21}NO_4$	$C_{25}H_{24}N_2O_3$	$C_{22}H_{18}N_2O_3$
M_r	387.42	400.46	358.38
Crystal system, space group	Monoclinic, $C2/c$	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$
a, b, c (Å)	18.7572 (14), 13.2095 (10), 16.750 (2)	9.1028 (8), 10.6434 (9), 11.2539 (10)	7.8838 (5), 10.1766 (8), 11.8719 (9)
α, β, γ (°)	90, 105.374 (5), 90	88.988 (3), 68.422 (3), 81.352 (3)	87.554 (3), 75.996 (2), 69.428 (2)
V (Å^3)	4001.7 (6)	1001.55 (15)	864.30 (11)
Z	8	2	2
μ (mm^{-1})	0.09	0.09	0.09
Crystal size (mm)	0.14 × 0.13 × 0.10	0.23 × 0.19 × 0.16	0.25 × 0.16 × 0.06
Data collection			
T_{min}, T_{max}	0.908, 0.991	0.946, 0.986	0.949, 0.994
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	18506, 4768, 3469	41110, 4590, 4033	55488, 4333, 3760
R_{int}	0.051	0.037	0.045
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.659	0.650	0.670
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.047, 0.110, 1.02	0.038, 0.098, 1.05	0.038, 0.101, 1.07
No. of reflections	4768	4590	4333
No. of parameters	266	276	247
No. of restraints	0	0	0
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.30, −0.26	0.30, −0.22	0.41, −0.22

	(If)	(II*a*)	(II*c*)
Crystal data			
Chemical formula	$C_{24}H_{19}NO_5$	$C_{23}H_{16}FNO_2$	$C_{24}H_{19}NO_3$
M_r	401.40	357.37	369.40
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/n$
a, b, c (Å)	11.8136 (7), 12.4987 (10), 13.5976 (11)	7.6021 (6), 20.4880 (13), 10.9319 (7)	4.9743 (2), 29.1957 (13), 12.4406 (6)
α, β, γ (°)	93.084 (3), 101.883 (2), 95.055 (2)	90, 96.986 (3), 90	90, 100.914 (2), 90
V (Å^3)	1951.7 (3)	1690.0 (2)	1774.05 (14)
Z	4	4	4
μ (mm^{-1})	0.10	0.10	0.09
Crystal size (mm)	0.25 × 0.16 × 0.06	0.14 × 0.14 × 0.10	0.45 × 0.06 × 0.04
Data collection			
T_{min}, T_{max}	0.957, 0.994	0.917, 0.990	0.948, 0.996
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	125792, 9691, 8134	3896, 3896, 2995	54500, 4142, 3643
R_{int}	0.049	N/A	0.048
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.667	0.652	0.653
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.039, 0.106, 1.08	0.058, 0.134, 1.05	0.037, 0.095, 1.06
No. of reflections	9691	3896	4142
No. of parameters	547	245	254
No. of restraints	0	0	0
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.38, −0.24	0.29, −0.28	0.27, −0.23

	(II*e*)	(II*g*)	(II*h*)
Crystal data			
Chemical formula	$C_{22}H_{16}N_2O_2$	$C_{21}H_{15}NO_2S$	$C_{23}H_{15}Cl_2NO_2$
M_r	340.37	345.40	408.26
Crystal system, space group	Monoclinic, $P2_1/n$	Orthorhombic, $Pbca$	Triclinic, $P\overline{1}$
a, b, c (Å)	7.3457 (6), 18.0675 (16), 13.1813 (13)	17.5058 (14), 8.8163 (6), 21.2092 (16)	8.2010 (6), 9.7629 (7), 12.1740 (9)

Table 1 (continued)

	(II*e*)	(II*g*)	(II*h*)
α, β, γ (°)	90, 105.994 (3), 90	90, 90, 90	76.755 (3), 87.675 (3), 76.211 (3)
V (Å^3)	1681.7 (3)	3273.4 (4)	921.34 (12)
Z	4	8	2
μ (mm^{-1})	0.09	0.21	0.37
Crystal size (mm)	0.16 × 0.15 × 0.12	0.15 × 0.07 × 0.05	0.40 × 0.16 × 0.07
Data collection			
T_{min}, T_{max}	0.878, 0.990	0.942, 0.989	0.924, 0.974
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	4167, 4167, 3281	32005, 4154, 3280	51788, 4558, 3867
R_{int}	N/A	0.065	0.053
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.668	0.672	0.667
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.044, 0.121, 1.09	0.038, 0.092, 1.04	0.032, 0.082, 1.11
No. of reflections	4167	4154	4558
No. of parameters	237	239	253
No. of restraints	0	10	0
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.29, −0.19	0.29, −0.31	0.44, −0.34

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

droxy compound (I) (0.50 mmol) in glacial acetic acid (2 ml) was stirred at ambient temperature for 10 min, during which time concentrated hydrochloric acid (0.1 ml) was added slowly. The resulting mixtures were then stirred at 333 K for a further 2 h. For each mixture, the pH was then brought to 7.0 by the addition of a concentrated aqueous solution of ammonia, and the resulting solid products were collected by filtration, washed with cold water and dried in air to give the products of type (II). Analytical data for compound (II*a*): yield 92%, m.p. 425–426 K; compound (II*b*): yield 85%, m.p. 417–418 K; compound (II*c*): yield 93%, m.p. 393 K; compound (II*e*): yield 97%, m.p. 398 K; compound (II*f*): yield 88%, m.p. 388 K; compound (II*g*): yield 91%, m.p. 388–389 K; compound (II*h*): yield 89%, m.p. 401–403 K; compound (II*i*): yield 83%, m.p. 376–378 K. Crystals of compounds (II*a*) (orange), and (II*b*), (II*c*), (II*e*) and (II*g*) (all red) suitable for single-crystal X-ray diffraction analysis were grown by slow evaporation, at ambient temperature and in the presence of air, of solutions in ethanol–dimethylformamide (initial composition 6:1 *v*/*v*).

Spectroscopic data for compounds (I*a*), (I*b*), (I*c*) and (I*g*) have been reported recently in the literature (Duan *et al.*, 2013; Satish *et al.*, 2015; Tripathi *et al.*, 2016). Spectroscopic characterization data (^{1}H and ^{13}C NMR, and mass spectra) for the other compounds reported here are provided in the supporting information.

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. Compound (II*a*) was handled as a non-merohedral twin with the twin matrix ($\bar{1}00$ $0\bar{1}0$ 0.350,0,1) and with refined twin fractions of 0.1953 (14) and 0.8047 (14). Compound (II*e*) was also handled as a non-merohedral twin, with the twin matrix ($\bar{1}00$ $0\bar{1}0$ 0.989,0,1) and refined twin fractions of 0.9654 (6) and 0.0346 (6). In compound (II*g*), the thiophene unit is disordered over two sets of atomic sites having unequal occupancies for the minor-disorder component, and the bonded and [1,2]-nonbonded distances were restrained to be the same as the corresponding distances in the major-disorder component, subject to s.u. values of 0.01 and 0.02 Å, respectively; in addition, the anisotropic displacement parameters of pairs of partial-occupancy atoms occupying essentially the same physical space were constrained to be equal. All H atoms, apart from those in the minor-disorder component of compound (II*g*), were located in difference maps. H atoms bonded to C atoms were then treated as riding atoms in geometrically idealized positions, with C—H = 0.95 (alkenyl, aryl and heteroaryl), 0.98 (CH_3) or 0.99 Å (CH_2) and with U_{iso}(H) = kU_{eq}(C), where k = 1.5 for the methyl groups, which were permitted to rotate but not to tilt, and 1.2 for all other H atoms bonded to C atoms; the H atoms in the minor-disorder component of compound (II*g*) were included on the same basis, giving refined disorder occupancies of 0.9387 (19) and 0.0613 (19). For the H atoms bonded to O atoms, the atomic coordinates were refined with U_{iso}(H) = 1.5U_{eq}(O), giving the O—H distances shown in Table 2.

3. Results and discussion

The title compounds were synthesized starting from the readily available isatins (**A**), (see Scheme 1, where X = H or Cl; Figs. 1–9). The *N*-alkylation of the starting isatins was explored using both benzyl bromide and 1-hexyl bromide in the presence of caesium carbonate as a weak non-nucleophilic base, giving isolated yields of the *N*-alkyl intermediates (**B**) consistently in excess of 90%. Focusing primarily on the N-benzyl intermediate of type (**B**), the subsequent reactions with aryl methyl ketones in the presence of piperidine did indeed provide generally much higher yields of the products of type (I), usually well above 80%, than had previously been achieved using isatin carrying no substituent at the N atom,

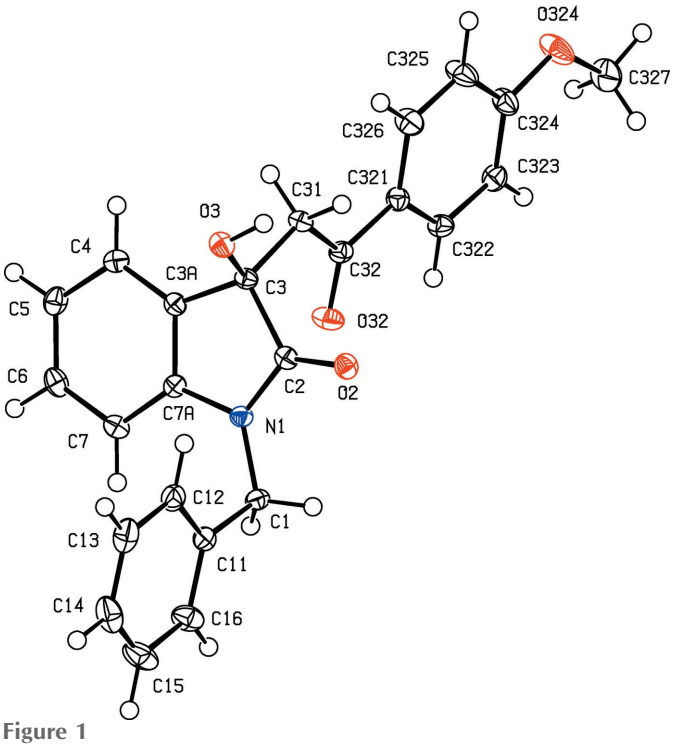


Figure 1
The molecular structure of the *R* enantiomer of compound (I*c*), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

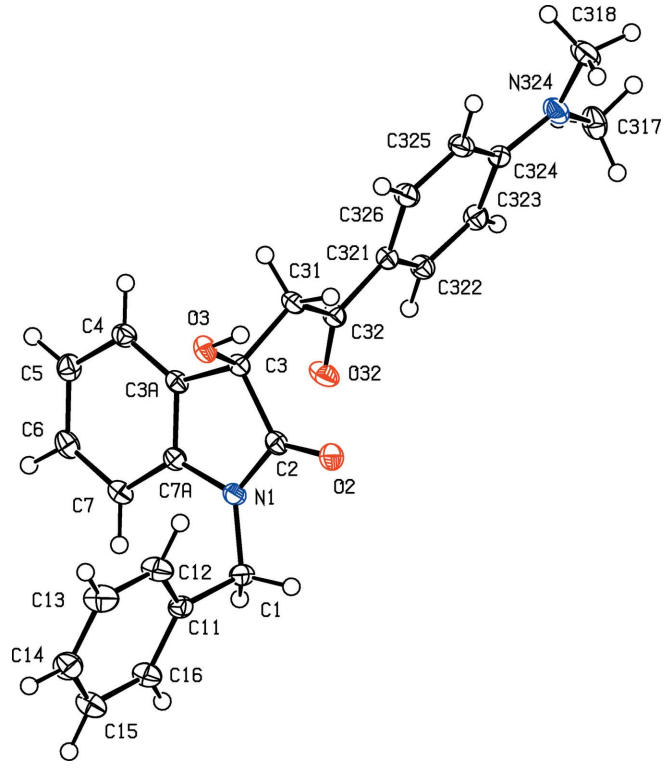


Figure 2
The molecular structure of the *R* enantiomer of compound (I*d*), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

which is consistent with the idea of partial proton transfer from the *N*-unsubstituted isatin to piperidine. The yields in both steps appear to be much the same regardless of whether the substituent at the N atom is benzyl or 1-hexyl, or whether the substituent at C5 is H or Cl. The only exception was found for compound (I*d*), where the yield was quite low, 36%, even after a much longer reaction time than that required for all the other type (I) products; this may be associated with the strongly electron-donating nature of the dimethylamino group. Acid-catalysed dehydration of nine of products (I) gave the *N*-substituted (*E*)-3-(2-aryl-2-oxoethylidene)indolin-2-ones (II), again with yields well above 80%, although, because of the slow formation and poor yields of (I*d*) in the first step, the dehydration of this intermediate was not pursued.

For all of the products of types (I) and (II) (see Scheme 1), the mass spectra confirm their overall compositions, while the NMR spectra contain all of the expected signals, thus confirming that all the reactions have proceeded as expected and confirming the identity of the products. Each of the aldol products of type (I) contains a stereogenic centre, at atom C3 in (I*c*), (I*d*) and (I*e*), and at atoms C13 and C23 in the two independent molecules in (I*f*). In every case, the reference molecule was selected as one having the *R* configuration at this atom, but the space groups (Table 1) confirm that, in each case, the compound has crystallized as a racemic mixture; in the absence from the synthesis of any agent capable of inducing enantioselectivity, it can confidently assumed that all of the other products of type (I) are also formed as racemic mixtures. For each of the type (II) products, only a single geometric isomer was isolated, with no chromatographic or spectroscopic evidence for the formation of even traces of any second isomer. As well as confirming the identity and racemic nature of the type (I) products, the crystal structure analyses have

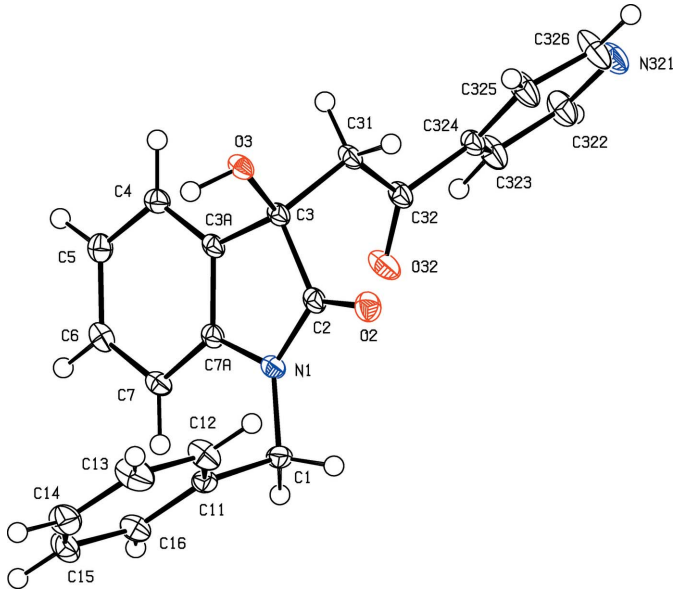


Figure 3
The molecular structure of the *R* enantiomer of compound (I*e*), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

Table 2
Hydrogen bonds and related short intramolecular contacts (Å, °) for compounds (I*c*)–(I*f*), (II*a*), (II*e*) and (II*g*).

*Cg*1, *Cg*2 and *Cg*3 represent the centroids of the C3*A*/C4–C7/C7*A*, C11–C16 and C13*A*/C14–C17/C17*A* rings, respectively.

	D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
(I*c*)	O3—H3···O2^{i}	0.86 (2)	2.10 (2)	2.9487 (15)	171.1 (18)
	C6—H6···O324^{ii}	0.95	2.41	3.297 (2)	155
	C31—H31*B*···O2^{i}	0.99	2.48	3.312 (2)	141
	C4—H4···*Cg*2^{iii}	0.95	2.93	3.6101 (18)	130
	C14—H14···*Cg*1^{iv}	0.95	2.82	3.709 (2)	156
(I*d*)	O3—H3···O2^{v}	0.868 (18)	1.918 (18)	2.7630 (12)	164.2 (18)
	C7—H7···O32^{vi}	0.95	2.44	3.3343 (16)	157
	C1—H1*B*···*Cg*1^{vi}	0.99	2.96	3.8375 (14)	149
(I*e*)	O3—H3···N321^{vii}	0.897 (17)	1.897 (17)	2.7915 (14)	174.9 (15)
	C4—H4···O3^{v}	0.95	2.46	3.3842 (14)	164
	C7—H7···O32^{viii}	0.95	2.51	3.3719 (14)	150
	C325—H325···O2^{ix}	0.95	2.32	3.2578 (15)	171
	C322—H322···*Cg*1^{ii}	0.95	2.68	3.5294 (14)	149
(I*f*)	O13—H13···O22	0.874 (17)	1.912 (17)	2.7794 (12)	171.1 (17)
	O23—H23···O12	0.874 (17)	1.912 (17)	2.7794 (12)	171.1 (17)
	C131–H13*A*···O22^{vi}	0.99	2.35	3.3075 (16)	161
	C147—H147···O141^{x}	0.95	2.56	3.4776 (18)	163
	C231—H23*A*···O12^{v}	0.99	2.37	3.3107 (15)	159
	C242—H24*A*···O23^{ii}	0.99	2.53	3.4889 (19)	163
	C142—H14*A*···*Cg*3^{ii}	0.99	2.53	3.3289 (15)	137
(II*a*)	C15—H15···O32^{xi}	0.95	2.49	3.278 (3)	141
(II*e*)	C14—H14···O2^{xii}	0.95	2.32	3.234 (3)	161
	C16—H16···O2^{xiii}	0.95	2.45	3.230 (2)	140
	C326—H326···O2^{xiv}	0.95	2.58	3.494 (2)	162
	C6—H6···*Cg*2^{xiii}	0.95	2.64	3.566 (2)	165
(II*g*)	C5—H5···O2^{xv}	0.95	2.59	3.5058 (19)	161
	C323—H323···*Cg*2^{xvi}	0.95	2.93	3.744 (3)	145

Symmetry codes: (i) $-x+\frac{3}{2}, -y+\frac{3}{2}, -z+1$; (ii) $x, y-1, z$; (iii) $x, -y+1, z+\frac{1}{2}$; (iv) $-x+\frac{3}{2}, -y+\frac{1}{2}, -z+1$; (v) $-x+1, -y+1, -z+1$; (vi) $-x, -y+1, -z+2$; (vii) $x, y+1, z$; (viii) $-x+1, -y+1, -z$; (ix) $-x, -y+1, -z+1$; (x) $-x, -y, -z+1$; (xi) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (xii) $x+\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$; (xiii) $x-\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$; (xiv) $x+\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$; (xv) $x-\frac{1}{2}, y, -z+\frac{1}{2}$; (xvi) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$.

established that in each of the products of type (II) examined here the chalcone unit has the *E* configuration.

In each of the aldol compounds of type (I) (Figs. 1–4), the orientation of the *N*-benzyl unit relative to the indolinone nucleus shows some variation, as indicated by the key torsion angles (Table 3), despite the fact that atoms from the benzyl unit participate in intermolecular hydrogen bonding only in aldol (I*c*) (Table 2). Similar variations in the orientation of the benzyl group are found in the chalcones of type (II) (Table 4), where this unit participates in intermolecular hydrogen bonding in both (II*a*) and (II*e*), but not in any of (II*c*), (II*g*) and (II*h*). On the other hand, the conformations of the rest of the molecule relative to the indolinone unit is broadly similar in both type (I) and type (II) compounds, aside from the pyridyl derivative (I*e*). In each of 4-methoxy derivatives (I*c*) and (II*c*), the two exocyclic C—C—O angles at atom C324 (Figs. 1 and 6) differ by *ca* 10°, as typically found (Seip & Seip, 1973; Ferguson *et al.*, 1996) for near-planar alkoxyarene units; the deviations of methyl atoms C327 from the planes of the adjacent aryl rings are only 0.112 (3) and 0.183 (2) Å in (I*c*) and (II*c*), respectively.

The molecules of compound (I*c*) are linked into a chain of rings by a combination of O—H···O, C—H···O and C—H···π(arene) hydrogen bonds (Table 2). Inversion-related pairs of molecules are linked by pairs of O—H···O hydrogen bonds, forming an $R_2^2(10)$ ring (Etter, 1990; Etter *et al.*, 1990; Bernstein *et al.*, 1995) centred at $(\frac{3}{4}, \frac{3}{4}, \frac{1}{2})$. A second centrosymmetric motif, now centred at $(\frac{3}{4}, \frac{1}{4}, \frac{1}{2})$, is generated by the C—H···π(arene) hydrogen bond having atom C14 as the donor and, in combination, these two motifs generate a chain of rings running parallel to the [010] direction, in which the $R_2^2(10)$ rings centred at $(\frac{3}{4}, \frac{3}{4}+n, \frac{1}{2})$ alternate with the rings generated by C—H···π hydrogen bonds, centred at $(\frac{3}{4}, \frac{1}{4}+n, \frac{1}{2})$, where n represents an integer in each case. The chain

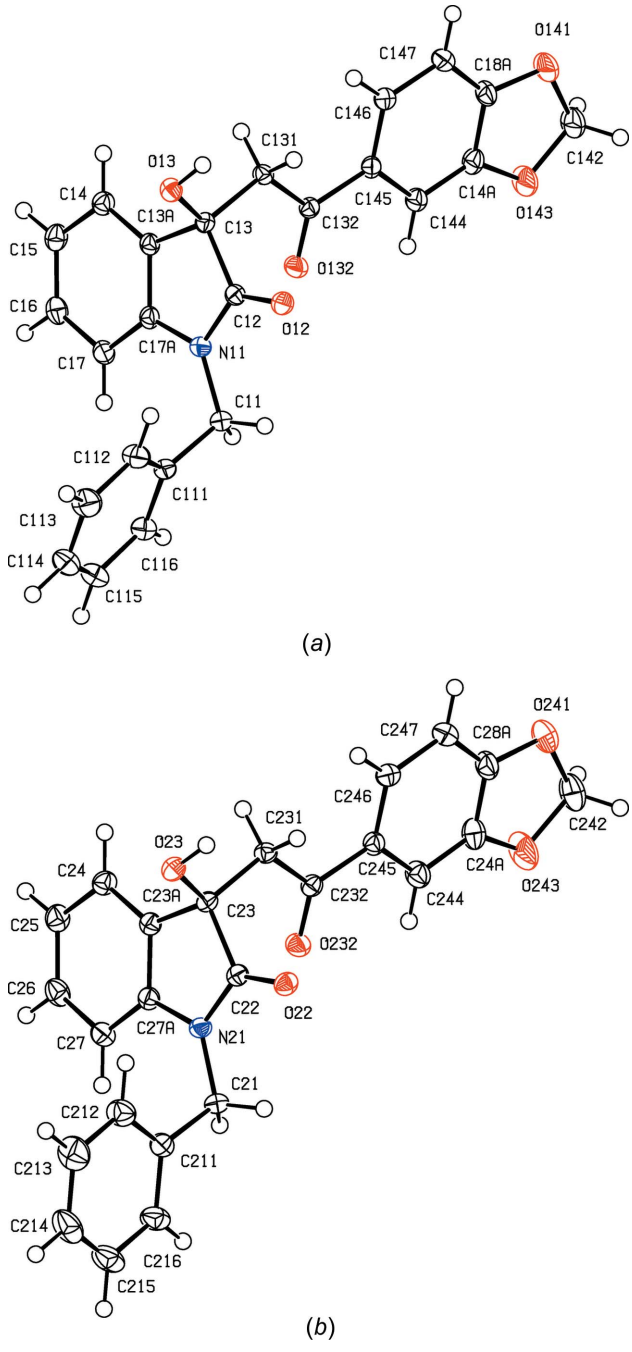


Figure 4
The *R* enantiomers of the two independent molecules of compound (I*f*), showing the atom-labelling schemes for (*a*) molecule 1 and (*b*) molecule 2. Displacement ellipsoids are drawn at the 30% probability level.

Table 3
Selected torsion angles (°) for compounds (I*c*)–(I*f*).

Parameter	(I*c*)	(I*d*)	(I*e*)	(I*f*), molecule 1 (x = 1)	(I*f*), molecule 2 (x = 2)
C*x*2—N*x*1—C*x*1—C*x*11	103.75 (16)	102.09 (12)	95.90 (12)	119.11 (13)	108.60 (13)
N*x*1—C*x*1—C*x*11—C*x*12	−28.5 (2)	−40.32 (15)	−76.98 (13)	−54.19 (16)	−27.57 (17)
N*x*1—C*x*2—C*x*3—C*x*31	−126.14 (13)	−133.07 (10)	−124.96 (9)	−123.72 (10)	−126.04 (10)
C*x*2—C*x*3—C*x*31—C*x*32	52.57 (17)	58.79 (13)	61.83 (12)	50.92 (13)	51.93 (13)
C3—C31—C32—C321	−176.52 (13)	179.12 (10)			
C3—C31—C32—C324			−179.45 (10)		
C31—C32—C321—C322	175.64 (14)	−176.22 (10)			
C31—C32—C324—C323			−149.73 (12)		
C*x*3—C*x*31—C*x*32—C*x*45				−174.30 (10)	−174.80 (10)
C*x*31—C*x*32—C*x*45—C*x*44				176.61 (10)	178.65 (11)

Table 4
Selected torsion angles (°) for compounds (II*a*), (II*c*), (II*e*), (II*g*) and (II*h*).

Parameter	(II*a*)	(II*c*)	(II*e*)	(II*g*)	(II*h*)
C2—N1—C1—C11	111.4 (2)	102.92 (13)	90.35 (19)	94.94 (16)	98.83 (15)
N1—C1—C11—C12	−41.8 (3)	−61.57 (14)	−1.1 (2)	−65.13 (18)	−62.18 (16)
N1—C2—C3—C31	−176.39 (19)	−177.72 (10)	179.61 (15)	177.30 (12)	−179.37 (12)
C2—C3—C31—C32	176.1 (2)	178.62 (11)	−178.71 (16)	178.91 (13)	177.52 (13)
C3—C31—C32—C321	−176.0 (2)	172.77 (11)			177.57 (14)
C3—C31—C32—C322				−175.99 (14)	
C3—C31—C32—C324			179.55 (16)		
C31—C32—C321—C322	−178.2 (2)	169.40 (10)			175.80 (12)
C31—C32—C322—S321				167.55 (10)	
C31—C32—C324—C323			173.76 (15)		

formation is augmented by a C—H···O hydrogen bond having atom C6 as the donor and linking molecules which are related by translation into a $C(13)$ chain motif (Fig. 10). There are two other short intermolecular contacts in the structure, involving atoms C4 and C31, but these are unlikely to be of real structural significance (Wood *et al.*, 2009).

The supramolecular assembly in compound (I*d*) is very simple, taking the form of a chain of rings running parallel to the [10$\bar{1}$] direction (Fig. 11). Rings of $R_2^2(10)$ type, containing O—H···O hydrogen bonds (Table 2) and centred at $(\frac{1}{2}+n, \frac{1}{2}, \frac{1}{2}-n)$ alternate with rings of $R_2^2(16)$ type, containing C—H···O hydrogen bonds and centred at $(n, \frac{1}{2}, \frac{1}{2}-n)$, where n represents an integer in each case. There is a long C—H···π(arene) contact within the chain, but there are no direction-specific interactions between adjacent chains.

In contrast to the simplicity of the assembly in 4-(dimethylamino)phenyl compound (I*d*), that in 4-pyridine derivative (I*e*) takes the form of the three-dimensional framework structure built from O—H···N, C—H···π(arene) and multiple C—H···O hydrogen bonds

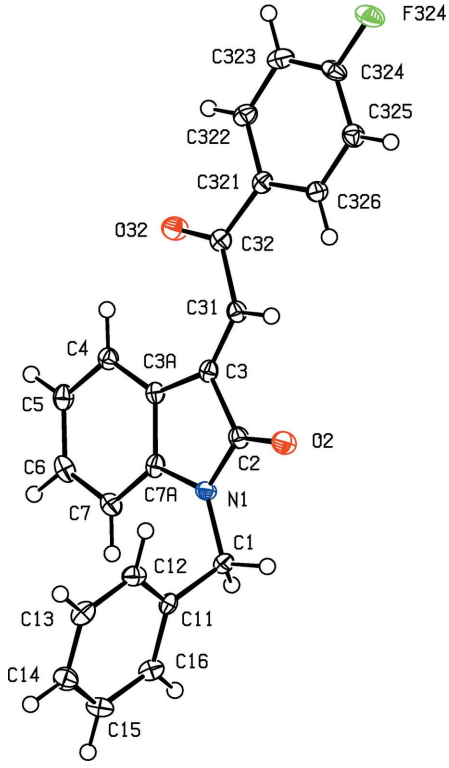


Figure 5
The molecular structure of compound (II*a*), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

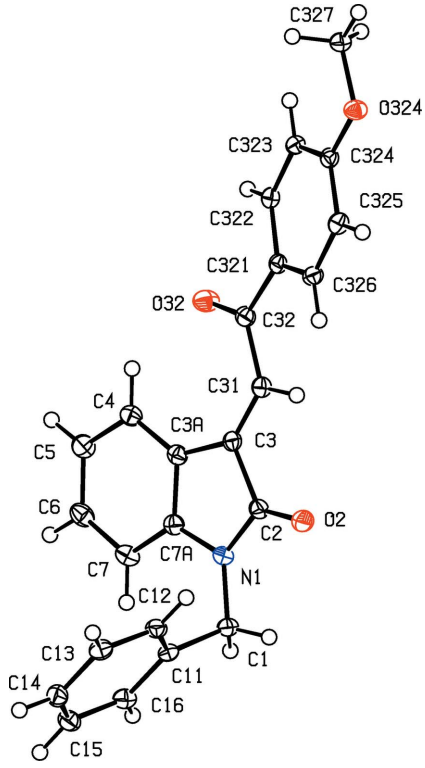


Figure 6
The molecular structure of compound (II*c*), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

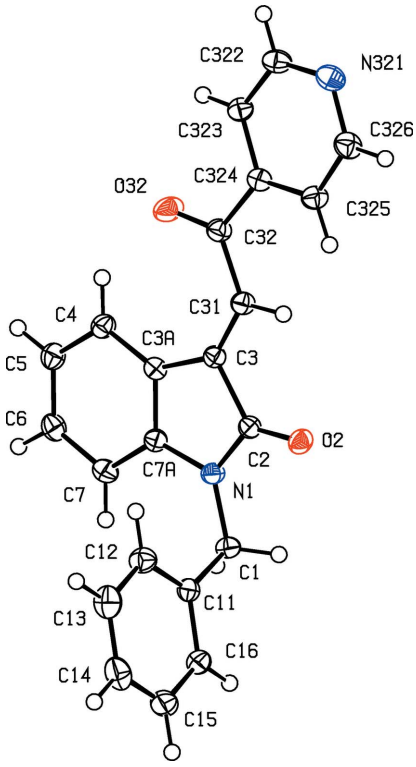


Figure 7
The molecular structure of compound (II*e*), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

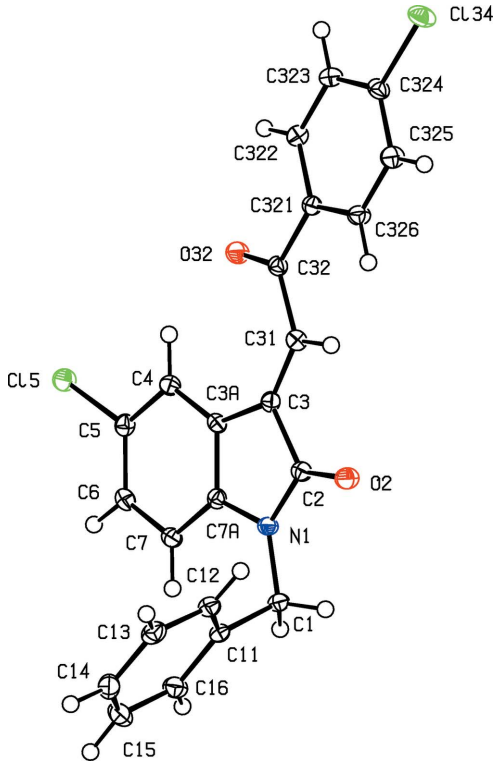


Figure 9
The molecular structure of compound (II*h*), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

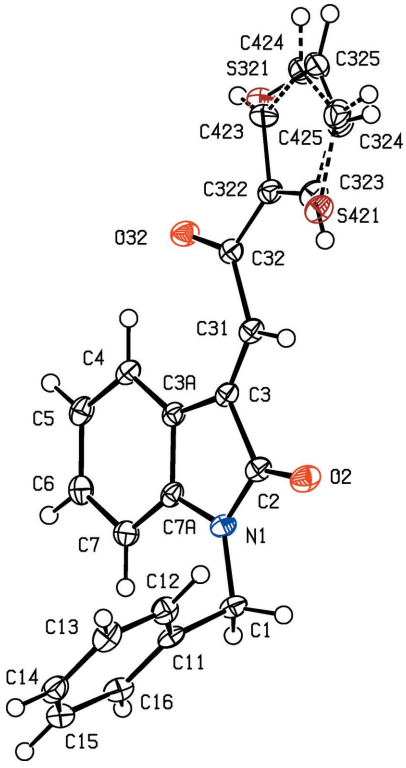


Figure 8
The molecular structure of compound (II*g*), showing the atom-labelling scheme and the disorder of the thiophene unit. The major-disorder component is drawn using full lines and the minor-disorder component has been drawn using broken lines. Displacement ellipsoids are drawn at the 30% probability level.

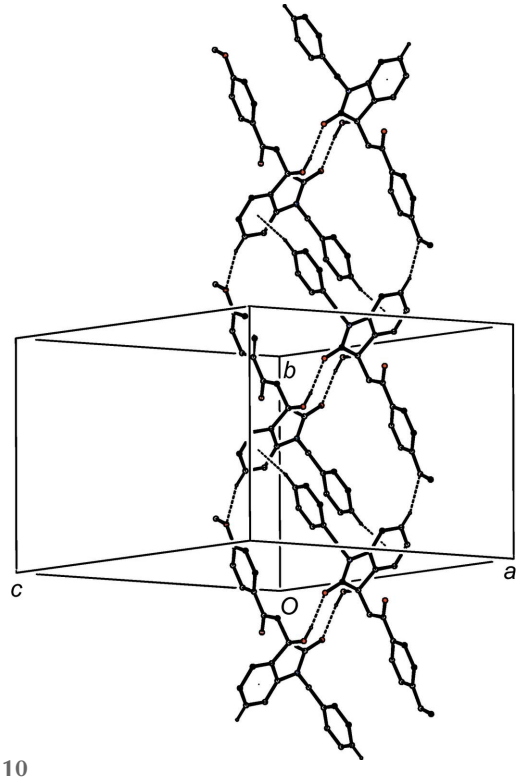


Figure 10
Part of the crystal structure of compound (I*c*), showing the formation of a chain of rings built from O—H···O, C—H···O and C—H···π(arene) hydrogen bonds. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms not involved in the motifs shown have been omitted.

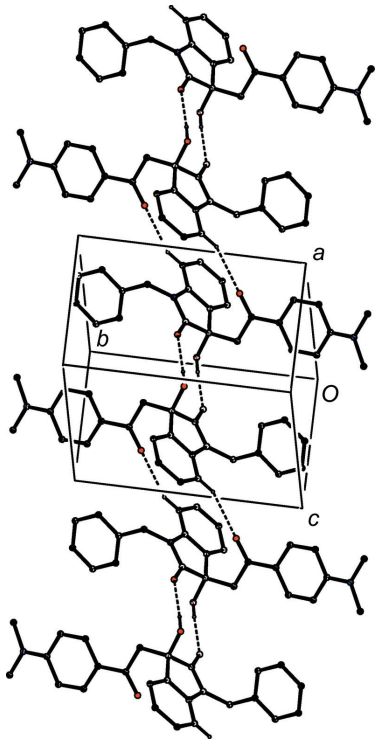


Figure 11
Part of the crystal structure of compound (I*d*), showing the formation of a chain of rings built from O—H···O and C—H···O hydrogen bonds. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms not involved in the motifs shown have been omitted.

(Table 2), but the formation of the framework structure is readily analysed in terms of three one-dimensional substructures (Ferguson *et al.*, 1998*a*,*b*; Gregson *et al.*, 2000). A combination of O—H···N and C—H···π(arene) hydrogen bonds links molecules which are related by translation into a chain of rings running parallel to the [010] direction (Fig. 12). In the second substructure, a combination of the two hydrogen bonds having atoms C4 and C7 as the donors generates a chain of centrosymmetric rings running parallel to the [001] direction, in which $R_2^2(10)$ rings centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2} + n)$ alternate with $R_2^2(16)$ rings centred at $(\frac{1}{2}, \frac{1}{2}, n)$, where n represents an integer (Fig. 13). In the final substructure, the combination of the two hydrogen bonds having atoms C4 and C322 as the donors generates a chain of centrosymmetric rings running parallel to the [100] direction, in which $R_2^2(10)$ rings centred at $(\frac{1}{2} + n, \frac{1}{2}, \frac{1}{2})$ alternate with $R_2^2(16)$ rings centred at $(n, \frac{1}{2}, n)$, where n represents an integer (Fig. 14). The combination of chains along [100], [010] and [001] suffices to generate the three-dimensional framework structure.

Compound (I*f*) crystallizes with two molecules in the asymmetric unit but, despite this and the resulting number of independent hydrogen bonds (Table 2), the supramolecular assembly in only two-dimensional and, as with (I*e*), this can be analysed in terms of low-dimensional substructures. The two molecules within the selected asymmetric unit (Fig. 15) are linked by two O—H···O hydrogen bonds to form a dimeric unit having approximate, but noncrystallographic, twofold

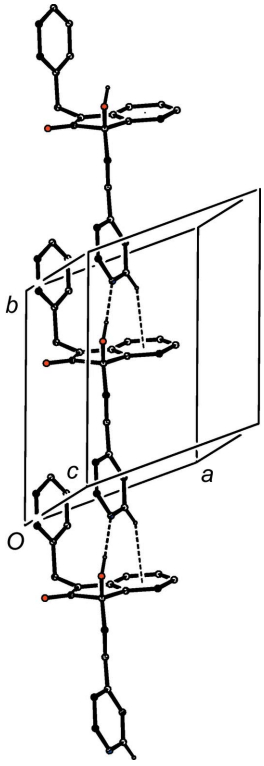


Figure 12
Part of the crystal structure of compound (I*e*), showing the formation of a chain of rings running parallel to the [010] direction and built from O—H···N and C—H···π(arene) hydrogen bonds. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms not involved in the motifs shown have been omitted.

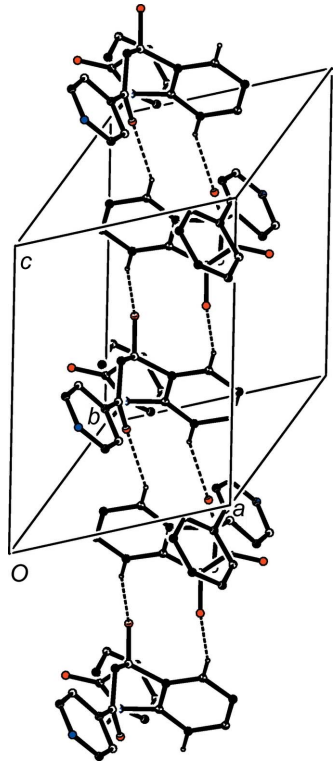


Figure 13
Part of the crystal structure of compound (I*e*), showing the formation of a chain of rings running parallel to the [001] direction and built from two types of C—H···O hydrogen bonds. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms not involved in the motifs shown have been omitted.

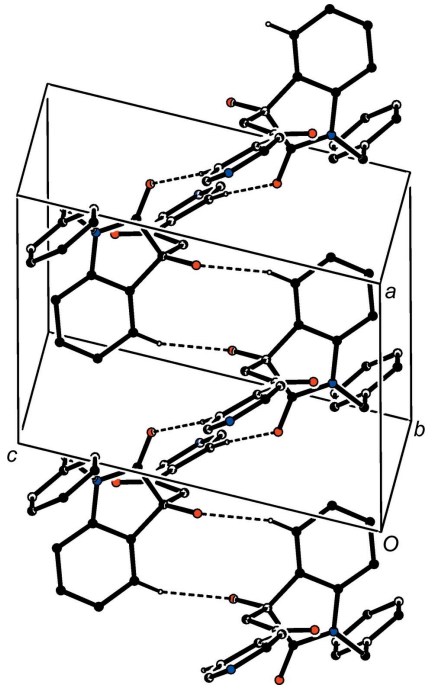


Figure 14
Part of the crystal structure of compound (I*e*), showing the formation of a chain of rings running parallel to the [100] direction and built from two types of C—H···O hydrogen bonds. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms not involved in the motifs shown have been omitted.

rotation symmetry (Fig. 15). This finite, or zero-dimensional, substructure can conveniently be regarded as the basic building block in the supramolecular assembly. These dimeric units are linked by the C—H···O hydrogen bonds having atoms C131 and C231 as the donors to form a chain of rings running parallel to the [100] direction, in which $R_4^4(12)$ rings centred at $(n, \frac{1}{2}, \frac{1}{2})$ alternate with $R_4^2(12)$ rings centred at $(\frac{1}{2}+n, \frac{1}{2}, \frac{1}{2})$, where n represents an integer in each case (Fig. 16). A second one-dimensional substructure arises from the linking of dimeric units which are related by translation by a combination of C—H···O and C—H···π(arene) hydrogen bonds to form a second chain of rings, this time running parallel to the [010] direction (Fig. 17). The combination of chains along [100] and [010] gives rise to complex sheets lying parallel to (001), but there are not direction-specific interactions between adjacent sheets.

The absence of hydroxy groups in the compounds of type (II) means that the hydrogen bonding in these structures is simpler than that found in compounds of type (I). Thus, for each of compounds (II*c*) and (II*h*), there are no significant intermolecular hydrogen bonds, while in compound (II*a*), a single C—H···O hydrogen bond (Table 2) links molecules which are related by a 2_1 screw axis into a $C(11)$ chain running parallel to the [010] direction (Fig. 18). The hydrogen bonding in compound (II*g*) is likewise very simple, with a single C—H···O hydrogen bond linking molecules which are related by the *a*-glide plane at $z = \frac{1}{4}$ to form a $C(7)$ chain running parallel to the [100] direction (Fig. 19).

A combination of three independent C—H···O hydrogen bonds links the molecules of compound (II*e*) into a sheet lying

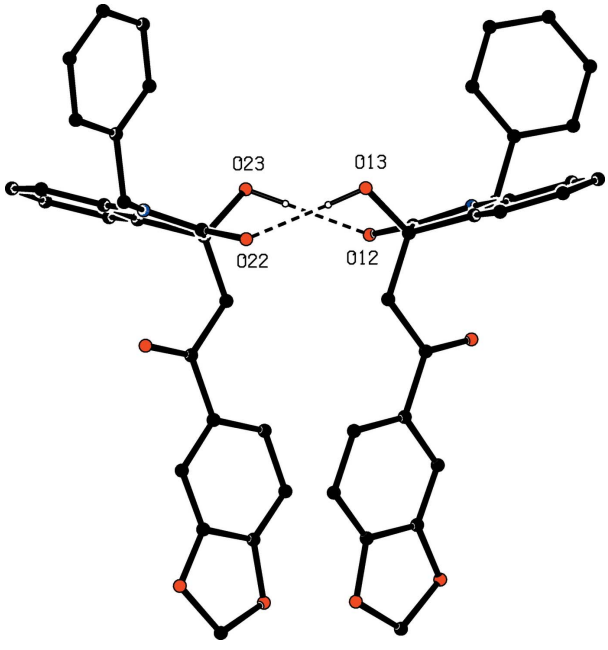


Figure 15
Part of the crystal structure of compound (I*f*), showing the linking of the two independent molecules by two independent O—H···O hydrogen bonds. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms not involved in the motifs shown have been omitted.

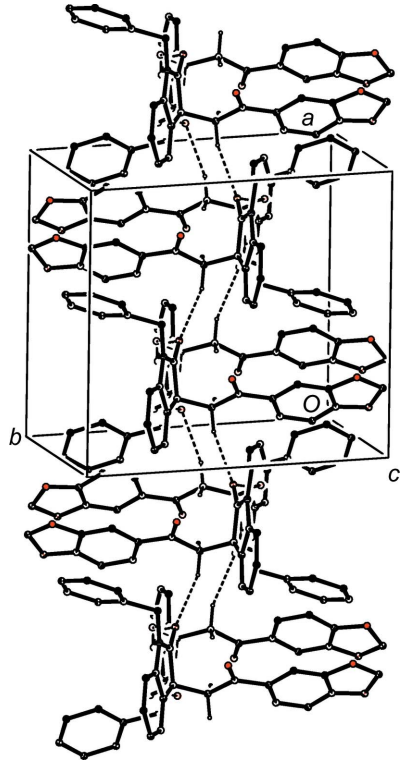


Figure 16
Part of the crystal structure of compound (I*f*), showing the formation of a chain of rings running parallel to the [100] direction and built from O—H···O and C—H···O hydrogen bonds. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms not involved in the motifs shown have been omitted.

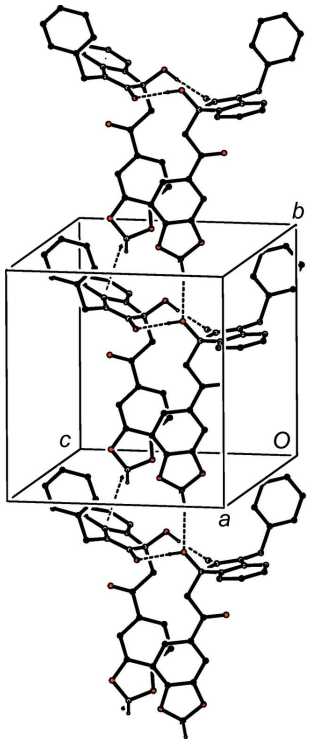


Figure 17
Part of the crystal structure of compound (I*f*), showing the formation of a chain of rings running parallel to the [010] direction and built from O—H···O, C—H···O and C—H···π(arene) hydrogen bonds. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms not involved in the motifs shown have been omitted.

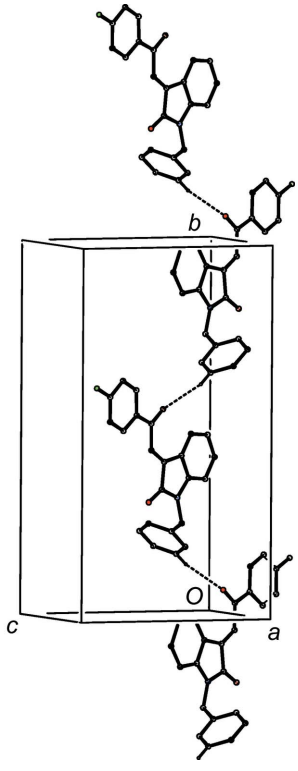


Figure 18
Part of the crystal structure of compound (II*a*), showing the formation of a hydrogen-bonded chain running parallel to [010]. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms not involved in the motif shown have been omitted.

parallel to (010) and lying in the domain $\frac{1}{2} < y < 1.0$ (Fig. 20); a second sheet, related to the first by inversion, lies in the domain $0 < y < \frac{1}{2}$, and adjacent sheets are linked by a C—H···π(arene) hydrogen bond, so generating a single three-dimensional framework structure.

Although there are no hydrogen bonds in the structure of compound (II*h*), the molecules are nonetheless linked into a chain by the combination of a C—Cl···π(arene) interaction and a π–π stacking interaction. In the first of these interactions, atom Cl34 in the molecule at (x, y, z) forms a short contact with the C3*A*/C4–C7/C7*A* ring in the molecule at ($-x$, $-y + 1$, $-z + 1$), with geometric parameters Cl···*Cg* = 3.6055 (8) Å and C—Cl···*Cg* = 88.71 (5)°, where *Cg* represents the centroid of the aryl ring. The Cl···*Cg* distance here may be compared with the average value of 2.6° deduced from a database analysis of such contacts (Imai *et al.*, 2008), and this interaction generates a cyclic centrosymmetric dimer. In addition, the C3*A*/C4–C7/C7*A* ring at (x, y, z) and the C321–C326 ring at ($-x + 1$, $-y + 1$, $-z + 1$) make a dihedral angle of only 7.58 (7)°. The ring-centroid separation is 3.7374 (9) Å and the shortest perpendicular distance from the centroid of one ring to the plane of the other is 3.3592 (6) Å, corresponding to a ring-centroid offset of *ca* 1.64 Å. The combination of these two interactions thus generates a chain of π-stacked dimers lying parallel to the [100] direction (Fig. 21).

The synthetic methodology described here is notable for its operational simplicity, broad substrate scope, good functional

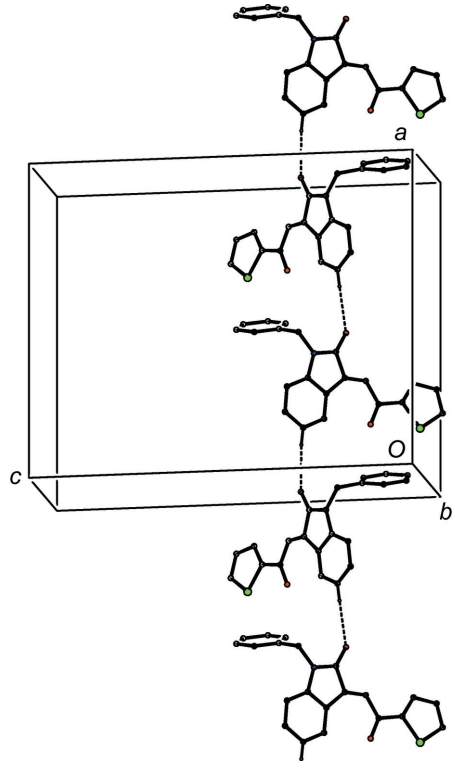


Figure 19
Part of the crystal structure of compound (II*g*), showing the formation of a hydrogen-bonded chain running parallel to [100]. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, the minor-disorder component and H atoms not involved in the motif shown have been omitted.

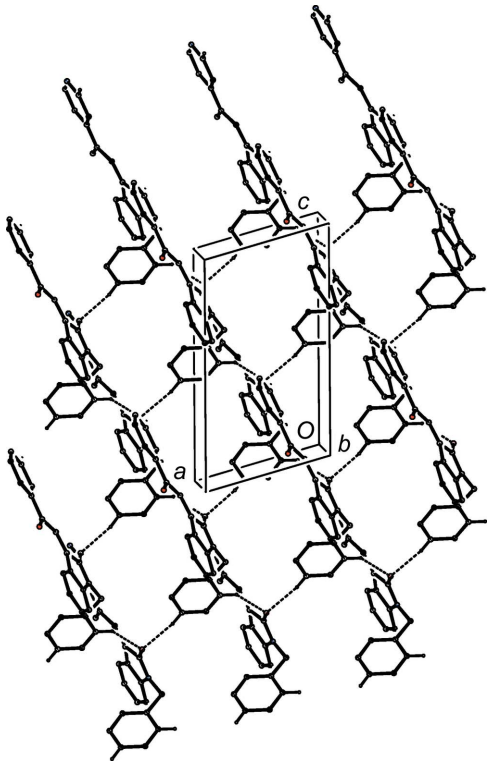


Figure 20
Part of the crystal structure of compound (II*e*), showing the formation of a sheet built from three C—H···O hydrogen bonds and lying parallel to (010). Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms not involved in the motifs shown have been omitted.

group compatibility, and eco-compatibility in terms of energy and waste. We note, in addition, that in each of the hydroxy compounds (I*c*), (I*d*) and (I*f*), paired O—H···O hydrogen bonds generate $R_2^2(10)$ motifs, which are centrosymmetric in each of (I*c*) and (I*d*), although the ring in (I*f*) exhibits no crystallographic symmetry. By contrast, the structure of pyridyl derivative (I*e*) contains no O—H···O hydrogen bonds (Table 2). It is interesting in this context to note that in a series of seven 3-alkyl-3-hydroxyindolin-2-ones, having no substituent on the N atom of the indoline ring, every structure contains a centrosymmetric $R_2^2(10)$ ring embedded within a more complex supramolecular assembly involving N—H···O hydrogen bonds and, in some cases, C—H···O and C—H···π(arene) hydrogen bonds also (Becerra *et al.*, 2010). Rings of the $R_2^2(10)$ type also occur in a number of related examples in the Cambridge Structural Database (CSD; Groom *et al.*, 2016), including examples having CSD refcodes MUMMAY (Chen *et al.*, 2009), TAWFAZ (Luppi *et al.*, 2005), TEQVUH (Luppi *et al.*, 2006) and YIFZIX (Xing *et al.*, 2007). Finally, we note that the reaction of isatin with cyclohexanone involves both of the α-methylene units of the cyclohexanone, leading to the formation of 3,3′-[(1*RS*,3*SR*)-2-oxocyclohexane-1,3-diyl]bis[(3*RS*,3′*SR*)-3-hydroxyindolin-2-one] which was crystallized as a dehydrate (Becerra *et al.*, 2013). The organic components, which exhibit approximate, but noncrystallographic, mirror symmetry are linked by a combination of N—H···O and O—H···O hydrogen bonds to form sheets containing rings of $R_2^2(8)$, $R_2^2(16)$ and $R_6^6(40)$ types; these sheets are further linked by water molecules, which themselves form cyclic centrosymmetric $R_4^2(8)$ tetramers.

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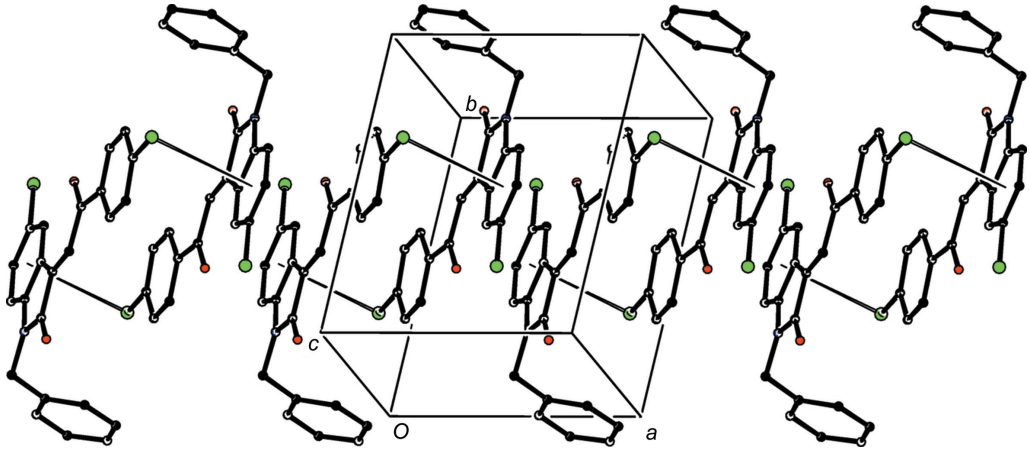

Figure 21
Part of the crystal structure of compound (II*h*), showing the formation of a chain of π-stacked dimers running parallel to the [100] direction. The Cl···(ring centroid) contacts are shown as tapered lines and, for the sake of clarity, H atoms have all been omitted.

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supporting information

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Synthesis of *N*-substituted 3-(2-aryl-2-oxoethyl)-3-hydroxyindolin-2-ones and their conversion to *N*-substituted (*E*)-3-(2-aryl-2-oxoethylidene)indolin-2-ones: synthetic sequence, spectroscopic characterization and structures of four 3-hydroxy compounds and five oxoethylidene products

Diana Becerra, Juan Castillo, Braulio Insuasty, Justo Cobo and Christopher Glidewell

Computing details

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

1-Benzyl-3-hydroxy-3-[2-(4-methoxyphenyl)-2-oxoethyl]indolin-2-one (Ic)

Crystal data

$C_{24}H_{21}NO_4$
M_r = 387.42
Monoclinic, $C2/c$
a = 18.7572 (14) Å
b = 13.2095 (10) Å
c = 16.750 (2) Å
β = 105.374 (5)°
V = 4001.7 (6) Å^3
Z = 8
$F(000)$ = 1632
D_x = 1.286 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4769 reflections
θ = 1.9–27.9°
μ = 0.09 mm^{-1}
T = 100 K
Block, colourless
0.14 × 0.13 × 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} = 0.908, T_{max} = 0.991
18506 measured reflections
4768 independent reflections
3469 reflections with $I > 2\sigma(I)$
R_{int} = 0.051
θ_{max} = 27.9°, θ_{min} = 2.1°
h = −24→22
k = −17→17
l = −22→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.110
S = 1.02
4768 reflections
266 parameters
0 restraints
Primary atom site location: difference Fourier map

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0428P)^2 + 3.9671P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.62730 (7)	0.52592 (9)	0.49110 (8)	0.0144 (3)
C2	0.66508 (8)	0.61463 (11)	0.49618 (10)	0.0142 (3)
O2	0.67273 (6)	0.66507 (8)	0.43745 (7)	0.0176 (2)
C3	0.70111 (8)	0.63794 (11)	0.58845 (9)	0.0142 (3)
O3	0.77962 (5)	0.63249 (8)	0.60183 (7)	0.0178 (2)
H3	0.7919 (10)	0.6897 (15)	0.5847 (12)	0.027*
C3A	0.67518 (8)	0.55016 (11)	0.63095 (9)	0.0142 (3)
C4	0.68731 (8)	0.52756 (11)	0.71359 (10)	0.0171 (3)
H4	0.7156	0.5716	0.7548	0.020*
C5	0.65713 (8)	0.43830 (12)	0.73571 (10)	0.0195 (3)
H5	0.6658	0.4206	0.7925	0.023*
C6	0.61469 (9)	0.37559 (12)	0.67508 (10)	0.0197 (3)
H6	0.5946	0.3153	0.6912	0.024*
C7	0.60061 (8)	0.39845 (11)	0.59091 (10)	0.0175 (3)
H7	0.5709	0.3558	0.5496	0.021*
C7A	0.63207 (8)	0.48611 (11)	0.57077 (9)	0.0142 (3)
C1	0.58919 (8)	0.47598 (11)	0.41444 (10)	0.0163 (3)
H1A	0.5816	0.5252	0.3684	0.020*
H1B	0.5399	0.4535	0.4184	0.020*
C11	0.63137 (8)	0.38522 (11)	0.39496 (9)	0.0164 (3)
C12	0.70791 (9)	0.37910 (12)	0.42166 (10)	0.0205 (3)
H12	0.7352	0.4324	0.4539	0.025*
C13	0.74512 (10)	0.29578 (13)	0.40179 (11)	0.0265 (4)
H13	0.7975	0.2925	0.4204	0.032*
C14	0.70605 (11)	0.21783 (13)	0.35505 (11)	0.0296 (4)
H14	0.7314	0.1608	0.3416	0.035*
C15	0.62979 (11)	0.22311 (13)	0.32796 (12)	0.0312 (4)
H15	0.6028	0.1697	0.2956	0.037*
C16	0.59252 (9)	0.30639 (13)	0.34795 (11)	0.0241 (4)
H16	0.5401	0.3094	0.3293	0.029*
C31	0.68121 (8)	0.74281 (11)	0.61383 (10)	0.0161 (3)
H31A	0.7019	0.7500	0.6744	0.019*
H31B	0.7057	0.7939	0.5868	0.019*
C32	0.59949 (8)	0.76693 (11)	0.59292 (9)	0.0155 (3)

O32	0.55330 (6)	0.70404 (8)	0.55953 (7)	0.0227 (3)
C321	0.57886 (8)	0.87039 (11)	0.61248 (10)	0.0164 (3)
C322	0.50481 (8)	0.89970 (12)	0.58966 (10)	0.0186 (3)
H322	0.4683	0.8528	0.5617	0.022*
C323	0.48334 (8)	0.99582 (12)	0.60699 (10)	0.0200 (3)
H323	0.4327	1.0148	0.5911	0.024*
C324	0.53700 (9)	1.06393 (12)	0.64785 (11)	0.0216 (4)
C325	0.61123 (9)	1.03573 (12)	0.67249 (12)	0.0278 (4)
H325	0.6475	1.0822	0.7017	0.033*
C326	0.63175 (8)	0.94024 (12)	0.65438 (11)	0.0229 (4)
H326	0.6824	0.9215	0.6705	0.027*
O324	0.52364 (6)	1.16070 (9)	0.66728 (9)	0.0326 (3)
C327	0.44939 (9)	1.19684 (14)	0.64073 (13)	0.0315 (4)
H37A	0.4180	1.1573	0.6675	0.047*
H37B	0.4312	1.1898	0.5805	0.047*
H37C	0.4479	1.2683	0.6560	0.047*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0156 (6)	0.0144 (6)	0.0135 (7)	−0.0009 (5)	0.0043 (5)	−0.0017 (5)
C2	0.0114 (7)	0.0141 (7)	0.0182 (8)	0.0018 (6)	0.0057 (6)	−0.0013 (6)
O2	0.0194 (5)	0.0169 (5)	0.0178 (6)	−0.0010 (4)	0.0075 (4)	0.0018 (4)
C3	0.0129 (7)	0.0142 (7)	0.0156 (8)	−0.0007 (6)	0.0040 (6)	−0.0017 (6)
O3	0.0125 (5)	0.0172 (5)	0.0237 (6)	−0.0013 (4)	0.0050 (4)	0.0003 (5)
C3A	0.0132 (7)	0.0138 (7)	0.0164 (8)	0.0017 (6)	0.0051 (6)	−0.0005 (6)
C4	0.0159 (7)	0.0186 (8)	0.0164 (8)	−0.0003 (6)	0.0039 (6)	−0.0022 (6)
C5	0.0220 (8)	0.0226 (8)	0.0159 (8)	0.0032 (6)	0.0083 (7)	0.0030 (6)
C6	0.0214 (8)	0.0160 (7)	0.0250 (9)	−0.0004 (6)	0.0122 (7)	0.0031 (6)
C7	0.0179 (7)	0.0156 (7)	0.0201 (8)	−0.0014 (6)	0.0069 (6)	−0.0026 (6)
C7A	0.0137 (7)	0.0158 (7)	0.0138 (8)	0.0024 (6)	0.0049 (6)	−0.0009 (6)
C1	0.0170 (7)	0.0178 (8)	0.0132 (8)	−0.0011 (6)	0.0023 (6)	−0.0018 (6)
C11	0.0225 (8)	0.0165 (7)	0.0121 (8)	−0.0010 (6)	0.0081 (6)	0.0006 (6)
C12	0.0254 (8)	0.0201 (8)	0.0161 (8)	0.0026 (7)	0.0055 (7)	0.0012 (6)
C13	0.0300 (9)	0.0292 (9)	0.0221 (9)	0.0107 (7)	0.0099 (7)	0.0059 (7)
C14	0.0475 (11)	0.0205 (9)	0.0282 (10)	0.0092 (8)	0.0232 (9)	0.0029 (7)
C15	0.0492 (11)	0.0213 (9)	0.0296 (10)	−0.0081 (8)	0.0219 (9)	−0.0100 (8)
C16	0.0285 (9)	0.0239 (8)	0.0232 (9)	−0.0050 (7)	0.0128 (7)	−0.0062 (7)
C31	0.0166 (7)	0.0139 (7)	0.0177 (8)	−0.0020 (6)	0.0041 (6)	−0.0029 (6)
C32	0.0170 (7)	0.0174 (7)	0.0126 (8)	−0.0019 (6)	0.0052 (6)	−0.0012 (6)
O32	0.0172 (5)	0.0207 (6)	0.0294 (7)	−0.0034 (5)	0.0051 (5)	−0.0085 (5)
C321	0.0169 (7)	0.0162 (7)	0.0166 (8)	−0.0011 (6)	0.0054 (6)	0.0008 (6)
C322	0.0159 (7)	0.0195 (8)	0.0188 (8)	−0.0030 (6)	0.0021 (6)	−0.0016 (6)
C323	0.0146 (7)	0.0211 (8)	0.0225 (9)	0.0029 (6)	0.0020 (6)	0.0015 (7)
C324	0.0202 (8)	0.0152 (8)	0.0291 (10)	0.0020 (6)	0.0061 (7)	−0.0010 (7)
C325	0.0162 (8)	0.0182 (8)	0.0454 (12)	−0.0024 (6)	0.0017 (7)	−0.0074 (8)
C326	0.0132 (7)	0.0194 (8)	0.0338 (10)	0.0012 (6)	0.0022 (7)	−0.0025 (7)
O324	0.0177 (6)	0.0168 (6)	0.0592 (9)	0.0037 (5)	0.0028 (6)	−0.0086 (6)

C327	0.0224 (8)	0.0253 (9)	0.0432 (12)	0.0111 (7)	0.0024 (8)	−0.0025 (8)

Geometric parameters (Å, º)

N1—C2	1.3603 (19)	C13—H13	0.9500
N1—C7A	1.4150 (19)	C14—C15	1.383 (3)
N1—C1	1.4520 (19)	C14—H14	0.9500
C2—O2	1.2277 (18)	C15—C16	1.391 (2)
C2—C3	1.545 (2)	C15—H15	0.9500
C3—O3	1.4315 (17)	C16—H16	0.9500
C3—C3A	1.507 (2)	C31—C32	1.513 (2)
C3—C31	1.524 (2)	C31—H31A	0.9900
O3—H3	0.86 (2)	C31—H31B	0.9900
C3A—C4	1.375 (2)	C32—O32	1.2234 (18)
C3A—C7A	1.398 (2)	C32—C321	1.481 (2)
C4—C5	1.400 (2)	C321—C322	1.394 (2)
C4—H4	0.9500	C321—C326	1.400 (2)
C5—C6	1.387 (2)	C322—C323	1.386 (2)
C5—H5	0.9500	C322—H322	0.9500
C6—C7	1.397 (2)	C323—C324	1.388 (2)
C6—H6	0.9500	C323—H323	0.9500
C7—C7A	1.381 (2)	C324—O324	1.3586 (19)
C7—H7	0.9500	C324—C325	1.394 (2)
C1—C11	1.519 (2)	C325—C326	1.376 (2)
C1—H1A	0.9900	C325—H325	0.9500
C1—H1B	0.9900	C326—H326	0.9500
C11—C12	1.388 (2)	O324—C327	1.4272 (19)
C11—C16	1.389 (2)	C327—H37A	0.9800
C12—C13	1.390 (2)	C327—H37B	0.9800
C12—H12	0.9500	C327—H37C	0.9800
C13—C14	1.381 (3)		
C2—N1—C7A	111.07 (12)	C12—C13—H13	119.9
C2—N1—C1	124.95 (13)	C13—C14—C15	119.72 (16)
C7A—N1—C1	123.94 (12)	C13—C14—H14	120.1
O2—C2—N1	125.94 (14)	C15—C14—H14	120.1
O2—C2—C3	125.48 (13)	C14—C15—C16	120.20 (16)
N1—C2—C3	108.47 (12)	C14—C15—H15	119.9
O3—C3—C3A	109.55 (12)	C16—C15—H15	119.9
O3—C3—C31	108.66 (11)	C11—C16—C15	120.50 (16)
C3A—C3—C31	115.72 (12)	C11—C16—H16	119.8
O3—C3—C2	107.80 (11)	C15—C16—H16	119.8
C3A—C3—C2	102.06 (11)	C32—C31—C3	115.77 (12)
C31—C3—C2	112.69 (12)	C32—C31—H31A	108.3
C3—O3—H3	105.0 (12)	C3—C31—H31A	108.3
C4—C3A—C7A	120.28 (14)	C32—C31—H31B	108.3
C4—C3A—C3	130.90 (14)	C3—C31—H31B	108.3
C7A—C3A—C3	108.82 (13)	H31A—C31—H31B	107.4

C3A—C4—C5	118.62 (14)	O32—C32—C321	122.21 (13)
C3A—C4—H4	120.7	O32—C32—C31	120.94 (13)
C5—C4—H4	120.7	C321—C32—C31	116.83 (12)
C6—C5—C4	120.22 (15)	C322—C321—C326	118.46 (14)
C6—C5—H5	119.9	C322—C321—C32	119.71 (13)
C4—C5—H5	119.9	C326—C321—C32	121.83 (13)
C5—C6—C7	121.89 (14)	C323—C322—C321	121.39 (14)
C5—C6—H6	119.1	C323—C322—H322	119.3
C7—C6—H6	119.1	C321—C322—H322	119.3
C7A—C7—C6	116.68 (14)	C322—C323—C324	118.96 (14)
C7A—C7—H7	121.7	C322—C323—H323	120.5
C6—C7—H7	121.7	C324—C323—H323	120.5
C7—C7A—C3A	122.30 (14)	O324—C324—C323	124.91 (14)
C7—C7A—N1	128.13 (14)	O324—C324—C325	114.44 (14)
C3A—C7A—N1	109.57 (13)	C323—C324—C325	120.65 (15)
N1—C1—C11	112.73 (12)	C326—C325—C324	119.71 (15)
N1—C1—H1A	109.0	C326—C325—H325	120.1
C11—C1—H1A	109.0	C324—C325—H325	120.1
N1—C1—H1B	109.0	C325—C326—C321	120.83 (14)
C11—C1—H1B	109.0	C325—C326—H326	119.6
H1A—C1—H1B	107.8	C321—C326—H326	119.6
C12—C11—C16	118.75 (14)	C324—O324—C327	117.83 (13)
C12—C11—C1	121.99 (14)	O324—C327—H37A	109.5
C16—C11—C1	119.24 (14)	O324—C327—H37B	109.5
C11—C12—C13	120.73 (16)	H37A—C327—H37B	109.5
C11—C12—H12	119.6	O324—C327—H37C	109.5
C13—C12—H12	119.6	H37A—C327—H37C	109.5
C14—C13—C12	120.10 (16)	H37B—C327—H37C	109.5
C14—C13—H13	119.9		

C7A—N1—C2—O2	177.66 (13)	C7A—N1—C1—C11	−73.86 (17)
C1—N1—C2—O2	−0.2 (2)	N1—C1—C11—C12	−28.5 (2)
C7A—N1—C2—C3	1.26 (15)	N1—C1—C11—C16	152.53 (14)
C1—N1—C2—C3	−176.61 (12)	C16—C11—C12—C13	0.0 (2)
O2—C2—C3—O3	−62.46 (18)	C1—C11—C12—C13	−178.99 (15)
N1—C2—C3—O3	113.96 (12)	C11—C12—C13—C14	0.0 (2)
O2—C2—C3—C3A	−177.79 (13)	C12—C13—C14—C15	0.1 (3)
N1—C2—C3—C3A	−1.37 (14)	C13—C14—C15—C16	−0.3 (3)
O2—C2—C3—C31	57.44 (18)	C12—C11—C16—C15	−0.1 (2)
N1—C2—C3—C31	−126.14 (13)	C1—C11—C16—C15	178.90 (15)
O3—C3—C3A—C4	67.16 (19)	C14—C15—C16—C11	0.3 (3)
C31—C3—C3A—C4	−56.1 (2)	O3—C3—C31—C32	171.97 (12)
C2—C3—C3A—C4	−178.79 (15)	C3A—C3—C31—C32	−64.34 (18)
O3—C3—C3A—C7A	−113.04 (13)	C2—C3—C31—C32	52.57 (17)
C31—C3—C3A—C7A	123.74 (14)	C3—C31—C32—O32	1.7 (2)
C2—C3—C3A—C7A	1.01 (14)	C3—C31—C32—C321	−176.52 (13)
C7A—C3A—C4—C5	1.5 (2)	O32—C32—C321—C322	−2.5 (2)
C3—C3A—C4—C5	−178.71 (14)	C31—C32—C321—C322	175.64 (14)

C3A—C4—C5—C6	−1.3 (2)	O32—C32—C321—C326	176.75 (15)
C4—C5—C6—C7	0.1 (2)	C31—C32—C321—C326	−5.1 (2)
C5—C6—C7—C7A	1.0 (2)	C326—C321—C322—C323	0.6 (2)
C6—C7—C7A—C3A	−0.8 (2)	C32—C321—C322—C323	179.91 (15)
C6—C7—C7A—N1	179.29 (14)	C321—C322—C323—C324	0.1 (2)
C4—C3A—C7A—C7	−0.5 (2)	C322—C323—C324—O324	178.83 (16)
C3—C3A—C7A—C7	179.72 (13)	C322—C323—C324—C325	−1.2 (3)
C4—C3A—C7A—N1	179.48 (13)	O324—C324—C325—C326	−178.42 (17)
C3—C3A—C7A—N1	−0.34 (16)	C323—C324—C325—C326	1.6 (3)
C2—N1—C7A—C7	179.33 (14)	C324—C325—C326—C321	−0.9 (3)
C1—N1—C7A—C7	−2.8 (2)	C322—C321—C326—C325	−0.2 (3)
C2—N1—C7A—C3A	−0.61 (16)	C32—C321—C326—C325	−179.47 (16)
C1—N1—C7A—C3A	177.30 (12)	C323—C324—O324—C327	−2.8 (3)
C2—N1—C1—C11	103.75 (16)	C325—C324—O324—C327	177.16 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O2^{i}	0.86 (2)	2.10 (2)	2.9487 (15)	171.1 (18)
C6—H6···O324^{ii}	0.95	2.41	3.297 (2)	155
C31—H31*B*···O2^{i}	0.99	2.48	3.312 (2)	141
C4—H4···*Cg*2^{iii}	0.95	2.93	3.6100 (2)	130
C14—H14···*Cg*1^{iv}	0.95	2.82	3.709 (2)	156

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

1-Benzyl-3-{2-[4-(dimethylamino)phenyl]-2-oxoethyl}-3-hydroxyindolin-2-one (Id)

Crystal data

$C_{25}H_{24}N_2O_3$
M_r = 400.46
Triclinic, $P\overline{1}$
a = 9.1028 (8) Å
b = 10.6434 (9) Å
c = 11.2539 (10) Å
α = 88.988 (3)°
β = 68.422 (3)°
γ = 81.352 (3)°
V = 1001.55 (15) Å^3

Z = 2
F(000) = 424
D_x = 1.328 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4590 reflections
θ = 2.4–27.5°
μ = 0.09 mm^{-1}
T = 100 K
Block, yellow
0.23 × 0.19 × 0.16 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} = 0.946, T_{max} = 0.986

41110 measured reflections
4590 independent reflections
4033 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 27.5°, θ_{min} = 2.4°
h = −11→11
k = −13→13
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.098$
$S = 1.05$
4590 reflections
276 parameters
0 restraints

Primary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0365P)^2 + 0.557P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ 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	0.21923 (12)	0.42207 (9)	0.80825 (9)	0.0170 (2)
C2	0.29276 (13)	0.47725 (11)	0.69674 (11)	0.0170 (2)
O2	0.29201 (10)	0.44841 (8)	0.59167 (8)	0.02181 (19)
C3	0.39277 (13)	0.57237 (10)	0.72117 (10)	0.0158 (2)
O3	0.55634 (10)	0.51351 (8)	0.66550 (8)	0.01902 (18)
H3	0.5876 (19)	0.5219 (15)	0.5834 (17)	0.029*
C3A	0.34407 (13)	0.57037 (10)	0.86439 (11)	0.0163 (2)
C4	0.38692 (14)	0.63774 (11)	0.94672 (11)	0.0192 (2)
H4	0.4525	0.7018	0.9161	0.023*
C5	0.33187 (15)	0.60992 (11)	1.07608 (11)	0.0213 (2)
H5	0.3592	0.6559	1.1345	0.026*
C6	0.23729 (15)	0.51525 (12)	1.11959 (11)	0.0216 (2)
H6	0.2023	0.4968	1.2077	0.026*
C7	0.19207 (14)	0.44631 (11)	1.03756 (11)	0.0189 (2)
H7	0.1268	0.3820	1.0677	0.023*
C7A	0.24741 (13)	0.47679 (10)	0.91005 (11)	0.0163 (2)
C1	0.14460 (14)	0.30851 (11)	0.81849 (11)	0.0192 (2)
H1A	0.1233	0.2956	0.7398	0.023*
H1B	0.0409	0.3206	0.8916	0.023*
C11	0.24998 (14)	0.19161 (11)	0.83744 (11)	0.0179 (2)
C12	0.41445 (15)	0.17355 (12)	0.77052 (13)	0.0257 (3)
H12	0.4613	0.2350	0.7123	0.031*
C13	0.51067 (16)	0.06643 (13)	0.78816 (14)	0.0298 (3)
H13	0.6229	0.0553	0.7424	0.036*
C14	0.44390 (16)	−0.02404 (12)	0.87200 (14)	0.0273 (3)
H14	0.5101	−0.0970	0.8844	0.033*
C15	0.28005 (16)	−0.00807 (12)	0.93797 (13)	0.0273 (3)
H15	0.2335	−0.0708	0.9945	0.033*
C16	0.18366 (15)	0.10006 (12)	0.92129 (12)	0.0229 (3)

H16	0.0715	0.1113	0.9677	0.027*
C31	0.37035 (13)	0.70199 (11)	0.66464 (11)	0.0174 (2)
H31A	0.4385	0.7565	0.6839	0.021*
H31B	0.4079	0.6904	0.5705	0.021*
C32	0.19918 (13)	0.77032 (11)	0.71393 (11)	0.0176 (2)
O32	0.09303 (10)	0.71812 (8)	0.78944 (9)	0.0254 (2)
C321	0.16513 (13)	0.89691 (11)	0.66678 (11)	0.0168 (2)
C322	0.00952 (14)	0.96476 (11)	0.71684 (11)	0.0189 (2)
H322	−0.0700	0.9303	0.7845	0.023*
C323	−0.03118 (14)	1.08007 (11)	0.67066 (11)	0.0195 (2)
H323	−0.1367	1.1251	0.7090	0.023*
C324	0.08214 (14)	1.13228 (11)	0.56692 (11)	0.0185 (2)
C325	0.24047 (14)	1.06692 (12)	0.52031 (12)	0.0215 (2)
H325	0.3210	1.1020	0.4541	0.026*
C326	0.28000 (14)	0.95258 (11)	0.56964 (12)	0.0203 (2)
H326	0.3876	0.9108	0.5369	0.024*
N324	0.03955 (13)	1.24116 (10)	0.51404 (11)	0.0246 (2)
C317	−0.12376 (16)	1.30688 (12)	0.56315 (12)	0.0263 (3)
H37A	−0.1955	1.2501	0.5556	0.039*
H37B	−0.1324	1.3826	0.5138	0.039*
H37C	−0.1539	1.3325	0.6533	0.039*
C318	0.15137 (17)	1.28623 (13)	0.39880 (14)	0.0313 (3)
H38A	0.2369	1.3164	0.4182	0.047*
H38B	0.0953	1.3562	0.3664	0.047*
H38C	0.1975	1.2166	0.3338	0.047*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0200 (5)	0.0151 (4)	0.0163 (4)	−0.0040 (4)	−0.0067 (4)	0.0023 (3)
C2	0.0167 (5)	0.0154 (5)	0.0175 (5)	−0.0001 (4)	−0.0058 (4)	0.0023 (4)
O2	0.0257 (4)	0.0241 (4)	0.0167 (4)	−0.0051 (3)	−0.0086 (3)	0.0017 (3)
C3	0.0153 (5)	0.0152 (5)	0.0159 (5)	−0.0019 (4)	−0.0049 (4)	0.0030 (4)
O3	0.0156 (4)	0.0216 (4)	0.0171 (4)	0.0006 (3)	−0.0044 (3)	0.0035 (3)
C3A	0.0163 (5)	0.0146 (5)	0.0167 (5)	−0.0007 (4)	−0.0052 (4)	0.0027 (4)
C4	0.0206 (5)	0.0153 (5)	0.0214 (6)	−0.0021 (4)	−0.0079 (4)	0.0020 (4)
C5	0.0254 (6)	0.0195 (6)	0.0196 (6)	−0.0007 (5)	−0.0100 (5)	−0.0016 (4)
C6	0.0245 (6)	0.0220 (6)	0.0153 (5)	−0.0003 (5)	−0.0053 (4)	0.0021 (4)
C7	0.0193 (5)	0.0168 (5)	0.0181 (5)	−0.0022 (4)	−0.0043 (4)	0.0035 (4)
C7A	0.0165 (5)	0.0143 (5)	0.0167 (5)	−0.0003 (4)	−0.0054 (4)	0.0003 (4)
C1	0.0196 (5)	0.0166 (5)	0.0228 (6)	−0.0056 (4)	−0.0084 (4)	0.0021 (4)
C11	0.0199 (5)	0.0158 (5)	0.0184 (5)	−0.0037 (4)	−0.0069 (4)	−0.0011 (4)
C12	0.0216 (6)	0.0202 (6)	0.0303 (6)	−0.0046 (5)	−0.0037 (5)	0.0037 (5)
C13	0.0196 (6)	0.0226 (6)	0.0418 (8)	−0.0010 (5)	−0.0058 (5)	−0.0002 (5)
C14	0.0281 (6)	0.0176 (6)	0.0372 (7)	0.0004 (5)	−0.0146 (6)	−0.0005 (5)
C15	0.0306 (7)	0.0201 (6)	0.0294 (7)	−0.0050 (5)	−0.0089 (5)	0.0064 (5)
C16	0.0213 (6)	0.0201 (6)	0.0241 (6)	−0.0040 (5)	−0.0044 (5)	0.0028 (5)
C31	0.0169 (5)	0.0158 (5)	0.0184 (5)	−0.0026 (4)	−0.0054 (4)	0.0054 (4)

C32	0.0179 (5)	0.0174 (5)	0.0171 (5)	−0.0031 (4)	−0.0059 (4)	0.0029 (4)
O32	0.0188 (4)	0.0231 (4)	0.0301 (5)	−0.0040 (3)	−0.0042 (4)	0.0113 (4)
C321	0.0180 (5)	0.0157 (5)	0.0174 (5)	−0.0024 (4)	−0.0073 (4)	0.0019 (4)
C322	0.0183 (5)	0.0194 (5)	0.0179 (5)	−0.0029 (4)	−0.0053 (4)	0.0020 (4)
C323	0.0175 (5)	0.0183 (5)	0.0219 (6)	0.0000 (4)	−0.0074 (4)	−0.0004 (4)
C324	0.0218 (6)	0.0144 (5)	0.0229 (6)	−0.0038 (4)	−0.0123 (5)	0.0020 (4)
C325	0.0193 (6)	0.0203 (6)	0.0253 (6)	−0.0061 (4)	−0.0078 (5)	0.0071 (5)
C326	0.0164 (5)	0.0190 (6)	0.0245 (6)	−0.0027 (4)	−0.0067 (5)	0.0049 (4)
N324	0.0242 (5)	0.0192 (5)	0.0320 (6)	−0.0024 (4)	−0.0129 (5)	0.0090 (4)
C317	0.0317 (7)	0.0204 (6)	0.0256 (6)	0.0068 (5)	−0.0133 (5)	−0.0012 (5)
C318	0.0283 (7)	0.0256 (6)	0.0418 (8)	−0.0073 (5)	−0.0146 (6)	0.0182 (6)

Geometric parameters (Å, º)

N1—C2	1.3572 (14)	C14—H14	0.9500
N1—C7A	1.4149 (14)	C15—C16	1.3926 (18)
N1—C1	1.4561 (14)	C15—H15	0.9500
C2—O2	1.2296 (14)	C16—H16	0.9500
C2—C3	1.5494 (16)	C31—C32	1.5173 (15)
C3—O3	1.4315 (13)	C31—H31A	0.9900
C3—C3A	1.5070 (15)	C31—H31B	0.9900
C3—C31	1.5262 (15)	C32—O32	1.2269 (14)
O3—H3	0.868 (17)	C32—C321	1.4708 (15)
C3A—C4	1.3777 (16)	C321—C326	1.4008 (16)
C3A—C7A	1.3939 (15)	C321—C322	1.4018 (16)
C4—C5	1.3968 (16)	C322—C323	1.3770 (16)
C4—H4	0.9500	C322—H322	0.9500
C5—C6	1.3892 (17)	C323—C324	1.4140 (16)
C5—H5	0.9500	C323—H323	0.9500
C6—C7	1.3980 (17)	C324—N324	1.3629 (15)
C6—H6	0.9500	C324—C325	1.4113 (16)
C7—C7A	1.3853 (16)	C325—C326	1.3820 (16)
C7—H7	0.9500	C325—H325	0.9500
C1—C11	1.5152 (16)	C326—H326	0.9500
C1—H1A	0.9900	N324—C317	1.4496 (16)
C1—H1B	0.9900	N324—C318	1.4498 (17)
C11—C16	1.3892 (16)	C317—H37A	0.9800
C11—C12	1.3909 (17)	C317—H37B	0.9800
C12—C13	1.3878 (18)	C317—H37C	0.9800
C12—H12	0.9500	C318—H38A	0.9800
C13—C14	1.3817 (19)	C318—H38B	0.9800
C13—H13	0.9500	C318—H38C	0.9800
C14—C15	1.3850 (19)		

C2—N1—C7A	110.74 (9)	C14—C15—C16	119.93 (12)
C2—N1—C1	124.55 (10)	C14—C15—H15	120.0
C7A—N1—C1	123.96 (9)	C16—C15—H15	120.0
O2—C2—N1	125.72 (11)	C11—C16—C15	120.61 (12)

O2—C2—C3	125.43 (10)	C11—C16—H16	119.7
N1—C2—C3	108.56 (9)	C15—C16—H16	119.7
O3—C3—C3A	107.60 (9)	C32—C31—C3	114.25 (9)
O3—C3—C31	110.11 (9)	C32—C31—H31A	108.7
C3A—C3—C31	116.70 (9)	C3—C31—H31A	108.7
O3—C3—C2	106.14 (9)	C32—C31—H31B	108.7
C3A—C3—C2	101.64 (9)	C3—C31—H31B	108.7
C31—C3—C2	113.83 (9)	H31A—C31—H31B	107.6
C3—O3—H3	107.7 (11)	O32—C32—C321	121.87 (10)
C4—C3A—C7A	120.36 (10)	O32—C32—C31	119.58 (10)
C4—C3A—C3	130.79 (10)	C321—C32—C31	118.54 (10)
C7A—C3A—C3	108.74 (10)	C326—C321—C322	117.41 (10)
C3A—C4—C5	118.61 (11)	C326—C321—C32	123.18 (10)
C3A—C4—H4	120.7	C322—C321—C32	119.37 (10)
C5—C4—H4	120.7	C323—C322—C321	121.76 (11)
C6—C5—C4	120.23 (11)	C323—C322—H322	119.1
C6—C5—H5	119.9	C321—C322—H322	119.1
C4—C5—H5	119.9	C322—C323—C324	120.82 (11)
C5—C6—C7	121.94 (11)	C322—C323—H323	119.6
C5—C6—H6	119.0	C324—C323—H323	119.6
C7—C6—H6	119.0	N324—C324—C325	121.51 (11)
C7A—C7—C6	116.47 (11)	N324—C324—C323	121.16 (11)
C7A—C7—H7	121.8	C325—C324—C323	117.33 (10)
C6—C7—H7	121.8	C326—C325—C324	120.91 (11)
C7—C7A—C3A	122.39 (11)	C326—C325—H325	119.5
C7—C7A—N1	127.76 (10)	C324—C325—H325	119.5
C3A—C7A—N1	109.85 (10)	C325—C326—C321	121.55 (11)
N1—C1—C11	111.53 (9)	C325—C326—H326	119.2
N1—C1—H1A	109.3	C321—C326—H326	119.2
C11—C1—H1A	109.3	C324—N324—C317	120.70 (11)
N1—C1—H1B	109.3	C324—N324—C318	120.84 (11)
C11—C1—H1B	109.3	C317—N324—C318	118.10 (10)
H1A—C1—H1B	108.0	N324—C317—H37A	109.5
C16—C11—C12	118.86 (11)	N324—C317—H37B	109.5
C16—C11—C1	120.49 (10)	H37A—C317—H37B	109.5
C12—C11—C1	120.65 (11)	N324—C317—H37C	109.5
C13—C12—C11	120.51 (12)	H37A—C317—H37C	109.5
C13—C12—H12	119.7	H37B—C317—H37C	109.5
C11—C12—H12	119.7	N324—C318—H38A	109.5
C14—C13—C12	120.30 (12)	N324—C318—H38B	109.5
C14—C13—H13	119.8	H38A—C318—H38B	109.5
C12—C13—H13	119.8	N324—C318—H38C	109.5
C13—C14—C15	119.77 (12)	H38A—C318—H38C	109.5
C13—C14—H14	120.1	H38B—C318—H38C	109.5
C15—C14—H14	120.1		

C7A—N1—C2—O2	178.77 (11)	N1—C1—C11—C16	140.20 (11)
C1—N1—C2—O2	8.34 (18)	N1—C1—C11—C12	−40.32 (15)

C7A—N1—C2—C3	4.82 (12)	C16—C11—C12—C13	−0.57 (19)
C1—N1—C2—C3	−165.60 (10)	C1—C11—C12—C13	179.94 (12)
O2—C2—C3—O3	−68.32 (13)	C11—C12—C13—C14	0.4 (2)
N1—C2—C3—O3	105.65 (10)	C12—C13—C14—C15	0.5 (2)
O2—C2—C3—C3A	179.29 (11)	C13—C14—C15—C16	−1.1 (2)
N1—C2—C3—C3A	−6.74 (11)	C12—C11—C16—C15	−0.11 (19)
O2—C2—C3—C31	52.96 (15)	C1—C11—C16—C15	179.38 (11)
N1—C2—C3—C31	−133.07 (10)	C14—C15—C16—C11	1.0 (2)
O3—C3—C3A—C4	70.98 (15)	O3—C3—C31—C32	177.82 (9)
C31—C3—C3A—C4	−53.30 (16)	C3A—C3—C31—C32	−59.18 (13)
C2—C3—C3A—C4	−177.73 (12)	C2—C3—C31—C32	58.79 (13)
O3—C3—C3A—C7A	−104.95 (10)	C3—C31—C32—O32	−2.32 (16)
C31—C3—C3A—C7A	130.76 (10)	C3—C31—C32—C321	179.12 (10)
C2—C3—C3A—C7A	6.33 (11)	O32—C32—C321—C326	−172.29 (12)
C7A—C3A—C4—C5	0.17 (17)	C31—C32—C321—C326	6.24 (17)
C3—C3A—C4—C5	−175.37 (11)	O32—C32—C321—C322	5.26 (17)
C3A—C4—C5—C6	0.64 (17)	C31—C32—C321—C322	−176.22 (10)
C4—C5—C6—C7	−0.94 (18)	C326—C321—C322—C323	1.74 (17)
C5—C6—C7—C7A	0.39 (17)	C32—C321—C322—C323	−175.94 (11)
C6—C7—C7A—C3A	0.44 (17)	C321—C322—C323—C324	2.42 (18)
C6—C7—C7A—N1	−179.93 (11)	C322—C323—C324—N324	174.68 (11)
C4—C3A—C7A—C7	−0.73 (17)	C322—C323—C324—C325	−5.10 (17)
C3—C3A—C7A—C7	175.70 (10)	N324—C324—C325—C326	−176.04 (12)
C4—C3A—C7A—N1	179.59 (10)	C323—C324—C325—C326	3.74 (17)
C3—C3A—C7A—N1	−3.98 (12)	C324—C325—C326—C321	0.35 (19)
C2—N1—C7A—C7	179.72 (11)	C322—C321—C326—C325	−3.12 (18)
C1—N1—C7A—C7	−9.79 (18)	C32—C321—C326—C325	174.46 (11)
C2—N1—C7A—C3A	−0.62 (13)	C325—C324—N324—C317	179.93 (11)
C1—N1—C7A—C3A	169.87 (10)	C323—C324—N324—C317	0.16 (18)
C2—N1—C1—C11	102.05 (12)	C325—C324—N324—C318	6.91 (18)
C7A—N1—C1—C11	−67.14 (14)	C323—C324—N324—C318	−172.87 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O2^{i}	0.868 (18)	1.918 (18)	2.7630 (12)	164.2 (18)
C7—H7···O32^{ii}	0.95	2.44	3.3343 (16)	157
C1—H1*B*···*Cg*1^{ii}	0.99	2.96	3.8375 (14)	149

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

1-Benzyl-3-hydroxy-3-[2-oxo-2-(pyridin-4-yl)ethyl]indolin-2-one (Ie)

Crystal data

$C_{22}H_{18}N_2O_3$
M_r = 358.38
Triclinic, $P\overline{1}$
a = 7.8838 (5) Å
b = 10.1766 (8) Å
c = 11.8719 (9) Å
α = 87.554 (3)°
β = 75.996 (2)°
γ = 69.428 (2)°
V = 864.30 (11) Å^3

$Z = 2$
$F(000) = 376$
$D_x = 1.377$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4333 reflections
$\theta = 2.1$–$28.4°$
$\mu = 0.09$ mm^{-1}
$T = 100$ K
Plate, yellow
0.25 × 0.16 × 0.06 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
$T_{min} = 0.949$, $T_{max} = 0.994$
55488 measured reflections
4333 independent reflections
3760 reflections with $I > 2\sigma(I)$
$R_{int} = 0.045$
$\theta_{max} = 28.4°$, $\theta_{min} = 2.1°$
$h = -10 \rightarrow 9$
$k = -13 \rightarrow 13$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.101$
$S = 1.07$
4333 reflections
247 parameters
0 restraints
Primary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0383P)^2 + 0.4879P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ 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	0.24495 (13)	0.64127 (10)	0.18673 (8)	0.01464 (19)
C2	0.16480 (16)	0.61073 (11)	0.29576 (9)	0.0150 (2)
O2	−0.00152 (12)	0.63587 (9)	0.33661 (7)	0.01994 (18)
C3	0.32150 (16)	0.54461 (11)	0.36118 (9)	0.0139 (2)
O3	0.28239 (12)	0.63180 (8)	0.46198 (7)	0.01649 (17)
H3	0.309 (2)	0.7091 (18)	0.4396 (14)	0.025*
C3A	0.49328 (15)	0.54471 (11)	0.27087 (9)	0.0136 (2)
C4	0.67770 (16)	0.50366 (11)	0.27635 (10)	0.0158 (2)
H4	0.7150	0.4641	0.3443	0.019*
C5	0.80840 (16)	0.52184 (12)	0.17922 (10)	0.0173 (2)
H5	0.9355	0.4961	0.1817	0.021*
C6	0.75361 (16)	0.57706 (12)	0.07952 (10)	0.0176 (2)
H6	0.8448	0.5870	0.0142	0.021*
C7	0.56727 (16)	0.61859 (11)	0.07256 (9)	0.0159 (2)

H7	0.5303	0.6554	0.0039	0.019*
C7A	0.43947 (15)	0.60344 (11)	0.17049 (9)	0.0139 (2)
C1	0.13877 (16)	0.72563 (12)	0.10687 (10)	0.0165 (2)
H1A	0.0121	0.7203	0.1260	0.020*
H1B	0.2013	0.6869	0.0263	0.020*
C11	0.12346 (16)	0.87771 (12)	0.11464 (10)	0.0161 (2)
C12	−0.00309 (18)	0.96473 (13)	0.20838 (11)	0.0233 (3)
H12	−0.0816	0.9292	0.2651	0.028*
C13	−0.0148 (2)	1.10359 (14)	0.21910 (12)	0.0291 (3)
H13	−0.1017	1.1627	0.2828	0.035*
C14	0.1004 (2)	1.15553 (13)	0.13666 (12)	0.0261 (3)
H14	0.0936	1.2499	0.1446	0.031*
C15	0.22536 (18)	1.06996 (13)	0.04287 (12)	0.0241 (3)
H15	0.3039	1.1057	−0.0137	0.029*
C16	0.23575 (17)	0.93145 (12)	0.03164 (11)	0.0201 (2)
H16	0.3203	0.8733	−0.0333	0.024*
C31	0.32254 (16)	0.40140 (11)	0.40685 (9)	0.0156 (2)
H31A	0.4190	0.3665	0.4519	0.019*
H31B	0.2001	0.4142	0.4609	0.019*
C32	0.35969 (17)	0.29114 (12)	0.31438 (10)	0.0171 (2)
O32	0.38952 (15)	0.31315 (9)	0.21135 (8)	0.0268 (2)
N321	0.35555 (17)	−0.12102 (11)	0.40608 (9)	0.0241 (2)
C322	0.46952 (19)	−0.10040 (13)	0.30937 (11)	0.0248 (3)
H322	0.5506	−0.1795	0.2595	0.030*
C323	0.47504 (19)	0.03098 (13)	0.27837 (11)	0.0236 (3)
H323	0.5555	0.0415	0.2076	0.028*
C324	0.36135 (17)	0.14756 (12)	0.35208 (10)	0.0168 (2)
C325	0.2467 (2)	0.12723 (13)	0.45451 (11)	0.0269 (3)
H325	0.1704	0.2034	0.5085	0.032*
C326	0.2468 (2)	−0.00871 (14)	0.47597 (12)	0.0315 (3)
H326	0.1642	−0.0220	0.5446	0.038*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0170 (4)	0.0131 (4)	0.0139 (4)	−0.0056 (3)	−0.0038 (3)	0.0028 (3)
C2	0.0206 (5)	0.0109 (5)	0.0144 (5)	−0.0075 (4)	−0.0030 (4)	0.0000 (4)
O2	0.0187 (4)	0.0220 (4)	0.0196 (4)	−0.0093 (3)	−0.0020 (3)	0.0003 (3)
C3	0.0201 (5)	0.0107 (5)	0.0118 (5)	−0.0071 (4)	−0.0031 (4)	0.0005 (4)
O3	0.0266 (4)	0.0120 (4)	0.0115 (4)	−0.0089 (3)	−0.0025 (3)	0.0000 (3)
C3A	0.0193 (5)	0.0090 (5)	0.0128 (5)	−0.0061 (4)	−0.0030 (4)	0.0005 (4)
C4	0.0214 (5)	0.0114 (5)	0.0151 (5)	−0.0055 (4)	−0.0054 (4)	0.0004 (4)
C5	0.0175 (5)	0.0131 (5)	0.0206 (6)	−0.0050 (4)	−0.0037 (4)	−0.0013 (4)
C6	0.0195 (5)	0.0140 (5)	0.0171 (5)	−0.0067 (4)	0.0009 (4)	−0.0002 (4)
C7	0.0213 (5)	0.0124 (5)	0.0129 (5)	−0.0059 (4)	−0.0025 (4)	0.0014 (4)
C7A	0.0177 (5)	0.0099 (5)	0.0144 (5)	−0.0054 (4)	−0.0038 (4)	0.0004 (4)
C1	0.0218 (5)	0.0142 (5)	0.0160 (5)	−0.0067 (4)	−0.0087 (4)	0.0031 (4)
C11	0.0186 (5)	0.0135 (5)	0.0176 (5)	−0.0051 (4)	−0.0083 (4)	0.0036 (4)

C12	0.0270 (6)	0.0182 (6)	0.0208 (6)	−0.0063 (5)	−0.0013 (5)	0.0026 (4)
C13	0.0391 (8)	0.0170 (6)	0.0239 (6)	−0.0037 (5)	−0.0029 (5)	−0.0010 (5)
C14	0.0356 (7)	0.0141 (5)	0.0302 (7)	−0.0076 (5)	−0.0133 (6)	0.0039 (5)
C15	0.0263 (6)	0.0178 (6)	0.0290 (6)	−0.0093 (5)	−0.0073 (5)	0.0085 (5)
C16	0.0222 (6)	0.0160 (5)	0.0195 (5)	−0.0044 (4)	−0.0041 (4)	0.0037 (4)
C31	0.0233 (5)	0.0112 (5)	0.0129 (5)	−0.0081 (4)	−0.0028 (4)	0.0019 (4)
C32	0.0238 (5)	0.0126 (5)	0.0158 (5)	−0.0088 (4)	−0.0031 (4)	0.0013 (4)
O32	0.0507 (6)	0.0194 (4)	0.0147 (4)	−0.0193 (4)	−0.0058 (4)	0.0028 (3)
N321	0.0396 (6)	0.0156 (5)	0.0187 (5)	−0.0141 (4)	−0.0031 (4)	0.0014 (4)
C322	0.0338 (7)	0.0142 (5)	0.0236 (6)	−0.0100 (5)	0.0010 (5)	−0.0026 (4)
C323	0.0321 (7)	0.0168 (6)	0.0194 (6)	−0.0117 (5)	0.0030 (5)	−0.0011 (4)
C324	0.0246 (6)	0.0127 (5)	0.0149 (5)	−0.0092 (4)	−0.0045 (4)	0.0012 (4)
C325	0.0415 (8)	0.0157 (6)	0.0198 (6)	−0.0140 (5)	0.0058 (5)	−0.0026 (4)
C326	0.0525 (9)	0.0199 (6)	0.0194 (6)	−0.0204 (6)	0.0080 (6)	−0.0007 (5)

Geometric parameters (Å, º)

N1—C2	1.3708 (14)	C12—C13	1.3927 (18)
N1—C7A	1.4091 (14)	C12—H12	0.9500
N1—C1	1.4649 (14)	C13—C14	1.388 (2)
C2—O2	1.2194 (14)	C13—H13	0.9500
C2—C3	1.5549 (15)	C14—C15	1.3848 (19)
C3—O3	1.4263 (13)	C14—H14	0.9500
C3—C3A	1.5105 (15)	C15—C16	1.3933 (17)
C3—C31	1.5311 (14)	C15—H15	0.9500
O3—H3	0.896 (17)	C16—H16	0.9500
C3A—C4	1.3818 (16)	C31—C32	1.5063 (15)
C3A—C7A	1.4011 (15)	C31—H31A	0.9900
C4—C5	1.4022 (16)	C31—H31B	0.9900
C4—H4	0.9500	C32—O32	1.2149 (14)
C5—C6	1.3869 (16)	C32—C324	1.5063 (15)
C5—H5	0.9500	N321—C326	1.3334 (17)
C6—C7	1.4010 (16)	N321—C322	1.3351 (16)
C6—H6	0.9500	C322—C323	1.3842 (17)
C7—C7A	1.3848 (15)	C322—H322	0.9500
C7—H7	0.9500	C323—C324	1.3922 (16)
C1—C11	1.5145 (15)	C323—H323	0.9500
C1—H1A	0.9900	C324—C325	1.3844 (16)
C1—H1B	0.9900	C325—C326	1.3958 (17)
C11—C16	1.3877 (16)	C325—H325	0.9500
C11—C12	1.3943 (17)	C326—H326	0.9500
C2—N1—C7A	110.92 (9)	C13—C12—C11	120.22 (12)
C2—N1—C1	123.95 (9)	C13—C12—H12	119.9
C7A—N1—C1	124.18 (9)	C11—C12—H12	119.9
O2—C2—N1	126.14 (11)	C14—C13—C12	120.00 (12)
O2—C2—C3	125.50 (10)	C14—C13—H13	120.0
N1—C2—C3	108.29 (9)	C12—C13—H13	120.0

O3—C3—C3A	113.12 (9)	C15—C14—C13	120.06 (12)
O3—C3—C31	105.36 (8)	C15—C14—H14	120.0
C3A—C3—C31	116.25 (9)	C13—C14—H14	120.0
O3—C3—C2	108.52 (9)	C14—C15—C16	119.90 (12)
C3A—C3—C2	101.91 (8)	C14—C15—H15	120.1
C31—C3—C2	111.62 (9)	C16—C15—H15	120.1
C3—O3—H3	108.5 (10)	C11—C16—C15	120.51 (11)
C4—C3A—C7A	120.39 (10)	C11—C16—H16	119.7
C4—C3A—C3	130.80 (10)	C15—C16—H16	119.7
C7A—C3A—C3	108.75 (9)	C32—C31—C3	114.81 (9)
C3A—C4—C5	118.37 (10)	C32—C31—H31A	108.6
C3A—C4—H4	120.8	C3—C31—H31A	108.6
C5—C4—H4	120.8	C32—C31—H31B	108.6
C6—C5—C4	120.52 (11)	C3—C31—H31B	108.6
C6—C5—H5	119.7	H31A—C31—H31B	107.5
C4—C5—H5	119.7	O32—C32—C31	122.73 (10)
C5—C6—C7	121.72 (10)	O32—C32—C324	119.07 (10)
C5—C6—H6	119.1	C31—C32—C324	118.19 (9)
C7—C6—H6	119.1	C326—N321—C322	117.27 (11)
C7A—C7—C6	116.88 (10)	N321—C322—C323	123.01 (11)
C7A—C7—H7	121.6	N321—C322—H322	118.5
C6—C7—H7	121.6	C323—C322—H322	118.5
C7—C7A—C3A	122.07 (10)	C322—C323—C324	119.28 (11)
C7—C7A—N1	127.80 (10)	C322—C323—H323	120.4
C3A—C7A—N1	110.12 (9)	C324—C323—H323	120.4
N1—C1—C11	111.15 (9)	C325—C324—C323	118.37 (11)
N1—C1—H1A	109.4	C325—C324—C32	122.62 (10)
C11—C1—H1A	109.4	C323—C324—C32	118.92 (10)
N1—C1—H1B	109.4	C324—C325—C326	117.94 (11)
C11—C1—H1B	109.4	C324—C325—H325	121.0
H1A—C1—H1B	108.0	C326—C325—H325	121.0
C16—C11—C12	119.30 (11)	N321—C326—C325	124.05 (12)
C16—C11—C1	121.32 (11)	N321—C326—H326	118.0
C12—C11—C1	119.37 (10)	C325—C326—H326	118.0

C7A—N1—C2—O2	177.25 (10)	C1—N1—C7A—C3A	169.65 (9)
C1—N1—C2—O2	7.98 (17)	C2—N1—C1—C11	95.90 (12)
C7A—N1—C2—C3	−0.10 (12)	C7A—N1—C1—C11	−71.96 (13)
C1—N1—C2—C3	−169.37 (9)	N1—C1—C11—C16	101.61 (12)
O2—C2—C3—O3	−58.00 (14)	N1—C1—C11—C12	−76.98 (13)
N1—C2—C3—O3	119.37 (9)	C16—C11—C12—C13	−0.76 (19)
O2—C2—C3—C3A	−177.59 (10)	C1—C11—C12—C13	177.86 (12)
N1—C2—C3—C3A	−0.22 (11)	C11—C12—C13—C14	−0.3 (2)
O2—C2—C3—C31	57.67 (14)	C12—C13—C14—C15	0.9 (2)
N1—C2—C3—C31	−124.96 (9)	C13—C14—C15—C16	−0.3 (2)
O3—C3—C3A—C4	61.23 (15)	C12—C11—C16—C15	1.37 (18)
C31—C3—C3A—C4	−60.88 (15)	C1—C11—C16—C15	−177.22 (11)
C2—C3—C3A—C4	177.52 (11)	C14—C15—C16—C11	−0.87 (19)

O3—C3—C3A—C7A	−115.83 (10)	O3—C3—C31—C32	179.42 (9)
C31—C3—C3A—C7A	122.05 (10)	C3A—C3—C31—C32	−54.47 (13)
C2—C3—C3A—C7A	0.46 (11)	C2—C3—C31—C32	61.83 (12)
C7A—C3A—C4—C5	−0.24 (15)	C3—C31—C32—O32	1.12 (17)
C3—C3A—C4—C5	−177.01 (10)	C3—C31—C32—C324	−179.45 (10)
C3A—C4—C5—C6	−1.30 (16)	C326—N321—C322—C323	−1.7 (2)
C4—C5—C6—C7	1.07 (17)	N321—C322—C323—C324	1.9 (2)
C5—C6—C7—C7A	0.70 (16)	C322—C323—C324—C325	0.2 (2)
C6—C7—C7A—C3A	−2.28 (16)	C322—C323—C324—C32	−176.38 (12)
C6—C7—C7A—N1	177.80 (10)	O32—C32—C324—C325	−146.70 (13)
C4—C3A—C7A—C7	2.09 (16)	C31—C32—C324—C325	33.85 (17)
C3—C3A—C7A—C7	179.52 (10)	O32—C32—C324—C323	29.72 (18)
C4—C3A—C7A—N1	−177.98 (9)	C31—C32—C324—C323	−149.73 (12)
C3—C3A—C7A—N1	−0.55 (12)	C323—C324—C325—C326	−2.3 (2)
C2—N1—C7A—C7	−179.66 (10)	C32—C324—C325—C326	174.14 (13)
C1—N1—C7A—C7	−10.42 (17)	C322—N321—C326—C325	−0.6 (2)
C2—N1—C7A—C3A	0.41 (12)	C324—C325—C326—N321	2.7 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···N321^{i}	0.897 (17)	1.897 (17)	2.7915 (14)	174.9 (15)
C4—H4···O3^{ii}	0.95	2.46	3.3842 (14)	164
C7—H7···O32^{iii}	0.95	2.51	3.3719 (14)	150
C325—H325···O2^{iv}	0.95	2.32	3.2578 (15)	171
C322—H322···*Cg*1^{v}	0.95	2.68	3.5294 (14)	149

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

3-[2-(Benzo[*d*][1,3]dioxol-5-yl)-2-oxoethyl]-1-benzyl-3-hydroxyindolin-2-one (If)

Crystal data

$C_{24}H_{19}NO_5$
M_r = 401.40
Triclinic, $P\overline{1}$
a = 11.8136 (7) Å
b = 12.4987 (10) Å
c = 13.5976 (11) Å
α = 93.084 (3)°
β = 101.883 (2)°
γ = 95.055 (2)°
V = 1951.7 (3) Å^3

Z = 4
F(000) = 840
D_x = 1.366 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9692 reflections
θ = 2.2–28.3°
μ = 0.10 mm^{-1}
T = 100 K
Plate, yellow
0.25 × 0.16 × 0.06 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.957, T_{max} = 0.994
125792 measured reflections
9691 independent reflections
8134 reflections with $I > 2\sigma(I)$
R_{int} = 0.049

θ_{max} = 28.3°, θ_{min} = 2.2°
h = −13→15
k = −16→16
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.106
S = 1.08
9691 reflections
547 parameters
0 restraints
Primary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0386P)^2 + 1.0922P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 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	0.36578 (9)	0.70223 (8)	0.75521 (8)	0.0180 (2)
C12	0.33088 (10)	0.64863 (9)	0.66308 (9)	0.0163 (2)
O12	0.39483 (7)	0.62008 (7)	0.60785 (7)	0.02009 (18)
C13	0.19674 (10)	0.63051 (9)	0.63676 (9)	0.0155 (2)
O13	0.15366 (8)	0.68949 (7)	0.55269 (7)	0.01875 (17)
H13	0.1495 (15)	0.6486 (14)	0.4976 (13)	0.028*
C13A	0.16621 (10)	0.68140 (9)	0.72932 (9)	0.0162 (2)
C14	0.05913 (11)	0.69289 (10)	0.75156 (9)	0.0198 (2)
H14	−0.0104	0.6659	0.7051	0.024*
C15	0.05531 (11)	0.74530 (10)	0.84426 (10)	0.0223 (2)
H15	−0.0176	0.7544	0.8610	0.027*
C16	0.15748 (12)	0.78406 (10)	0.91178 (10)	0.0222 (2)
H16	0.1533	0.8188	0.9746	0.027*
C17	0.26690 (11)	0.77310 (10)	0.88929 (9)	0.0203 (2)
H17	0.3368	0.7999	0.9354	0.024*
C17A	0.26833 (10)	0.72162 (9)	0.79709 (9)	0.0170 (2)
C11	0.48659 (10)	0.72091 (10)	0.80967 (10)	0.0207 (2)
H11A	0.5360	0.6858	0.7697	0.025*
H11B	0.4943	0.6865	0.8743	0.025*
C111	0.53080 (10)	0.83877 (10)	0.83120 (10)	0.0197 (2)
C112	0.52255 (12)	0.90761 (11)	0.75373 (10)	0.0259 (3)
H112	0.4860	0.8815	0.6868	0.031*
C113	0.56794 (13)	1.01485 (12)	0.77435 (12)	0.0317 (3)
H113	0.5618	1.0620	0.7214	0.038*
C114	0.62220 (13)	1.05348 (12)	0.87201 (12)	0.0328 (3)
H114	0.6536	1.1267	0.8857	0.039*

C115	0.63031 (13)	0.98508 (12)	0.94904 (11)	0.0306 (3)
H115	0.6675	1.0111	1.0158	0.037*
C116	0.58414 (11)	0.87823 (11)	0.92887 (10)	0.0244 (3)
H116	0.5890	0.8317	0.9822	0.029*
C131	0.15502 (10)	0.51079 (9)	0.61318 (9)	0.0160 (2)
H13A	0.0695	0.5013	0.6056	0.019*
H13B	0.1726	0.4874	0.5477	0.019*
C132	0.20854 (10)	0.43795 (9)	0.69182 (9)	0.0160 (2)
O132	0.27327 (8)	0.47573 (7)	0.77033 (7)	0.02184 (19)
O141	0.12237 (10)	−0.00809 (8)	0.62667 (9)	0.0338 (2)
C142	0.20004 (14)	−0.03580 (11)	0.71514 (12)	0.0309 (3)
H14A	0.1567	−0.0811	0.7554	0.037*
H14B	0.2609	−0.0770	0.6958	0.037*
O143	0.25219 (10)	0.06174 (8)	0.77321 (8)	0.0332 (2)
C14A	0.20977 (11)	0.14395 (10)	0.71810 (10)	0.0218 (2)
C144	0.23604 (11)	0.25218 (10)	0.74070 (9)	0.0195 (2)
H144	0.2886	0.2805	0.8010	0.023*
C145	0.18104 (10)	0.32004 (9)	0.66977 (9)	0.0167 (2)
C146	0.10375 (10)	0.27679 (10)	0.58197 (9)	0.0195 (2)
H146	0.0680	0.3242	0.5355	0.023*
C147	0.07750 (11)	0.16566 (10)	0.56043 (10)	0.0232 (3)
H147	0.0248	0.1360	0.5008	0.028*
C18A	0.13246 (11)	0.10199 (10)	0.63066 (10)	0.0230 (3)
N21	0.14702 (8)	0.57608 (8)	0.21220 (8)	0.0171 (2)
C22	0.18051 (10)	0.56438 (9)	0.31195 (9)	0.0164 (2)
O22	0.11699 (7)	0.56189 (7)	0.37299 (6)	0.01978 (18)
C23	0.31364 (10)	0.55891 (9)	0.33838 (9)	0.0153 (2)
O23	0.36630 (7)	0.65315 (7)	0.40061 (7)	0.01939 (18)
H23	0.3659 (15)	0.6426 (14)	0.4653 (14)	0.029*
C23A	0.34379 (10)	0.56497 (9)	0.23629 (9)	0.0171 (2)
C24	0.44927 (11)	0.56227 (10)	0.20825 (10)	0.0207 (2)
H24	0.5185	0.5574	0.2570	0.025*
C25	0.45181 (12)	0.56686 (11)	0.10592 (10)	0.0238 (3)
H25	0.5235	0.5650	0.0849	0.029*
C26	0.35020 (12)	0.57406 (11)	0.03520 (10)	0.0245 (3)
H26	0.3532	0.5753	−0.0340	0.029*
C27	0.24316 (11)	0.57957 (10)	0.06365 (10)	0.0215 (2)
H27	0.1740	0.5866	0.0155	0.026*
C27A	0.24281 (10)	0.57436 (9)	0.16460 (9)	0.0175 (2)
C21	0.02682 (10)	0.57967 (10)	0.16062 (9)	0.0199 (2)
H21A	−0.0246	0.5556	0.2059	0.024*
H21B	0.0097	0.5281	0.1003	0.024*
C211	−0.00175 (11)	0.68995 (10)	0.12808 (9)	0.0207 (2)
C212	0.05811 (13)	0.78375 (11)	0.17857 (11)	0.0283 (3)
H212	0.1215	0.7799	0.2336	0.034*
C213	0.02605 (15)	0.88369 (13)	0.14936 (13)	0.0378 (4)
H213	0.0680	0.9478	0.1841	0.045*
C214	−0.06704 (15)	0.88994 (13)	0.06965 (13)	0.0372 (4)

H214	−0.0893	0.9582	0.0500	0.045*
C215	−0.12708 (13)	0.79678 (14)	0.01898 (12)	0.0337 (3)
H215	−0.1907	0.8009	−0.0358	0.040*
C216	−0.09496 (12)	0.69690 (12)	0.04770 (10)	0.0271 (3)
H216	−0.1367	0.6330	0.0124	0.032*
C231	0.34489 (10)	0.45854 (9)	0.39264 (9)	0.0168 (2)
H23A	0.4296	0.4544	0.4009	0.020*
H23B	0.3279	0.4665	0.4608	0.020*
C232	0.28175 (10)	0.35343 (9)	0.34022 (9)	0.0170 (2)
O232	0.21756 (8)	0.35194 (7)	0.25714 (7)	0.02298 (19)
O241	0.34212 (10)	−0.03455 (9)	0.51919 (9)	0.0387 (3)
C242	0.25730 (16)	−0.10257 (12)	0.44568 (13)	0.0373 (4)
H24A	0.2936	−0.1640	0.4205	0.045*
H24B	0.1930	−0.1314	0.4764	0.045*
O243	0.21368 (12)	−0.03916 (9)	0.36449 (9)	0.0452 (3)
C24A	0.25954 (13)	0.06508 (11)	0.39521 (11)	0.0274 (3)
C244	0.23952 (12)	0.15577 (10)	0.34531 (10)	0.0246 (3)
H244	0.1874	0.1532	0.2817	0.030*
C245	0.29995 (10)	0.25330 (10)	0.39278 (9)	0.0189 (2)
C246	0.37567 (11)	0.25505 (11)	0.48632 (10)	0.0235 (3)
H246	0.4147	0.3219	0.5173	0.028*
C247	0.39570 (12)	0.16122 (12)	0.53564 (11)	0.0288 (3)
H247	0.4477	0.1624	0.5991	0.035*
C28A	0.33634 (12)	0.06743 (11)	0.48759 (11)	0.0276 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N11	0.0149 (5)	0.0179 (5)	0.0202 (5)	0.0007 (4)	0.0027 (4)	−0.0021 (4)
C12	0.0160 (5)	0.0135 (5)	0.0192 (5)	0.0009 (4)	0.0035 (4)	0.0016 (4)
O12	0.0175 (4)	0.0222 (4)	0.0212 (4)	0.0025 (3)	0.0059 (3)	−0.0015 (3)
C13	0.0149 (5)	0.0142 (5)	0.0172 (5)	0.0021 (4)	0.0026 (4)	0.0016 (4)
O13	0.0225 (4)	0.0166 (4)	0.0171 (4)	0.0033 (3)	0.0028 (3)	0.0037 (3)
C13A	0.0177 (5)	0.0129 (5)	0.0183 (5)	0.0017 (4)	0.0043 (4)	0.0019 (4)
C14	0.0191 (6)	0.0185 (6)	0.0224 (6)	0.0023 (4)	0.0053 (5)	0.0025 (5)
C15	0.0222 (6)	0.0225 (6)	0.0250 (6)	0.0047 (5)	0.0100 (5)	0.0024 (5)
C16	0.0287 (6)	0.0199 (6)	0.0199 (6)	0.0050 (5)	0.0085 (5)	0.0003 (5)
C17	0.0233 (6)	0.0175 (6)	0.0196 (6)	0.0031 (4)	0.0034 (5)	−0.0001 (4)
C17A	0.0181 (5)	0.0138 (5)	0.0198 (6)	0.0027 (4)	0.0047 (4)	0.0020 (4)
C11	0.0155 (5)	0.0194 (6)	0.0250 (6)	0.0017 (4)	0.0001 (4)	−0.0023 (5)
C111	0.0145 (5)	0.0202 (6)	0.0233 (6)	0.0013 (4)	0.0024 (4)	−0.0025 (5)
C112	0.0248 (6)	0.0262 (7)	0.0243 (6)	−0.0008 (5)	0.0013 (5)	0.0002 (5)
C113	0.0323 (7)	0.0253 (7)	0.0360 (8)	−0.0004 (6)	0.0042 (6)	0.0077 (6)
C114	0.0305 (7)	0.0208 (6)	0.0430 (8)	−0.0030 (5)	0.0020 (6)	−0.0030 (6)
C115	0.0298 (7)	0.0268 (7)	0.0298 (7)	−0.0009 (5)	−0.0020 (6)	−0.0082 (6)
C116	0.0224 (6)	0.0240 (6)	0.0243 (6)	0.0017 (5)	0.0004 (5)	−0.0010 (5)
C131	0.0160 (5)	0.0139 (5)	0.0174 (5)	0.0002 (4)	0.0026 (4)	0.0014 (4)
C132	0.0147 (5)	0.0156 (5)	0.0182 (5)	0.0015 (4)	0.0043 (4)	0.0008 (4)

O132	0.0234 (4)	0.0188 (4)	0.0205 (4)	0.0021 (3)	−0.0014 (3)	−0.0002 (3)
O141	0.0399 (6)	0.0142 (4)	0.0427 (6)	0.0034 (4)	−0.0021 (5)	0.0012 (4)
C142	0.0380 (8)	0.0169 (6)	0.0371 (8)	0.0049 (5)	0.0050 (6)	0.0046 (5)
O143	0.0433 (6)	0.0166 (5)	0.0360 (6)	0.0068 (4)	−0.0028 (5)	0.0058 (4)
C14A	0.0238 (6)	0.0178 (6)	0.0246 (6)	0.0057 (5)	0.0048 (5)	0.0055 (5)
C144	0.0207 (6)	0.0174 (6)	0.0201 (6)	0.0031 (4)	0.0033 (4)	0.0018 (4)
C145	0.0161 (5)	0.0145 (5)	0.0203 (6)	0.0021 (4)	0.0054 (4)	0.0014 (4)
C146	0.0187 (5)	0.0172 (6)	0.0217 (6)	0.0021 (4)	0.0021 (4)	0.0012 (4)
C147	0.0228 (6)	0.0179 (6)	0.0261 (6)	0.0009 (5)	0.0000 (5)	−0.0017 (5)
C18A	0.0241 (6)	0.0136 (5)	0.0311 (7)	0.0011 (4)	0.0062 (5)	−0.0005 (5)
N21	0.0142 (4)	0.0196 (5)	0.0177 (5)	0.0031 (4)	0.0030 (4)	0.0021 (4)
C22	0.0159 (5)	0.0134 (5)	0.0197 (5)	0.0016 (4)	0.0032 (4)	0.0001 (4)
O22	0.0171 (4)	0.0234 (4)	0.0193 (4)	0.0019 (3)	0.0056 (3)	0.0003 (3)
C23	0.0138 (5)	0.0147 (5)	0.0168 (5)	0.0008 (4)	0.0027 (4)	−0.0006 (4)
O23	0.0199 (4)	0.0165 (4)	0.0200 (4)	−0.0012 (3)	0.0027 (3)	−0.0028 (3)
C23A	0.0178 (5)	0.0147 (5)	0.0189 (5)	0.0021 (4)	0.0044 (4)	0.0009 (4)
C24	0.0186 (6)	0.0215 (6)	0.0231 (6)	0.0038 (4)	0.0061 (5)	0.0013 (5)
C25	0.0234 (6)	0.0253 (6)	0.0263 (6)	0.0058 (5)	0.0116 (5)	0.0042 (5)
C26	0.0295 (7)	0.0260 (6)	0.0214 (6)	0.0073 (5)	0.0104 (5)	0.0055 (5)
C27	0.0230 (6)	0.0220 (6)	0.0203 (6)	0.0048 (5)	0.0047 (5)	0.0038 (5)
C27A	0.0176 (5)	0.0157 (5)	0.0200 (6)	0.0027 (4)	0.0051 (4)	0.0020 (4)
C21	0.0145 (5)	0.0228 (6)	0.0215 (6)	0.0028 (4)	0.0011 (4)	0.0031 (5)
C211	0.0194 (6)	0.0244 (6)	0.0209 (6)	0.0069 (5)	0.0077 (5)	0.0044 (5)
C212	0.0291 (7)	0.0243 (7)	0.0311 (7)	0.0063 (5)	0.0033 (5)	0.0029 (5)
C213	0.0435 (9)	0.0242 (7)	0.0474 (9)	0.0090 (6)	0.0111 (7)	0.0038 (6)
C214	0.0438 (9)	0.0340 (8)	0.0428 (9)	0.0223 (7)	0.0187 (7)	0.0170 (7)
C215	0.0315 (7)	0.0456 (9)	0.0301 (7)	0.0211 (7)	0.0104 (6)	0.0137 (6)
C216	0.0227 (6)	0.0358 (8)	0.0243 (6)	0.0108 (5)	0.0045 (5)	0.0050 (5)
C231	0.0158 (5)	0.0163 (5)	0.0180 (5)	0.0029 (4)	0.0022 (4)	0.0007 (4)
C232	0.0158 (5)	0.0174 (5)	0.0181 (5)	0.0019 (4)	0.0044 (4)	0.0009 (4)
O232	0.0244 (4)	0.0205 (4)	0.0211 (4)	−0.0006 (3)	−0.0010 (3)	0.0021 (3)
O241	0.0433 (6)	0.0249 (5)	0.0484 (7)	0.0041 (5)	0.0066 (5)	0.0181 (5)
C242	0.0525 (10)	0.0191 (7)	0.0439 (9)	0.0049 (6)	0.0160 (7)	0.0100 (6)
O243	0.0675 (8)	0.0177 (5)	0.0440 (7)	−0.0068 (5)	0.0000 (6)	0.0076 (5)
C24A	0.0341 (7)	0.0172 (6)	0.0315 (7)	−0.0007 (5)	0.0095 (6)	0.0029 (5)
C244	0.0295 (7)	0.0194 (6)	0.0235 (6)	0.0002 (5)	0.0033 (5)	0.0019 (5)
C245	0.0181 (5)	0.0172 (6)	0.0223 (6)	0.0024 (4)	0.0057 (4)	0.0033 (4)
C246	0.0189 (6)	0.0231 (6)	0.0269 (6)	0.0012 (5)	0.0011 (5)	0.0048 (5)
C247	0.0238 (6)	0.0294 (7)	0.0315 (7)	0.0031 (5)	−0.0003 (5)	0.0118 (6)
C28A	0.0279 (7)	0.0228 (6)	0.0356 (7)	0.0062 (5)	0.0106 (6)	0.0125 (5)

Geometric parameters (Å, º)

N11—C12	1.3551 (15)	N21—C22	1.3522 (15)
N11—C17A	1.4186 (15)	N21—C27A	1.4158 (15)
N11—C11	1.4585 (15)	N21—C21	1.4542 (15)
C12—O12	1.2306 (14)	C22—O22	1.2280 (14)
C12—C13	1.5444 (16)	C22—C23	1.5480 (16)

C13—O13	1.4235 (14)	C23—O23	1.4273 (14)
C13—C13A	1.5033 (16)	C23—C23A	1.5066 (16)
C13—C131	1.5291 (16)	C23—C231	1.5243 (16)
O13—H13	0.874 (18)	O23—H23	0.898 (18)
C13A—C14	1.3774 (16)	C23A—C24	1.3788 (16)
C13A—C17A	1.3939 (16)	C23A—C27A	1.3947 (16)
C14—C15	1.3999 (18)	C24—C25	1.4020 (18)
C14—H14	0.9500	C24—H24	0.9500
C15—C16	1.3883 (19)	C25—C26	1.3881 (19)
C15—H15	0.9500	C25—H25	0.9500
C16—C17	1.4045 (17)	C26—C27	1.4027 (18)
C16—H16	0.9500	C26—H26	0.9500
C17—C17A	1.3820 (17)	C27—C27A	1.3785 (17)
C17—H17	0.9500	C27—H27	0.9500
C11—C111	1.5111 (17)	C21—C211	1.5152 (17)
C11—H11A	0.9900	C21—H21A	0.9900
C11—H11B	0.9900	C21—H21B	0.9900
C111—C112	1.3892 (19)	C211—C212	1.3831 (19)
C111—C116	1.3907 (18)	C211—C216	1.3951 (18)
C112—C113	1.391 (2)	C212—C213	1.392 (2)
C112—H112	0.9500	C212—H212	0.9500
C113—C114	1.391 (2)	C213—C214	1.387 (2)
C113—H113	0.9500	C213—H213	0.9500
C114—C115	1.381 (2)	C214—C215	1.379 (2)
C114—H114	0.9500	C214—H214	0.9500
C115—C116	1.3882 (19)	C215—C216	1.389 (2)
C115—H115	0.9500	C215—H215	0.9500
C116—H116	0.9500	C216—H216	0.9500
C131—C132	1.5166 (16)	C231—C232	1.5158 (16)
C131—H13A	0.9900	C231—H23A	0.9900
C131—H13B	0.9900	C231—H23B	0.9900
C132—O132	1.2221 (15)	C232—O232	1.2223 (15)
C132—C145	1.4816 (16)	C232—C245	1.4846 (16)
O141—C18A	1.3676 (15)	O241—C28A	1.3697 (16)
O141—C142	1.4336 (18)	O241—C242	1.440 (2)
C142—O143	1.4328 (17)	C242—O243	1.4297 (19)
C142—H14A	0.9900	C242—H24A	0.9900
C142—H14B	0.9900	C242—H24B	0.9900
O143—C14A	1.3738 (15)	O243—C24A	1.3745 (17)
C14A—C144	1.3649 (17)	C24A—C244	1.3657 (19)
C14A—C18A	1.3872 (19)	C24A—C28A	1.387 (2)
C144—C145	1.4138 (16)	C244—C245	1.4087 (18)
C144—H144	0.9500	C244—H244	0.9500
C145—C146	1.3950 (17)	C245—C246	1.3940 (18)
C146—C147	1.3979 (17)	C246—C247	1.3961 (18)
C146—H146	0.9500	C246—H246	0.9500
C147—C18A	1.3727 (18)	C247—C28A	1.370 (2)
C147—H147	0.9500	C247—H247	0.9500

C12—N11—C17A	110.46 (10)	C22—N21—C27A	110.76 (10)
C12—N11—C11	124.04 (10)	C22—N21—C21	123.82 (10)
C17A—N11—C11	124.77 (10)	C27A—N21—C21	125.18 (10)
O12—C12—N11	126.00 (11)	O22—C22—N21	125.99 (11)
O12—C12—C13	124.91 (11)	O22—C22—C23	124.97 (11)
N11—C12—C13	109.08 (10)	N21—C22—C23	109.00 (10)
O13—C13—C13A	109.04 (9)	O23—C23—C23A	109.13 (9)
O13—C13—C131	110.14 (9)	O23—C23—C231	109.93 (9)
C13A—C13—C131	115.27 (10)	C23A—C23—C231	115.70 (9)
O13—C13—C12	109.43 (9)	O23—C23—C22	108.55 (9)
C13A—C13—C12	101.69 (9)	C23A—C23—C22	101.49 (9)
C131—C13—C12	110.90 (9)	C231—C23—C22	111.59 (9)
C13—O13—H13	108.4 (11)	C23—O23—H23	109.7 (11)
C14—C13A—C17A	120.87 (11)	C24—C23A—C27A	120.57 (11)
C14—C13A—C13	130.11 (11)	C24—C23A—C23	130.41 (11)
C17A—C13A—C13	109.02 (10)	C27A—C23A—C23	109.02 (10)
C13A—C14—C15	118.39 (12)	C23A—C24—C25	118.35 (12)
C13A—C14—H14	120.8	C23A—C24—H24	120.8
C15—C14—H14	120.8	C25—C24—H24	120.8
C16—C15—C14	120.37 (12)	C26—C25—C24	120.40 (12)
C16—C15—H15	119.8	C26—C25—H25	119.8
C14—C15—H15	119.8	C24—C25—H25	119.8
C15—C16—C17	121.50 (12)	C25—C26—C27	121.46 (12)
C15—C16—H16	119.2	C25—C26—H26	119.3
C17—C16—H16	119.2	C27—C26—H26	119.3
C17A—C17—C16	117.02 (12)	C27A—C27—C26	117.03 (12)
C17A—C17—H17	121.5	C27A—C27—H27	121.5
C16—C17—H17	121.5	C26—C27—H27	121.5
C17—C17A—C13A	121.86 (11)	C27—C27A—C23A	122.16 (11)
C17—C17A—N11	128.41 (11)	C27—C27A—N21	128.18 (11)
C13A—C17A—N11	109.73 (10)	C23A—C27A—N21	109.66 (10)
N11—C11—C111	113.60 (10)	N21—C21—C211	114.11 (10)
N11—C11—H11A	108.8	N21—C21—H21A	108.7
C111—C11—H11A	108.8	C211—C21—H21A	108.7
N11—C11—H11B	108.8	N21—C21—H21B	108.7
C111—C11—H11B	108.8	C211—C21—H21B	108.7
H11A—C11—H11B	107.7	H21A—C21—H21B	107.6
C112—C111—C116	119.45 (12)	C212—C211—C216	119.10 (12)
C112—C111—C11	120.65 (11)	C212—C211—C21	122.04 (12)
C116—C111—C11	119.86 (12)	C216—C211—C21	118.79 (12)
C111—C112—C113	119.85 (13)	C211—C212—C213	120.46 (14)
C111—C112—H112	120.1	C211—C212—H212	119.8
C113—C112—H112	120.1	C213—C212—H212	119.8
C114—C113—C112	120.38 (14)	C214—C213—C212	120.11 (15)
C114—C113—H113	119.8	C214—C213—H213	119.9
C112—C113—H113	119.8	C212—C213—H213	119.9
C115—C114—C113	119.77 (13)	C215—C214—C213	119.74 (14)

C115—C114—H114	120.1	C215—C214—H214	120.1
C113—C114—H114	120.1	C213—C214—H214	120.1
C114—C115—C116	120.00 (13)	C214—C215—C216	120.27 (14)
C114—C115—H115	120.0	C214—C215—H215	119.9
C116—C115—H115	120.0	C216—C215—H215	119.9
C115—C116—C111	120.55 (13)	C215—C216—C211	120.32 (14)
C115—C116—H116	119.7	C215—C216—H216	119.8
C111—C116—H116	119.7	C211—C216—H216	119.8
C132—C131—C13	114.79 (9)	C232—C231—C23	115.06 (10)
C132—C131—H13A	108.6	C232—C231—H23A	108.5
C13—C131—H13A	108.6	C23—C231—H23A	108.5
C132—C131—H13B	108.6	C232—C231—H23B	108.5
C13—C131—H13B	108.6	C23—C231—H23B	108.5
H13A—C131—H13B	107.5	H23A—C231—H23B	107.5
O132—C132—C145	121.30 (11)	O232—C232—C245	121.35 (11)
O132—C132—C131	120.76 (10)	O232—C232—C231	120.52 (11)
C145—C132—C131	117.93 (10)	C245—C232—C231	118.12 (10)
C18A—O141—C142	105.91 (11)	C28A—O241—C242	105.66 (12)
O143—C142—O141	108.36 (10)	O243—C242—O241	107.99 (11)
O143—C142—H14A	110.0	O243—C242—H24A	110.1
O141—C142—H14A	110.0	O241—C242—H24A	110.1
O143—C142—H14B	110.0	O243—C242—H24B	110.1
O141—C142—H14B	110.0	O241—C242—H24B	110.1
H14A—C142—H14B	108.4	H24A—C242—H24B	108.4
C14A—O143—C142	105.69 (11)	C24A—O243—C242	105.97 (12)
C144—C14A—O143	127.95 (12)	C244—C24A—O243	128.03 (14)
C144—C14A—C18A	122.10 (12)	C244—C24A—C28A	122.26 (13)
O143—C14A—C18A	109.95 (11)	O243—C24A—C28A	109.68 (12)
C14A—C144—C145	116.55 (11)	C24A—C244—C245	116.73 (12)
C14A—C144—H144	121.7	C24A—C244—H244	121.6
C145—C144—H144	121.7	C245—C244—H244	121.6
C146—C145—C144	120.77 (11)	C246—C245—C244	120.57 (12)
C146—C145—C132	121.42 (11)	C246—C245—C232	121.11 (11)
C144—C145—C132	117.81 (11)	C244—C245—C232	118.32 (11)
C145—C146—C147	121.82 (11)	C245—C246—C247	121.78 (13)
C145—C146—H146	119.1	C245—C246—H246	119.1
C147—C146—H146	119.1	C247—C246—H246	119.1
C18A—C147—C146	116.00 (12)	C28A—C247—C246	116.47 (13)
C18A—C147—H147	122.0	C28A—C247—H247	121.8
C146—C147—H147	122.0	C246—C247—H247	121.8
O141—C18A—C147	127.20 (12)	O241—C28A—C247	127.70 (14)
O141—C18A—C14A	110.03 (11)	O241—C28A—C24A	110.12 (13)
C147—C18A—C14A	122.77 (12)	C247—C28A—C24A	122.18 (12)
C17A—N11—C12—O12	−179.90 (11)	C27A—N21—C22—O22	−179.43 (11)
C11—N11—C12—O12	−9.28 (19)	C21—N21—C22—O22	−4.74 (19)
C17A—N11—C12—C13	1.18 (13)	C27A—N21—C22—C23	2.74 (13)
C11—N11—C12—C13	171.80 (10)	C21—N21—C22—C23	177.43 (10)

O12—C12—C13—O13	−64.35 (15)	O22—C22—C23—O23	−65.20 (14)
N11—C12—C13—O13	114.58 (11)	N21—C22—C23—O23	112.66 (11)
O12—C12—C13—C13A	−179.59 (11)	O22—C22—C23—C23A	179.92 (11)
N11—C12—C13—C13A	−0.65 (12)	N21—C22—C23—C23A	−2.23 (12)
O12—C12—C13—C131	57.35 (15)	O22—C22—C23—C231	56.11 (15)
N11—C12—C13—C131	−123.72 (10)	N21—C22—C23—C231	−126.04 (10)
O13—C13—C13A—C14	63.36 (16)	O23—C23—C23A—C24	66.66 (16)
C131—C13—C13A—C14	−61.09 (16)	C231—C23—C23A—C24	−57.91 (17)
C12—C13—C13A—C14	178.88 (12)	C22—C23—C23A—C24	−178.88 (12)
O13—C13—C13A—C17A	−115.62 (10)	O23—C23—C23A—C27A	−113.52 (11)
C131—C13—C13A—C17A	119.92 (11)	C231—C23—C23A—C27A	121.91 (11)
C12—C13—C13A—C17A	−0.11 (12)	C22—C23—C23A—C27A	0.94 (12)
C17A—C13A—C14—C15	−0.45 (18)	C27A—C23A—C24—C25	−1.35 (18)
C13—C13A—C14—C15	−179.34 (12)	C23—C23A—C24—C25	178.45 (12)
C13A—C14—C15—C16	−0.27 (19)	C23A—C24—C25—C26	0.07 (19)
C14—C15—C16—C17	0.6 (2)	C24—C25—C26—C27	1.6 (2)
C15—C16—C17—C17A	−0.26 (19)	C25—C26—C27—C27A	−1.8 (2)
C16—C17—C17A—C13A	−0.47 (18)	C26—C27—C27A—C23A	0.54 (19)
C16—C17—C17A—N11	178.49 (11)	C26—C27—C27A—N21	−178.70 (12)
C14—C13A—C17A—C17	0.84 (18)	C24—C23A—C27A—C27	1.06 (19)
C13—C13A—C17A—C17	179.94 (11)	C23—C23A—C27A—C27	−178.78 (11)
C14—C13A—C17A—N11	−178.29 (11)	C24—C23A—C27A—N21	−179.58 (11)
C13—C13A—C17A—N11	0.80 (13)	C23—C23A—C27A—N21	0.58 (13)
C12—N11—C17A—C17	179.67 (12)	C22—N21—C27A—C27	177.17 (12)
C11—N11—C17A—C17	9.12 (19)	C21—N21—C27A—C27	2.6 (2)
C12—N11—C17A—C13A	−1.27 (14)	C22—N21—C27A—C23A	−2.14 (14)
C11—N11—C17A—C13A	−171.81 (11)	C21—N21—C27A—C23A	−176.75 (11)
C12—N11—C11—C111	119.11 (13)	C22—N21—C21—C211	108.60 (13)
C17A—N11—C11—C111	−71.60 (15)	C27A—N21—C21—C211	−77.47 (15)
N11—C11—C111—C112	−54.19 (16)	N21—C21—C211—C212	−27.57 (17)
N11—C11—C111—C116	128.16 (12)	N21—C21—C211—C216	155.57 (11)
C116—C111—C112—C113	0.3 (2)	C216—C211—C212—C213	−0.2 (2)
C11—C111—C112—C113	−177.40 (12)	C21—C211—C212—C213	−177.07 (13)
C111—C112—C113—C114	0.4 (2)	C211—C212—C213—C214	0.5 (2)
C112—C113—C114—C115	−0.5 (2)	C212—C213—C214—C215	−0.5 (2)
C113—C114—C115—C116	−0.2 (2)	C213—C214—C215—C216	0.2 (2)
C114—C115—C116—C111	0.8 (2)	C214—C215—C216—C211	0.1 (2)
C112—C111—C116—C115	−0.9 (2)	C212—C211—C216—C215	−0.1 (2)
C11—C111—C116—C115	176.81 (12)	C21—C211—C216—C215	176.90 (12)
O13—C13—C131—C132	172.21 (9)	O23—C23—C231—C232	172.42 (9)
C13A—C13—C131—C132	−63.91 (13)	C23A—C23—C231—C232	−63.42 (13)
C12—C13—C131—C132	50.92 (13)	C22—C23—C231—C232	51.93 (13)
C13—C131—C132—O132	4.93 (16)	C23—C231—C232—O232	4.98 (16)
C13—C131—C132—C145	−174.30 (10)	C23—C231—C232—C245	−174.80 (10)
C18A—O141—C142—O143	2.44 (16)	C28A—O241—C242—O243	7.40 (17)
O141—C142—O143—C14A	−2.36 (15)	O241—C242—O243—C24A	−7.49 (17)
C142—O143—C14A—C144	−178.25 (14)	C242—O243—C24A—C244	−177.11 (15)
C142—O143—C14A—C18A	1.39 (15)	C242—O243—C24A—C28A	4.78 (17)

O143—C14A—C144—C145	179.07 (12)	O243—C24A—C244—C245	−178.62 (14)
C18A—C14A—C144—C145	−0.52 (19)	C28A—C24A—C244—C245	−0.7 (2)
C14A—C144—C145—C146	0.32 (17)	C24A—C244—C245—C246	−0.24 (19)
C14A—C144—C145—C132	−179.53 (11)	C24A—C244—C245—C232	179.15 (12)
O132—C132—C145—C146	177.53 (11)	O232—C232—C245—C246	178.27 (12)
C131—C132—C145—C146	−3.24 (16)	C231—C232—C245—C246	−1.95 (17)
O132—C132—C145—C144	−2.62 (17)	O232—C232—C245—C244	−1.13 (18)
C131—C132—C145—C144	176.61 (10)	C231—C232—C245—C244	178.65 (11)
C144—C145—C146—C147	0.00 (19)	C244—C245—C246—C247	0.9 (2)
C132—C145—C146—C147	179.85 (11)	C232—C245—C246—C247	−178.52 (12)
C145—C146—C147—C18A	−0.13 (19)	C245—C246—C247—C28A	−0.5 (2)
C142—O141—C18A—C147	177.74 (14)	C242—O241—C28A—C247	176.00 (15)
C142—O141—C18A—C14A	−1.60 (15)	C242—O241—C28A—C24A	−4.52 (16)
C146—C147—C18A—O141	−179.34 (13)	C246—C247—C28A—O241	178.93 (14)
C146—C147—C18A—C14A	−0.1 (2)	C246—C247—C28A—C24A	−0.5 (2)
C144—C14A—C18A—O141	179.80 (12)	C244—C24A—C28A—O241	−178.39 (13)
O143—C14A—C18A—O141	0.14 (16)	O243—C24A—C28A—O241	−0.14 (17)
C144—C14A—C18A—C147	0.4 (2)	C244—C24A—C28A—C247	1.1 (2)
O143—C14A—C18A—C147	−179.24 (12)	O243—C24A—C28A—C247	179.37 (14)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O13—H13···O22	0.874 (17)	1.912 (17)	2.7794 (12)	171.1 (17)
O23—H23···O12	0.897 (18)	1.938 (19)	2.8256 (13)	169.8 (17)
C131—H13*A*···O22^{i}	0.99	2.35	3.3075 (16)	161
C147—H147···O141^{ii}	0.95	2.56	3.4776 (18)	163
C231—H23*A*···O12^{iii}	0.99	2.37	3.3107 (15)	159
C242—H24*A*···O23^{iv}	0.99	2.53	3.4889 (19)	163
C142—H14*A*···*Cg*3^{iv}	0.99	2.53	3.3289 (15)	137

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

(*E*)-1-Benzyl-3-[2-(4-fluorophenyl)-2-oxoethylidene]indolin-2-one (IIa)

Crystal data

$C_{23}H_{16}FNO_2$
M_r = 357.37
Monoclinic, $P2_1/c$
a = 7.6021 (6) Å
b = 20.4880 (13) Å
c = 10.9319 (7) Å
β = 96.986 (3)°
V = 1690.0 (2) Å^3
Z = 4
$F(000)$ = 744
D_x = 1.405 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3896 reflections
θ = 2.1–27.6°
μ = 0.10 mm^{-1}
T = 100 K
Block, red
0.14 × 0.14 × 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} = 0.917, T_{max} = 0.990
3896 measured reflections
3896 independent reflections
2995 reflections with $I > 2\sigma(I)$
θ_{max} = 27.6°, θ_{min} = 2.1°
h = −9→9
k = 0→26
l = 0→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.134
S = 1.05
3896 reflections
245 parameters
0 restraints
Primary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0606P)^2 + 0.910P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 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.

Refinement. Refined as a 2-component twin.

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

	x	y	z	U_{iso}*/U_{eq}
N1	0.6199 (2)	0.31996 (8)	0.37330 (16)	0.0156 (4)
C2	0.7010 (3)	0.34442 (10)	0.4817 (2)	0.0164 (4)
O2	0.7641 (2)	0.31249 (7)	0.57079 (14)	0.0230 (4)
C3	0.6989 (3)	0.41837 (10)	0.46925 (19)	0.0148 (4)
C3A	0.6043 (3)	0.43166 (9)	0.34734 (19)	0.0141 (4)
C4	0.5544 (3)	0.48812 (10)	0.2810 (2)	0.0166 (5)
H4	0.5831	0.5299	0.3154	0.020*
C5	0.4619 (3)	0.48238 (10)	0.1635 (2)	0.0192 (5)
H5	0.4271	0.5207	0.1178	0.023*
C6	0.4195 (3)	0.42170 (11)	0.1120 (2)	0.0203 (5)
H6	0.3572	0.4191	0.0314	0.024*
C7	0.4668 (3)	0.36458 (11)	0.1764 (2)	0.0194 (5)
H7	0.4373	0.3229	0.1416	0.023*
C7A	0.5584 (3)	0.37070 (10)	0.2930 (2)	0.0158 (4)
C1	0.6064 (3)	0.25053 (10)	0.3443 (2)	0.0180 (5)
H1A	0.6825	0.2259	0.4083	0.022*
H1B	0.6519	0.2428	0.2645	0.022*
C11	0.4190 (3)	0.22472 (9)	0.33689 (19)	0.0156 (5)
C12	0.3066 (3)	0.24356 (10)	0.4221 (2)	0.0186 (5)

H12	0.3480	0.2730	0.4863	0.022*
C13	0.1344 (3)	0.21970 (10)	0.4140 (2)	0.0201 (5)
H13	0.0591	0.2326	0.4729	0.024*
C14	0.0728 (3)	0.17722 (10)	0.3201 (2)	0.0221 (5)
H14	−0.0453	0.1613	0.3138	0.027*
C15	0.1841 (3)	0.15792 (10)	0.2352 (2)	0.0223 (5)
H15	0.1419	0.1287	0.1708	0.027*
C16	0.3566 (3)	0.18102 (10)	0.2440 (2)	0.0194 (5)
H16	0.4326	0.1670	0.1864	0.023*
C31	0.7821 (3)	0.45231 (9)	0.5646 (2)	0.0156 (4)
H31	0.8289	0.4273	0.6343	0.019*
C32	0.8098 (3)	0.52374 (10)	0.57446 (19)	0.0163 (5)
O32	0.7480 (2)	0.56084 (7)	0.49270 (14)	0.0240 (4)
C321	0.9176 (3)	0.54977 (10)	0.68739 (19)	0.0155 (5)
C322	0.9382 (3)	0.61761 (10)	0.6968 (2)	0.0195 (5)
H322	0.8854	0.6447	0.6319	0.023*
C323	1.0337 (3)	0.64590 (10)	0.7986 (2)	0.0215 (5)
H323	1.0465	0.6919	0.8051	0.026*
C324	1.1100 (3)	0.60517 (11)	0.8905 (2)	0.0194 (5)
F324	1.20475 (18)	0.63155 (6)	0.99166 (12)	0.0271 (3)
C325	1.0949 (3)	0.53824 (10)	0.8854 (2)	0.0193 (5)
H325	1.1498	0.5116	0.9503	0.023*
C326	0.9974 (3)	0.51083 (10)	0.7831 (2)	0.0178 (5)
H326	0.9846	0.4647	0.7780	0.021*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0179 (10)	0.0131 (8)	0.0150 (9)	−0.0015 (7)	−0.0011 (8)	0.0004 (7)
C2	0.0139 (11)	0.0179 (10)	0.0175 (11)	−0.0014 (9)	0.0025 (9)	0.0011 (9)
O2	0.0285 (9)	0.0181 (7)	0.0200 (8)	−0.0015 (7)	−0.0061 (7)	0.0042 (7)
C3	0.0123 (10)	0.0163 (9)	0.0166 (11)	0.0006 (8)	0.0044 (8)	0.0029 (8)
C3A	0.0121 (11)	0.0176 (10)	0.0133 (10)	0.0005 (8)	0.0041 (9)	0.0008 (8)
C4	0.0138 (11)	0.0179 (10)	0.0186 (11)	−0.0009 (9)	0.0036 (9)	0.0012 (9)
C5	0.0169 (11)	0.0220 (10)	0.0190 (11)	0.0019 (9)	0.0029 (9)	0.0055 (9)
C6	0.0150 (11)	0.0296 (11)	0.0158 (11)	−0.0023 (9)	0.0001 (9)	0.0023 (9)
C7	0.0172 (11)	0.0226 (10)	0.0178 (11)	−0.0013 (9)	0.0004 (9)	−0.0012 (9)
C7A	0.0130 (11)	0.0181 (10)	0.0169 (11)	0.0014 (9)	0.0049 (9)	0.0038 (8)
C1	0.0191 (12)	0.0140 (9)	0.0201 (11)	0.0027 (9)	−0.0001 (10)	−0.0007 (9)
C11	0.0177 (12)	0.0122 (9)	0.0161 (11)	0.0030 (8)	−0.0010 (9)	0.0047 (8)
C12	0.0263 (13)	0.0147 (9)	0.0142 (11)	−0.0005 (9)	−0.0003 (9)	0.0013 (8)
C13	0.0241 (13)	0.0185 (10)	0.0190 (11)	0.0033 (9)	0.0079 (10)	0.0022 (9)
C14	0.0197 (12)	0.0201 (10)	0.0269 (13)	−0.0046 (9)	0.0039 (10)	0.0016 (9)
C15	0.0265 (13)	0.0191 (10)	0.0212 (12)	−0.0058 (10)	0.0027 (10)	−0.0051 (9)
C16	0.0245 (13)	0.0154 (10)	0.0192 (11)	−0.0005 (9)	0.0064 (10)	−0.0004 (9)
C31	0.0153 (11)	0.0167 (10)	0.0146 (10)	−0.0002 (9)	0.0012 (9)	0.0019 (8)
C32	0.0152 (11)	0.0176 (10)	0.0171 (11)	−0.0011 (8)	0.0057 (9)	0.0013 (9)
O32	0.0315 (10)	0.0187 (7)	0.0203 (8)	−0.0026 (7)	−0.0029 (7)	0.0038 (7)

C321	0.0141 (11)	0.0175 (10)	0.0160 (11)	0.0009 (8)	0.0064 (9)	0.0001 (8)
C322	0.0214 (12)	0.0160 (10)	0.0220 (12)	0.0000 (9)	0.0060 (10)	0.0007 (9)
C323	0.0238 (13)	0.0153 (10)	0.0271 (13)	−0.0025 (9)	0.0098 (10)	−0.0039 (9)
C324	0.0172 (11)	0.0251 (11)	0.0168 (11)	−0.0056 (9)	0.0053 (9)	−0.0088 (9)
F324	0.0286 (8)	0.0300 (7)	0.0225 (7)	−0.0063 (6)	0.0020 (6)	−0.0116 (6)
C325	0.0196 (12)	0.0213 (10)	0.0166 (11)	0.0004 (9)	0.0012 (9)	−0.0002 (9)
C326	0.0170 (11)	0.0162 (9)	0.0204 (11)	−0.0002 (9)	0.0027 (9)	−0.0020 (9)

Geometric parameters (Å, º)

N1—C2	1.363 (3)	C12—H12	0.9500
N1—C7A	1.404 (3)	C13—C14	1.383 (3)
N1—C1	1.458 (3)	C13—H13	0.9500
C2—O2	1.222 (3)	C14—C15	1.387 (3)
C2—C3	1.521 (3)	C14—H14	0.9500
C3—C31	1.346 (3)	C15—C16	1.386 (3)
C3—C3A	1.461 (3)	C15—H15	0.9500
C3A—C4	1.394 (3)	C16—H16	0.9500
C3A—C7A	1.409 (3)	C31—C32	1.481 (3)
C4—C5	1.392 (3)	C31—H31	0.9500
C4—H4	0.9500	C32—O32	1.223 (3)
C5—C6	1.386 (3)	C32—C321	1.495 (3)
C5—H5	0.9500	C321—C326	1.395 (3)
C6—C7	1.391 (3)	C321—C322	1.401 (3)
C6—H6	0.9500	C322—C323	1.380 (3)
C7—C7A	1.381 (3)	C322—H322	0.9500
C7—H7	0.9500	C323—C324	1.379 (3)
C1—C11	1.513 (3)	C323—H323	0.9500
C1—H1A	0.9900	C324—F324	1.356 (2)
C1—H1B	0.9900	C324—C325	1.377 (3)
C11—C12	1.393 (3)	C325—C326	1.383 (3)
C11—C16	1.393 (3)	C325—H325	0.9500
C12—C13	1.390 (3)	C326—H326	0.9500
C2—N1—C7A	110.63 (17)	C11—C12—H12	119.7
C2—N1—C1	124.06 (17)	C14—C13—C12	120.0 (2)
C7A—N1—C1	125.26 (17)	C14—C13—H13	120.0
O2—C2—N1	126.03 (19)	C12—C13—H13	120.0
O2—C2—C3	127.10 (19)	C13—C14—C15	119.8 (2)
N1—C2—C3	106.86 (17)	C13—C14—H14	120.1
C31—C3—C3A	138.04 (19)	C15—C14—H14	120.1
C31—C3—C2	116.53 (18)	C16—C15—C14	120.3 (2)
C3A—C3—C2	105.40 (17)	C16—C15—H15	119.8
C4—C3A—C7A	118.59 (19)	C14—C15—H15	119.8
C4—C3A—C3	134.61 (19)	C15—C16—C11	120.4 (2)
C7A—C3A—C3	106.79 (17)	C15—C16—H16	119.8
C5—C4—C3A	119.03 (19)	C11—C16—H16	119.8
C5—C4—H4	120.5	C3—C31—C32	128.0 (2)

C3A—C4—H4	120.5	C3—C31—H31	116.0
C6—C5—C4	121.1 (2)	C32—C31—H31	116.0
C6—C5—H5	119.5	O32—C32—C31	121.5 (2)
C4—C5—H5	119.5	O32—C32—C321	120.36 (18)
C5—C6—C7	121.1 (2)	C31—C32—C321	118.11 (18)
C5—C6—H6	119.5	C326—C321—C322	118.5 (2)
C7—C6—H6	119.5	C326—C321—C32	124.09 (18)
C7A—C7—C6	117.5 (2)	C322—C321—C32	117.38 (19)
C7A—C7—H7	121.3	C323—C322—C321	121.3 (2)
C6—C7—H7	121.3	C323—C322—H322	119.4
C7—C7A—N1	127.00 (19)	C321—C322—H322	119.4
C7—C7A—C3A	122.74 (19)	C324—C323—C322	117.8 (2)
N1—C7A—C3A	110.26 (18)	C324—C323—H323	121.1
N1—C1—C11	113.18 (17)	C322—C323—H323	121.1
N1—C1—H1A	108.9	F324—C324—C325	117.6 (2)
C11—C1—H1A	108.9	F324—C324—C323	119.19 (19)
N1—C1—H1B	108.9	C325—C324—C323	123.2 (2)
C11—C1—H1B	108.9	C324—C325—C326	118.1 (2)
H1A—C1—H1B	107.8	C324—C325—H325	120.9
C12—C11—C16	118.9 (2)	C326—C325—H325	120.9
C12—C11—C1	121.19 (19)	C325—C326—C321	121.0 (2)
C16—C11—C1	119.9 (2)	C325—C326—H326	119.5
C13—C12—C11	120.6 (2)	C321—C326—H326	119.5
C13—C12—H12	119.7		

C7A—N1—C2—O2	178.1 (2)	N1—C1—C11—C12	−41.8 (3)
C1—N1—C2—O2	−4.2 (3)	N1—C1—C11—C16	138.60 (19)
C7A—N1—C2—C3	−2.5 (2)	C16—C11—C12—C13	−0.6 (3)
C1—N1—C2—C3	175.26 (18)	C1—C11—C12—C13	179.74 (19)
O2—C2—C3—C31	3.1 (3)	C11—C12—C13—C14	−0.5 (3)
N1—C2—C3—C31	−176.39 (19)	C12—C13—C14—C15	0.8 (3)
O2—C2—C3—C3A	−178.4 (2)	C13—C14—C15—C16	0.0 (3)
N1—C2—C3—C3A	2.1 (2)	C14—C15—C16—C11	−1.1 (3)
C31—C3—C3A—C4	−3.4 (5)	C12—C11—C16—C15	1.4 (3)
C2—C3—C3A—C4	178.6 (2)	C1—C11—C16—C15	−178.94 (19)
C31—C3—C3A—C7A	177.0 (3)	C3A—C3—C31—C32	−1.7 (4)
C2—C3—C3A—C7A	−0.9 (2)	C2—C3—C31—C32	176.1 (2)
C7A—C3A—C4—C5	−0.2 (3)	C3—C31—C32—O32	3.5 (4)
C3—C3A—C4—C5	−179.8 (2)	C3—C31—C32—C321	−176.0 (2)
C3A—C4—C5—C6	−0.2 (3)	O32—C32—C321—C326	−178.0 (2)
C4—C5—C6—C7	0.5 (3)	C31—C32—C321—C326	1.5 (3)
C5—C6—C7—C7A	−0.4 (3)	O32—C32—C321—C322	2.4 (3)
C6—C7—C7A—N1	−179.5 (2)	C31—C32—C321—C322	−178.2 (2)
C6—C7—C7A—C3A	0.0 (3)	C326—C321—C322—C323	−0.5 (3)
C2—N1—C7A—C7	−178.6 (2)	C32—C321—C322—C323	179.2 (2)
C1—N1—C7A—C7	3.7 (3)	C321—C322—C323—C324	0.6 (3)
C2—N1—C7A—C3A	1.9 (2)	C322—C323—C324—F324	179.96 (19)
C1—N1—C7A—C3A	−175.75 (19)	C322—C323—C324—C325	−0.1 (4)

C4—C3A—C7A—C7	0.4 (3)	F324—C324—C325—C326	179.52 (19)
C3—C3A—C7A—C7	180.0 (2)	C323—C324—C325—C326	−0.4 (4)
C4—C3A—C7A—N1	179.88 (19)	C324—C325—C326—C321	0.5 (3)
C3—C3A—C7A—N1	−0.5 (2)	C322—C321—C326—C325	0.0 (3)
C2—N1—C1—C11	111.4 (2)	C32—C321—C326—C325	−179.7 (2)
C7A—N1—C1—C11	−71.2 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15—H15···O32^{i}	0.95	2.49	3.278 (3)	141

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

(*E*)-1-Benzyl-3-[2-(4-methoxyphenyl)-2-oxoethylidene]indolin-2-one (IIc)

Crystal data

$C_{24}H_{19}NO_3$
M_r = 369.40
Monoclinic, $P2_1/n$
a = 4.9743 (2) Å
b = 29.1957 (13) Å
c = 12.4406 (6) Å
β = 100.914 (2)°
V = 1774.05 (14) Å^3
Z = 4
$F(000)$ = 776
D_x = 1.383 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4142 reflections
θ = 2.2–27.6°
μ = 0.09 mm^{-1}
T = 100 K
Needle, orange
0.45 × 0.06 × 0.04 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} = 0.948, T_{max} = 0.996
54500 measured reflections
4142 independent reflections
3643 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 27.6°, θ_{min} = 2.2°
h = −6→6
k = −38→38
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.095
S = 1.06
4142 reflections
254 parameters
0 restraints
Primary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0377P)^2 + 0.9412P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 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	−0.4211 (2)	0.41865 (3)	0.34758 (8)	0.0170 (2)
C2	−0.2448 (2)	0.41242 (4)	0.44575 (10)	0.0171 (2)
O2	−0.25042 (19)	0.43355 (3)	0.52982 (7)	0.0232 (2)
C3	−0.0448 (2)	0.37514 (4)	0.42781 (9)	0.0158 (2)
C3A	−0.1171 (2)	0.36335 (4)	0.31117 (9)	0.0159 (2)
C4	−0.0083 (2)	0.33398 (4)	0.24221 (10)	0.0187 (2)
H4	0.1466	0.3155	0.2702	0.022*
C5	−0.1295 (3)	0.33197 (4)	0.13163 (10)	0.0222 (3)
H5	−0.0546	0.3123	0.0841	0.027*
C6	−0.3579 (3)	0.35832 (4)	0.09007 (10)	0.0224 (3)
H6	−0.4396	0.3559	0.0148	0.027*
C7	−0.4691 (3)	0.38834 (4)	0.15729 (10)	0.0203 (2)
H7	−0.6244	0.4066	0.1290	0.024*
C7A	−0.3456 (2)	0.39055 (4)	0.26640 (10)	0.0165 (2)
C1	−0.6225 (2)	0.45543 (4)	0.32653 (10)	0.0187 (2)
H1A	−0.6531	0.4681	0.3971	0.022*
H1B	−0.7987	0.4429	0.2869	0.022*
C11	−0.5288 (2)	0.49338 (4)	0.25932 (10)	0.0168 (2)
C12	−0.2897 (2)	0.51747 (4)	0.30076 (10)	0.0181 (2)
H12	−0.1902	0.5106	0.3719	0.022*
C13	−0.1958 (3)	0.55135 (4)	0.23909 (10)	0.0195 (2)
H13	−0.0315	0.5674	0.2676	0.023*
C14	−0.3429 (3)	0.56185 (4)	0.13518 (10)	0.0208 (3)
H14	−0.2787	0.5850	0.0928	0.025*
C15	−0.5824 (3)	0.53855 (4)	0.09371 (10)	0.0216 (3)
H15	−0.6838	0.5460	0.0232	0.026*
C16	−0.6744 (2)	0.50419 (4)	0.15556 (10)	0.0195 (2)
H16	−0.8379	0.4880	0.1267	0.023*
C31	0.1436 (2)	0.36197 (4)	0.51499 (10)	0.0174 (2)
H31	0.1410	0.3775	0.5819	0.021*
C32	0.3545 (2)	0.32586 (4)	0.51787 (10)	0.0169 (2)
O32	0.35542 (19)	0.30061 (3)	0.43906 (7)	0.0241 (2)
C321	0.5657 (2)	0.32005 (4)	0.61873 (9)	0.0152 (2)
C322	0.7320 (2)	0.28116 (4)	0.62600 (9)	0.0159 (2)
H322	0.6977	0.2589	0.5692	0.019*
C323	0.9451 (2)	0.27408 (4)	0.71355 (9)	0.0162 (2)
H323	1.0562	0.2475	0.7167	0.019*
C324	0.9944 (2)	0.30663 (4)	0.79719 (9)	0.0156 (2)
C325	0.8272 (2)	0.34535 (4)	0.79242 (10)	0.0174 (2)

H325	0.8589	0.3672	0.8501	0.021*
C326	0.6158 (2)	0.35207 (4)	0.70410 (10)	0.0167 (2)
H326	0.5039	0.3786	0.7013	0.020*
O324	1.19899 (17)	0.30348 (3)	0.88633 (7)	0.01893 (19)
C327	1.3906 (2)	0.26658 (4)	0.88692 (10)	0.0185 (2)
H37A	1.2960	0.2373	0.8893	0.028*
H37B	1.5372	0.2693	0.9514	0.028*
H37C	1.4692	0.2680	0.8205	0.028*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0166 (5)	0.0173 (5)	0.0168 (5)	0.0025 (4)	0.0022 (4)	0.0022 (4)
C2	0.0164 (5)	0.0176 (5)	0.0177 (6)	0.0005 (4)	0.0040 (4)	0.0037 (4)
O2	0.0254 (5)	0.0267 (5)	0.0178 (4)	0.0073 (4)	0.0045 (4)	−0.0006 (4)
C3	0.0154 (5)	0.0151 (5)	0.0176 (6)	−0.0008 (4)	0.0052 (4)	0.0019 (4)
C3A	0.0151 (5)	0.0145 (5)	0.0179 (6)	−0.0025 (4)	0.0027 (4)	0.0022 (4)
C4	0.0197 (6)	0.0167 (5)	0.0196 (6)	0.0005 (4)	0.0029 (5)	0.0015 (4)
C5	0.0282 (7)	0.0181 (6)	0.0199 (6)	0.0014 (5)	0.0039 (5)	−0.0020 (5)
C6	0.0276 (7)	0.0200 (6)	0.0176 (6)	−0.0006 (5)	−0.0008 (5)	0.0000 (5)
C7	0.0207 (6)	0.0181 (6)	0.0201 (6)	0.0004 (5)	−0.0009 (5)	0.0018 (5)
C7A	0.0164 (5)	0.0138 (5)	0.0194 (6)	−0.0021 (4)	0.0038 (4)	0.0015 (4)
C1	0.0157 (5)	0.0192 (6)	0.0215 (6)	0.0037 (4)	0.0044 (5)	0.0038 (5)
C11	0.0174 (5)	0.0152 (5)	0.0186 (6)	0.0049 (4)	0.0052 (4)	0.0003 (4)
C12	0.0194 (6)	0.0171 (5)	0.0173 (6)	0.0043 (4)	0.0025 (4)	−0.0002 (4)
C13	0.0193 (6)	0.0165 (5)	0.0227 (6)	0.0015 (5)	0.0039 (5)	−0.0020 (5)
C14	0.0251 (6)	0.0177 (6)	0.0210 (6)	0.0022 (5)	0.0076 (5)	0.0018 (5)
C15	0.0251 (6)	0.0216 (6)	0.0171 (6)	0.0049 (5)	0.0017 (5)	0.0014 (5)
C16	0.0178 (6)	0.0191 (6)	0.0206 (6)	0.0021 (5)	0.0010 (5)	−0.0008 (5)
C31	0.0171 (5)	0.0180 (5)	0.0176 (6)	−0.0001 (4)	0.0042 (4)	0.0013 (4)
C32	0.0152 (5)	0.0179 (5)	0.0177 (6)	−0.0012 (4)	0.0030 (4)	0.0019 (4)
O32	0.0242 (5)	0.0252 (5)	0.0206 (4)	0.0051 (4)	−0.0018 (4)	−0.0047 (4)
C321	0.0136 (5)	0.0169 (5)	0.0156 (5)	−0.0011 (4)	0.0041 (4)	0.0019 (4)
C322	0.0166 (5)	0.0164 (5)	0.0154 (5)	−0.0012 (4)	0.0049 (4)	−0.0002 (4)
C323	0.0160 (5)	0.0157 (5)	0.0176 (5)	0.0020 (4)	0.0051 (4)	0.0010 (4)
C324	0.0133 (5)	0.0185 (5)	0.0152 (5)	−0.0013 (4)	0.0032 (4)	0.0013 (4)
C325	0.0185 (6)	0.0164 (5)	0.0177 (6)	−0.0013 (4)	0.0042 (4)	−0.0029 (4)
C326	0.0158 (5)	0.0153 (5)	0.0196 (6)	0.0013 (4)	0.0048 (4)	0.0004 (4)
O324	0.0166 (4)	0.0210 (4)	0.0175 (4)	0.0024 (3)	−0.0011 (3)	−0.0023 (3)
C327	0.0157 (5)	0.0200 (6)	0.0194 (6)	0.0022 (4)	0.0026 (4)	0.0018 (4)

Geometric parameters (Å, º)

N1—C2	1.3741 (15)	C13—H13	0.9500
N1—C7A	1.4063 (15)	C14—C15	1.3842 (18)
N1—C1	1.4583 (15)	C14—H14	0.9500
C2—O2	1.2191 (15)	C15—C16	1.3932 (17)
C2—C3	1.5193 (16)	C15—H15	0.9500

C3—C31	1.3482 (17)	C16—H16	0.9500
C3—C3A	1.4682 (16)	C31—C32	1.4827 (16)
C3A—C4	1.3930 (16)	C31—H31	0.9500
C3A—C7A	1.4115 (16)	C32—O32	1.2275 (15)
C4—C5	1.3947 (17)	C32—C321	1.4859 (16)
C4—H4	0.9500	C321—C322	1.3975 (16)
C5—C6	1.3883 (18)	C321—C326	1.4015 (16)
C5—H5	0.9500	C322—C323	1.3841 (16)
C6—C7	1.3959 (18)	C322—H322	0.9500
C6—H6	0.9500	C323—C324	1.3962 (16)
C7—C7A	1.3815 (17)	C323—H323	0.9500
C7—H7	0.9500	C324—O324	1.3588 (14)
C1—C11	1.5137 (16)	C324—C325	1.3982 (16)
C1—H1A	0.9900	C325—C326	1.3834 (17)
C1—H1B	0.9900	C325—H325	0.9500
C11—C16	1.3917 (17)	C326—H326	0.9500
C11—C12	1.3939 (17)	O324—C327	1.4377 (14)
C12—C13	1.3859 (17)	C327—H37A	0.9800
C12—H12	0.9500	C327—H37B	0.9800
C13—C14	1.3937 (18)	C327—H37C	0.9800

C2—N1—C7A	110.41 (10)	C14—C13—H13	120.1
C2—N1—C1	123.94 (10)	C15—C14—C13	120.09 (12)
C7A—N1—C1	124.65 (10)	C15—C14—H14	120.0
O2—C2—N1	125.52 (11)	C13—C14—H14	120.0
O2—C2—C3	127.64 (11)	C14—C15—C16	119.84 (12)
N1—C2—C3	106.84 (10)	C14—C15—H15	120.1
C31—C3—C3A	136.99 (11)	C16—C15—H15	120.1
C31—C3—C2	117.44 (11)	C11—C16—C15	120.52 (12)
C3A—C3—C2	105.56 (10)	C11—C16—H16	119.7
C4—C3A—C7A	118.76 (11)	C15—C16—H16	119.7
C4—C3A—C3	134.57 (11)	C3—C31—C32	127.09 (11)
C7A—C3A—C3	106.65 (10)	C3—C31—H31	116.5
C3A—C4—C5	119.17 (11)	C32—C31—H31	116.5
C3A—C4—H4	120.4	O32—C32—C31	121.15 (11)
C5—C4—H4	120.4	O32—C32—C321	119.79 (11)
C6—C5—C4	120.96 (12)	C31—C32—C321	119.05 (10)
C6—C5—H5	119.5	C322—C321—C326	118.35 (11)
C4—C5—H5	119.5	C322—C321—C32	117.56 (10)
C5—C6—C7	120.93 (12)	C326—C321—C32	124.02 (10)
C5—C6—H6	119.5	C323—C322—C321	121.87 (11)
C7—C6—H6	119.5	C323—C322—H322	119.1
C7A—C7—C6	117.71 (11)	C321—C322—H322	119.1
C7A—C7—H7	121.1	C322—C323—C324	119.02 (11)
C6—C7—H7	121.1	C322—C323—H323	120.5
C7—C7A—N1	127.11 (11)	C324—C323—H323	120.5
C7—C7A—C3A	122.44 (11)	O324—C324—C323	124.13 (10)
N1—C7A—C3A	110.45 (10)	O324—C324—C325	115.90 (10)

N1—C1—C11	111.41 (9)	C323—C324—C325	119.97 (11)
N1—C1—H1A	109.3	C326—C325—C324	120.33 (11)
C11—C1—H1A	109.3	C326—C325—H325	119.8
N1—C1—H1B	109.3	C324—C325—H325	119.8
C11—C1—H1B	109.3	C325—C326—C321	120.44 (11)
H1A—C1—H1B	108.0	C325—C326—H326	119.8
C16—C11—C12	119.11 (11)	C321—C326—H326	119.8
C16—C11—C1	121.17 (11)	C324—O324—C327	116.62 (9)
C12—C11—C1	119.71 (11)	O324—C327—H37A	109.5
C13—C12—C11	120.59 (11)	O324—C327—H37B	109.5
C13—C12—H12	119.7	H37A—C327—H37B	109.5
C11—C12—H12	119.7	O324—C327—H37C	109.5
C12—C13—C14	119.85 (12)	H37A—C327—H37C	109.5
C12—C13—H13	120.1	H37B—C327—H37C	109.5

C7A—N1—C2—O2	176.39 (11)	N1—C1—C11—C12	−61.57 (14)
C1—N1—C2—O2	7.38 (19)	C16—C11—C12—C13	−0.83 (17)
C7A—N1—C2—C3	−2.95 (12)	C1—C11—C12—C13	177.95 (11)
C1—N1—C2—C3	−171.96 (10)	C11—C12—C13—C14	0.71 (18)
O2—C2—C3—C31	2.97 (18)	C12—C13—C14—C15	0.09 (18)
N1—C2—C3—C31	−177.72 (10)	C13—C14—C15—C16	−0.77 (18)
O2—C2—C3—C3A	−176.46 (12)	C12—C11—C16—C15	0.15 (17)
N1—C2—C3—C3A	2.86 (12)	C1—C11—C16—C15	−178.62 (11)
C31—C3—C3A—C4	−2.9 (2)	C14—C15—C16—C11	0.64 (18)
C2—C3—C3A—C4	176.40 (13)	C3A—C3—C31—C32	−2.2 (2)
C31—C3—C3A—C7A	179.04 (14)	C2—C3—C31—C32	178.62 (11)
C2—C3—C3A—C7A	−1.71 (12)	C3—C31—C32—O32	−7.94 (19)
C7A—C3A—C4—C5	−0.66 (17)	C3—C31—C32—C321	172.77 (11)
C3—C3A—C4—C5	−178.59 (12)	O32—C32—C321—C322	−9.91 (16)
C3A—C4—C5—C6	−0.80 (19)	C31—C32—C321—C322	169.40 (10)
C4—C5—C6—C7	1.4 (2)	O32—C32—C321—C326	167.07 (11)
C5—C6—C7—C7A	−0.56 (19)	C31—C32—C321—C326	−13.62 (17)
C6—C7—C7A—N1	179.08 (11)	C326—C321—C322—C323	−1.14 (17)
C6—C7—C7A—C3A	−0.95 (18)	C32—C321—C322—C323	176.02 (10)
C2—N1—C7A—C7	−178.08 (11)	C321—C322—C323—C324	0.31 (17)
C1—N1—C7A—C7	−9.17 (19)	C322—C323—C324—O324	−179.36 (10)
C2—N1—C7A—C3A	1.94 (13)	C322—C323—C324—C325	0.89 (17)
C1—N1—C7A—C3A	170.86 (10)	O324—C324—C325—C326	178.99 (10)
C4—C3A—C7A—C7	1.56 (17)	C323—C324—C325—C326	−1.24 (17)
C3—C3A—C7A—C7	−179.98 (11)	C324—C325—C326—C321	0.39 (18)
C4—C3A—C7A—N1	−178.46 (10)	C322—C321—C326—C325	0.78 (17)
C3—C3A—C7A—N1	0.00 (13)	C32—C321—C326—C325	−176.18 (11)
C2—N1—C1—C11	102.92 (13)	C323—C324—O324—C327	6.74 (16)
C7A—N1—C1—C11	−64.54 (15)	C325—C324—O324—C327	−173.51 (10)
N1—C1—C11—C16	117.19 (12)		

(*E*)-1-Benzyl-3-[2-oxo-2-(pyridin-4-yl)ethylidene]indolin-2-one (IIe)

Crystal data

$C_{22}H_{16}N_2O_2$
$M_r = 340.37$
Monoclinic, $P2_1/n$
$a = 7.3457$ (6) Å
$b = 18.0675$ (16) Å
$c = 13.1813$ (13) Å
$\beta = 105.994$ (3)°
$V = 1681.7$ (3) Å^3
$Z = 4$
$F(000) = 712$
$D_x = 1.344$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4167 reflections
$\theta = 2.8$–$28.3°$
$\mu = 0.09$ mm^{-1}
$T = 100$ K
Block, red
0.16 × 0.15 × 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)
$T_{min} = 0.878$, $T_{max} = 0.990$
4167 measured reflections
4167 independent reflections
3281 reflections with $I > 2\sigma(I)$
$\theta_{max} = 28.4°$, $\theta_{min} = 2.8°$
$h = -9 \rightarrow 9$
$k = -24 \rightarrow 24$
$l = -7 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.121$
$S = 1.09$
4167 reflections
237 parameters
0 restraints
Primary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0398P)^2 + 1.0992P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015b), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0093 (13)

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.

Refinement. Refined as a 2-component twin.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.43527 (19)	0.59990 (8)	0.24839 (10)	0.0195 (3)
C2	0.5093 (2)	0.62189 (9)	0.35066 (13)	0.0192 (3)
O2	0.49338 (17)	0.68369 (7)	0.38499 (9)	0.0233 (3)
C3	0.6154 (2)	0.55610 (9)	0.41041 (13)	0.0182 (3)
C3A	0.5946 (2)	0.49693 (9)	0.33226 (12)	0.0190 (3)
C4	0.6559 (2)	0.42378 (9)	0.33563 (14)	0.0221 (3)

H4	0.7272	0.4023	0.4002	0.027*
C5	0.6107 (3)	0.38262 (10)	0.24260 (14)	0.0249 (4)
H5	0.6504	0.3325	0.2443	0.030*
C6	0.5089 (3)	0.41357 (10)	0.14767 (14)	0.0250 (4)
H6	0.4813	0.3844	0.0853	0.030*
C7	0.4460 (2)	0.48691 (10)	0.14209 (13)	0.0228 (3)
H7	0.3774	0.5085	0.0770	0.027*
C7A	0.4877 (2)	0.52654 (9)	0.23489 (13)	0.0194 (3)
C1	0.3330 (2)	0.64906 (9)	0.16509 (13)	0.0207 (3)
H1A	0.2665	0.6868	0.1960	0.025*
H1B	0.2359	0.6201	0.1134	0.025*
C11	0.4581 (2)	0.68798 (9)	0.10759 (13)	0.0197 (3)
C12	0.6527 (2)	0.67827 (10)	0.13385 (14)	0.0259 (4)
H12	0.7129	0.6462	0.1903	0.031*
C13	0.7603 (3)	0.71524 (11)	0.07792 (16)	0.0320 (4)
H13	0.8934	0.7079	0.0960	0.038*
C14	0.6747 (3)	0.76263 (11)	−0.00386 (16)	0.0328 (4)
H14	0.7485	0.7879	−0.0418	0.039*
C15	0.4800 (3)	0.77283 (10)	−0.02986 (15)	0.0312 (4)
H15	0.4206	0.8056	−0.0854	0.037*
C16	0.3717 (3)	0.73544 (10)	0.02474 (14)	0.0246 (4)
H16	0.2383	0.7421	0.0057	0.029*
C31	0.7009 (2)	0.56643 (9)	0.51380 (13)	0.0203 (3)
H31	0.6854	0.6139	0.5411	0.024*
C32	0.8155 (2)	0.51324 (9)	0.58972 (13)	0.0215 (3)
O32	0.8429 (2)	0.44965 (7)	0.56511 (10)	0.0337 (3)
N321	1.0570 (2)	0.57448 (9)	0.91573 (12)	0.0274 (3)
C322	1.0660 (2)	0.50551 (10)	0.87931 (13)	0.0253 (4)
H322	1.1279	0.4687	0.9281	0.030*
C323	0.9904 (2)	0.48488 (10)	0.77511 (13)	0.0217 (3)
H323	1.0010	0.4353	0.7536	0.026*
C324	0.8983 (2)	0.53816 (9)	0.70199 (13)	0.0195 (3)
C325	0.8883 (2)	0.60966 (10)	0.73844 (13)	0.0235 (4)
H325	0.8268	0.6476	0.6915	0.028*
C326	0.9697 (3)	0.62504 (10)	0.84461 (14)	0.0269 (4)
H326	0.9630	0.6744	0.8681	0.032*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0216 (7)	0.0181 (7)	0.0174 (7)	0.0010 (5)	0.0031 (5)	0.0009 (5)
C2	0.0192 (7)	0.0193 (8)	0.0195 (8)	0.0001 (6)	0.0060 (6)	0.0007 (6)
O2	0.0261 (6)	0.0179 (6)	0.0250 (6)	0.0019 (5)	0.0055 (5)	−0.0014 (5)
C3	0.0183 (7)	0.0173 (7)	0.0192 (8)	−0.0007 (6)	0.0056 (6)	0.0006 (6)
C3A	0.0199 (7)	0.0197 (8)	0.0177 (7)	−0.0013 (6)	0.0056 (6)	−0.0003 (6)
C4	0.0236 (8)	0.0186 (8)	0.0242 (8)	0.0004 (6)	0.0066 (6)	0.0008 (6)
C5	0.0291 (9)	0.0197 (8)	0.0262 (9)	0.0005 (7)	0.0083 (7)	−0.0031 (7)
C6	0.0284 (9)	0.0247 (9)	0.0225 (8)	−0.0031 (7)	0.0077 (7)	−0.0056 (7)

C7	0.0231 (8)	0.0261 (9)	0.0185 (8)	−0.0023 (7)	0.0045 (6)	−0.0001 (7)
C7A	0.0197 (7)	0.0182 (8)	0.0206 (8)	−0.0009 (6)	0.0064 (6)	0.0008 (6)
C1	0.0203 (7)	0.0213 (8)	0.0194 (8)	0.0012 (6)	0.0035 (6)	0.0031 (6)
C11	0.0237 (8)	0.0164 (7)	0.0189 (8)	−0.0004 (6)	0.0054 (6)	−0.0021 (6)
C12	0.0229 (8)	0.0277 (9)	0.0260 (9)	−0.0005 (7)	0.0047 (7)	0.0018 (7)
C13	0.0258 (9)	0.0332 (10)	0.0390 (11)	−0.0049 (8)	0.0124 (8)	−0.0039 (8)
C14	0.0452 (11)	0.0231 (9)	0.0380 (11)	−0.0080 (8)	0.0245 (9)	−0.0038 (8)
C15	0.0467 (11)	0.0208 (9)	0.0293 (10)	0.0028 (8)	0.0157 (8)	0.0034 (7)
C16	0.0298 (9)	0.0203 (8)	0.0239 (8)	0.0035 (7)	0.0079 (7)	0.0011 (7)
C31	0.0229 (8)	0.0179 (8)	0.0194 (8)	−0.0012 (6)	0.0047 (6)	−0.0003 (6)
C32	0.0240 (8)	0.0204 (8)	0.0194 (8)	−0.0007 (7)	0.0047 (6)	0.0001 (6)
O32	0.0487 (8)	0.0221 (7)	0.0240 (7)	0.0089 (6)	−0.0007 (6)	−0.0027 (5)
N321	0.0285 (8)	0.0308 (8)	0.0212 (7)	−0.0043 (6)	0.0040 (6)	−0.0009 (6)
C322	0.0230 (8)	0.0288 (9)	0.0215 (8)	−0.0025 (7)	0.0018 (6)	0.0035 (7)
C323	0.0196 (7)	0.0218 (8)	0.0224 (8)	−0.0009 (6)	0.0039 (6)	0.0013 (6)
C324	0.0177 (7)	0.0217 (8)	0.0187 (8)	−0.0029 (6)	0.0041 (6)	0.0008 (6)
C325	0.0256 (8)	0.0210 (8)	0.0224 (8)	−0.0005 (7)	0.0040 (7)	0.0015 (6)
C326	0.0309 (9)	0.0253 (9)	0.0231 (8)	−0.0033 (7)	0.0051 (7)	−0.0035 (7)

Geometric parameters (Å, º)

N1—C2	1.366 (2)	C12—H12	0.9500
N1—C7A	1.405 (2)	C13—C14	1.385 (3)
N1—C1	1.451 (2)	C13—H13	0.9500
C2—O2	1.222 (2)	C14—C15	1.388 (3)
C2—C3	1.517 (2)	C14—H14	0.9500
C3—C31	1.347 (2)	C15—C16	1.387 (3)
C3—C3A	1.463 (2)	C15—H15	0.9500
C3A—C4	1.393 (2)	C16—H16	0.9500
C3A—C7A	1.412 (2)	C31—C32	1.473 (2)
C4—C5	1.394 (2)	C31—H31	0.9500
C4—H4	0.9500	C32—O32	1.225 (2)
C5—C6	1.386 (3)	C32—C324	1.506 (2)
C5—H5	0.9500	N321—C326	1.339 (2)
C6—C7	1.399 (3)	N321—C322	1.344 (2)
C6—H6	0.9500	C322—C323	1.383 (2)
C7—C7A	1.377 (2)	C322—H322	0.9500
C7—H7	0.9500	C323—C324	1.398 (2)
C1—C11	1.515 (2)	C323—H323	0.9500
C1—H1A	0.9900	C324—C325	1.387 (2)
C1—H1B	0.9900	C325—C326	1.390 (2)
C11—C12	1.386 (2)	C325—H325	0.9500
C11—C16	1.396 (2)	C326—H326	0.9500
C12—C13	1.391 (3)		
C2—N1—C7A	110.54 (13)	C11—C12—H12	119.9
C2—N1—C1	123.43 (14)	C13—C12—H12	119.9
C7A—N1—C1	125.77 (14)	C14—C13—C12	120.48 (18)

O2—C2—N1	125.32 (15)	C14—C13—H13	119.8
O2—C2—C3	127.69 (15)	C12—C13—H13	119.8
N1—C2—C3	106.98 (13)	C13—C14—C15	119.36 (18)
C31—C3—C3A	137.95 (15)	C13—C14—H14	120.3
C31—C3—C2	116.49 (14)	C15—C14—H14	120.3
C3A—C3—C2	105.55 (13)	C16—C15—C14	120.45 (18)
C4—C3A—C7A	118.83 (15)	C16—C15—H15	119.8
C4—C3A—C3	134.48 (15)	C14—C15—H15	119.8
C7A—C3A—C3	106.68 (14)	C15—C16—C11	120.18 (17)
C3A—C4—C5	118.79 (16)	C15—C16—H16	119.9
C3A—C4—H4	120.6	C11—C16—H16	119.9
C5—C4—H4	120.6	C3—C31—C32	128.08 (16)
C6—C5—C4	121.15 (17)	C3—C31—H31	116.0
C6—C5—H5	119.4	C32—C31—H31	116.0
C4—C5—H5	119.4	O32—C32—C31	122.46 (15)
C5—C6—C7	121.19 (16)	O32—C32—C324	119.30 (15)
C5—C6—H6	119.4	C31—C32—C324	118.23 (14)
C7—C6—H6	119.4	C326—N321—C322	116.34 (15)
C7A—C7—C6	117.15 (16)	N321—C322—C323	123.99 (16)
C7A—C7—H7	121.4	N321—C322—H322	118.0
C6—C7—H7	121.4	C323—C322—H322	118.0
C7—C7A—N1	126.96 (15)	C322—C323—C324	118.99 (16)
C7—C7A—C3A	122.84 (16)	C322—C323—H323	120.5
N1—C7A—C3A	110.20 (14)	C324—C323—H323	120.5
N1—C1—C11	113.83 (14)	C325—C324—C323	117.66 (15)
N1—C1—H1A	108.8	C325—C324—C32	124.99 (15)
C11—C1—H1A	108.8	C323—C324—C32	117.35 (15)
N1—C1—H1B	108.8	C324—C325—C326	119.06 (16)
C11—C1—H1B	108.8	C324—C325—H325	120.5
H1A—C1—H1B	107.7	C326—C325—H325	120.5
C12—C11—C16	119.24 (16)	N321—C326—C325	123.96 (17)
C12—C11—C1	122.82 (15)	N321—C326—H326	118.0
C16—C11—C1	117.94 (15)	C325—C326—H326	118.0
C11—C12—C13	120.28 (17)		

C7A—N1—C2—O2	177.18 (16)	C7A—N1—C1—C11	−83.28 (19)
C1—N1—C2—O2	2.7 (3)	N1—C1—C11—C12	−1.1 (2)
C7A—N1—C2—C3	−1.75 (17)	N1—C1—C11—C16	178.84 (15)
C1—N1—C2—C3	−176.23 (14)	C16—C11—C12—C13	−0.2 (3)
O2—C2—C3—C31	0.7 (3)	C1—C11—C12—C13	179.72 (17)
N1—C2—C3—C31	179.61 (15)	C11—C12—C13—C14	0.6 (3)
O2—C2—C3—C3A	−178.37 (16)	C12—C13—C14—C15	−0.2 (3)
N1—C2—C3—C3A	0.53 (17)	C13—C14—C15—C16	−0.6 (3)
C31—C3—C3A—C4	1.7 (3)	C14—C15—C16—C11	1.0 (3)
C2—C3—C3A—C4	−179.57 (18)	C12—C11—C16—C15	−0.6 (3)
C31—C3—C3A—C7A	−177.92 (19)	C1—C11—C16—C15	179.49 (16)
C2—C3—C3A—C7A	0.85 (17)	C3A—C3—C31—C32	0.0 (3)
C7A—C3A—C4—C5	0.5 (2)	C2—C3—C31—C32	−178.71 (16)

C3—C3A—C4—C5	−179.06 (18)	C3—C31—C32—O32	−1.6 (3)
C3A—C4—C5—C6	0.9 (3)	C3—C31—C32—C324	179.55 (16)
C4—C5—C6—C7	−0.7 (3)	C326—N321—C322—C323	−0.4 (3)
C5—C6—C7—C7A	−0.7 (3)	N321—C322—C323—C324	−0.1 (3)
C6—C7—C7A—N1	−178.39 (16)	C322—C323—C324—C325	0.3 (2)
C6—C7—C7A—C3A	2.1 (2)	C322—C323—C324—C32	−179.12 (15)
C2—N1—C7A—C7	−177.17 (16)	O32—C32—C324—C325	175.58 (17)
C1—N1—C7A—C7	−2.8 (3)	C31—C32—C324—C325	−5.6 (2)
C2—N1—C7A—C3A	2.39 (19)	O32—C32—C324—C323	−5.1 (2)
C1—N1—C7A—C3A	176.71 (15)	C31—C32—C324—C323	173.76 (15)
C4—C3A—C7A—C7	−2.0 (2)	C323—C324—C325—C326	0.1 (2)
C3—C3A—C7A—C7	177.63 (15)	C32—C324—C325—C326	179.46 (16)
C4—C3A—C7A—N1	178.40 (14)	C322—N321—C326—C325	0.9 (3)
C3—C3A—C7A—N1	−1.94 (18)	C324—C325—C326—N321	−0.7 (3)
C2—N1—C1—C11	90.35 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14···O2^{i}	0.95	2.32	3.234 (3)	161
C16—H16···O2^{ii}	0.95	2.45	3.230 (2)	140
C326—H326···O2^{iii}	0.95	2.58	3.494 (2)	162
C6—H6···*Cg*2^{iv}	0.95	2.64	3.566 (2)	165

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

(*E*)-1-Benzyl-3-[2-oxo-2-(thiophen-2-yl)ethylidene]indolin-2-one (IIg)

Crystal data

$C_{21}H_{15}NO_2S$
M_r = 345.40
Orthorhombic, *Pbca*
a = 17.5058 (14) Å
b = 8.8163 (6) Å
c = 21.2092 (16) Å
V = 3273.4 (4) Å^3
Z = 8
F(000) = 1440
D_x = 1.402 Mg m^{-3}
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 5334 reflections
θ = 2.3–31.8°
μ = 0.21 mm^{-1}
T = 100 K
Block, red
0.15 × 0.07 × 0.05 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} = 0.942, T_{max} = 0.989
32005 measured reflections
4154 independent reflections
3280 reflections with $I > 2\sigma(I)$
R_{int} = 0.065
θ_{max} = 28.6°, θ_{min} = 2.3°
h = −23→23
k = −11→10
l = −28→28

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.092$
$S = 1.03$
4154 reflections
239 parameters
10 restraints

Primary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0319P)^2 + 1.8245P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
N1	0.37636 (7)	0.12043 (14)	0.26577 (6)	0.0185 (3)	
C2	0.38846 (8)	0.21509 (16)	0.21539 (7)	0.0188 (3)	
O2	0.45021 (6)	0.24289 (13)	0.19116 (5)	0.0250 (2)	
C3	0.31120 (8)	0.27834 (16)	0.19688 (7)	0.0172 (3)	
C3A	0.25610 (8)	0.20780 (15)	0.23972 (6)	0.0172 (3)	
C4	0.17701 (8)	0.21528 (16)	0.24639 (7)	0.0189 (3)	
H4	0.1473	0.2758	0.2186	0.023*	
C5	0.14182 (9)	0.13236 (17)	0.29472 (7)	0.0214 (3)	
H5	0.0879	0.1368	0.2997	0.026*	
C6	0.18516 (9)	0.04372 (17)	0.33542 (7)	0.0222 (3)	
H6	0.1603	−0.0114	0.3680	0.027*	
C7	0.26444 (9)	0.03387 (16)	0.32955 (7)	0.0205 (3)	
H7	0.2939	−0.0267	0.3575	0.025*	
C7A	0.29843 (8)	0.11577 (16)	0.28144 (7)	0.0173 (3)	
C1	0.43804 (8)	0.04754 (16)	0.30084 (7)	0.0197 (3)	
H1A	0.4832	0.0364	0.2731	0.024*	
H1B	0.4216	−0.0551	0.3140	0.024*	
C11	0.45984 (8)	0.13876 (16)	0.35853 (7)	0.0185 (3)	
C12	0.49197 (9)	0.28327 (17)	0.35161 (7)	0.0218 (3)	
H12	0.5010	0.3226	0.3106	0.026*	
C13	0.51075 (9)	0.36954 (17)	0.40400 (8)	0.0244 (3)	
H13	0.5325	0.4675	0.3988	0.029*	
C14	0.49771 (9)	0.31234 (18)	0.46414 (8)	0.0246 (3)	
H14	0.5108	0.3709	0.5002	0.030*	
C15	0.46549 (9)	0.16959 (18)	0.47144 (7)	0.0248 (3)	
H15	0.4561	0.1308	0.5125	0.030*	
C16	0.44701 (9)	0.08330 (17)	0.41872 (7)	0.0227 (3)	
H16	0.4254	−0.0147	0.4240	0.027*	
C31	0.31019 (8)	0.37996 (16)	0.14954 (7)	0.0186 (3)	

H31	0.3584	0.4054	0.1319	0.022*	
C32	0.24329 (8)	0.45662 (16)	0.12170 (6)	0.0178 (3)	
O32	0.17788 (6)	0.43849 (12)	0.14132 (5)	0.0231 (2)	
S321	0.17866 (3)	0.61557 (6)	0.02510 (2)	0.0216 (2)	0.9387 (19)
C322	0.25716 (9)	0.55556 (16)	0.06735 (7)	0.0191 (3)	0.9387 (19)
C323	0.32268 (15)	0.6140 (3)	0.04229 (13)	0.0228 (4)	0.9387 (19)
H323	0.3719	0.5940	0.0591	0.027*	0.9387 (19)
C324	0.31042 (13)	0.7066 (6)	−0.0109 (3)	0.0252 (6)	0.9387 (19)
H324	0.3500	0.7563	−0.0336	0.030*	0.9387 (19)
C325	0.23486 (12)	0.7161 (3)	−0.02602 (8)	0.0234 (5)	0.9387 (19)
H325	0.2156	0.7721	−0.0609	0.028*	0.9387 (19)
S421	0.3446 (5)	0.6175 (16)	0.0470 (6)	0.0228 (4)	0.0613 (19)
C422	0.25716 (9)	0.55556 (16)	0.06735 (7)	0.0191 (3)	0.0613 (19)
C423	0.2005 (8)	0.620 (4)	0.0322 (14)	0.02162 (13)	0.0613 (19)
H423	0.1477	0.5983	0.0375	0.026*	0.0613 (19)
C424	0.2297 (14)	0.722 (5)	−0.0131 (18)	0.0234 (5)	0.0613 (19)
H424	0.1988	0.7815	−0.0404	0.028*	0.0613 (19)
C425	0.3078 (15)	0.726 (12)	−0.013 (4)	0.0252 (6)	0.0613 (19)
H425	0.3378	0.7809	−0.0424	0.030*	0.0613 (19)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0142 (6)	0.0194 (6)	0.0219 (6)	0.0011 (5)	−0.0019 (5)	0.0006 (5)
C2	0.0161 (7)	0.0173 (7)	0.0230 (7)	−0.0005 (5)	−0.0023 (5)	−0.0018 (6)
O2	0.0147 (5)	0.0288 (6)	0.0315 (6)	0.0003 (4)	0.0004 (4)	0.0041 (5)
C3	0.0140 (7)	0.0169 (6)	0.0207 (7)	0.0008 (5)	−0.0022 (5)	−0.0045 (5)
C3A	0.0175 (7)	0.0150 (6)	0.0192 (6)	0.0008 (5)	−0.0017 (5)	−0.0040 (5)
C4	0.0159 (7)	0.0175 (6)	0.0234 (7)	0.0019 (5)	−0.0019 (6)	−0.0035 (5)
C5	0.0150 (7)	0.0224 (7)	0.0269 (7)	−0.0006 (6)	0.0023 (6)	−0.0044 (6)
C6	0.0228 (8)	0.0217 (7)	0.0219 (7)	−0.0024 (6)	0.0032 (6)	−0.0019 (6)
C7	0.0227 (8)	0.0185 (7)	0.0203 (7)	0.0005 (6)	−0.0016 (6)	−0.0014 (6)
C7A	0.0149 (7)	0.0160 (6)	0.0211 (7)	0.0008 (5)	−0.0013 (5)	−0.0054 (5)
C1	0.0162 (7)	0.0181 (7)	0.0248 (7)	0.0030 (5)	−0.0039 (6)	0.0001 (6)
C11	0.0109 (6)	0.0185 (7)	0.0261 (7)	0.0033 (5)	−0.0023 (5)	0.0005 (6)
C12	0.0164 (7)	0.0231 (7)	0.0259 (8)	−0.0017 (6)	0.0029 (6)	0.0019 (6)
C13	0.0172 (7)	0.0214 (7)	0.0345 (8)	−0.0028 (6)	0.0010 (6)	−0.0035 (6)
C14	0.0191 (8)	0.0261 (8)	0.0288 (8)	0.0052 (6)	−0.0035 (6)	−0.0059 (6)
C15	0.0245 (8)	0.0270 (8)	0.0230 (7)	0.0058 (6)	−0.0022 (6)	0.0025 (6)
C16	0.0197 (8)	0.0194 (7)	0.0289 (8)	0.0015 (6)	−0.0023 (6)	0.0039 (6)
C31	0.0156 (7)	0.0180 (7)	0.0223 (7)	−0.0003 (5)	−0.0015 (5)	−0.0027 (6)
C32	0.0173 (7)	0.0164 (7)	0.0198 (7)	−0.0003 (5)	−0.0030 (5)	−0.0046 (5)
O32	0.0169 (5)	0.0267 (6)	0.0257 (5)	0.0012 (4)	−0.0006 (4)	0.0033 (4)
S321	0.0202 (2)	0.0234 (2)	0.0213 (2)	0.00105 (19)	−0.00487 (17)	0.00193 (16)
C322	0.0196 (7)	0.0171 (6)	0.0206 (7)	0.0024 (5)	−0.0031 (6)	−0.0037 (5)
C323	0.0154 (12)	0.0235 (7)	0.0294 (9)	−0.0012 (11)	−0.0011 (11)	−0.0019 (6)
C324	0.0289 (9)	0.020 (2)	0.0271 (9)	0.0026 (7)	0.0055 (7)	0.0018 (9)
C325	0.0326 (9)	0.0207 (8)	0.0168 (11)	0.0030 (7)	0.0005 (7)	−0.0003 (9)

S421	0.0154 (12)	0.0235 (7)	0.0294 (9)	−0.0012 (11)	−0.0011 (11)	−0.0019 (6)
C422	0.0196 (7)	0.0171 (6)	0.0206 (7)	0.0024 (5)	−0.0031 (6)	−0.0037 (5)
C423	0.0202 (2)	0.0234 (2)	0.0213 (2)	0.00105 (19)	−0.00487 (17)	0.00193 (16)
C424	0.0326 (9)	0.0207 (8)	0.0168 (11)	0.0030 (7)	0.0005 (7)	−0.0003 (9)
C425	0.0289 (9)	0.020 (2)	0.0271 (9)	0.0026 (7)	0.0055 (7)	0.0018 (9)

Geometric parameters (Å, º)

N1—C2	1.3723 (18)	C13—H13	0.9500
N1—C7A	1.4048 (19)	C14—C15	1.388 (2)
N1—C1	1.4600 (18)	C14—H14	0.9500
C2—O2	1.2218 (18)	C15—C16	1.390 (2)
C2—C3	1.515 (2)	C15—H15	0.9500
C3—C31	1.346 (2)	C16—H16	0.9500
C3—C3A	1.464 (2)	C31—C32	1.475 (2)
C3A—C4	1.393 (2)	C31—H31	0.9500
C3A—C7A	1.411 (2)	C32—O32	1.2287 (18)
C4—C5	1.402 (2)	C32—C322	1.466 (2)
C4—H4	0.9500	S321—C325	1.711 (2)
C5—C6	1.390 (2)	S321—C322	1.7239 (15)
C5—H5	0.9500	C322—C323	1.365 (3)
C6—C7	1.396 (2)	C323—C324	1.410 (3)
C6—H6	0.9500	C323—H323	0.9500
C7—C7A	1.384 (2)	C324—C325	1.363 (3)
C7—H7	0.9500	C324—H324	0.9500
C1—C11	1.513 (2)	C325—H325	0.9500
C1—H1A	0.9900	S421—C425	1.716 (10)
C1—H1B	0.9900	C423—C424	1.414 (10)
C11—C16	1.385 (2)	C423—H423	0.9500
C11—C12	1.400 (2)	C424—C425	1.366 (10)
C12—C13	1.386 (2)	C424—H424	0.9500
C12—H12	0.9500	C425—H425	0.9500
C13—C14	1.390 (2)		

C2—N1—C7A	110.60 (12)	C12—C13—C14	119.83 (14)
C2—N1—C1	123.38 (12)	C12—C13—H13	120.1
C7A—N1—C1	125.79 (12)	C14—C13—H13	120.1
O2—C2—N1	125.88 (14)	C15—C14—C13	119.86 (14)
O2—C2—C3	127.38 (13)	C15—C14—H14	120.1
N1—C2—C3	106.74 (12)	C13—C14—H14	120.1
C31—C3—C3A	137.50 (14)	C14—C15—C16	120.07 (15)
C31—C3—C2	116.76 (13)	C14—C15—H15	120.0
C3A—C3—C2	105.73 (12)	C16—C15—H15	120.0
C4—C3A—C7A	119.04 (13)	C11—C16—C15	120.67 (14)
C4—C3A—C3	134.25 (13)	C11—C16—H16	119.7
C7A—C3A—C3	106.71 (12)	C15—C16—H16	119.7
C3A—C4—C5	119.10 (14)	C3—C31—C32	127.87 (14)
C3A—C4—H4	120.5	C3—C31—H31	116.1

C5—C4—H4	120.5	C32—C31—H31	116.1
C6—C5—C4	120.50 (14)	O32—C32—C322	119.87 (13)
C6—C5—H5	119.7	O32—C32—C31	123.00 (13)
C4—C5—H5	119.7	C322—C32—C31	117.11 (13)
C5—C6—C7	121.49 (14)	C325—S321—C322	91.72 (8)
C5—C6—H6	119.3	C323—C322—C32	132.06 (16)
C7—C6—H6	119.3	C323—C322—S321	110.59 (14)
C7A—C7—C6	117.41 (14)	C32—C322—S321	117.35 (11)
C7A—C7—H7	121.3	C322—C323—C324	113.7 (2)
C6—C7—H7	121.3	C322—C323—H323	123.1
C7—C7A—N1	127.37 (13)	C324—C323—H323	123.1
C7—C7A—C3A	122.45 (14)	C325—C324—C323	111.78 (19)
N1—C7A—C3A	110.18 (12)	C325—C324—H324	124.1
N1—C1—C11	111.38 (12)	C323—C324—H324	124.1
N1—C1—H1A	109.4	C324—C325—S321	112.17 (15)
C11—C1—H1A	109.4	C324—C325—H325	123.9
N1—C1—H1B	109.4	S321—C325—H325	123.9
C11—C1—H1B	109.4	C424—C423—H423	124.0
H1A—C1—H1B	108.0	C425—C424—C423	112.0 (11)
C16—C11—C12	118.86 (14)	C425—C424—H424	124.0
C16—C11—C1	121.11 (13)	C423—C424—H424	124.0
C12—C11—C1	120.01 (13)	C424—C425—S421	111.4 (10)
C13—C12—C11	120.70 (14)	C424—C425—H425	124.3
C13—C12—H12	119.7	S421—C425—H425	124.3
C11—C12—H12	119.7		

C7A—N1—C2—O2	179.62 (14)	C7A—N1—C1—C11	−79.03 (17)
C1—N1—C2—O2	4.8 (2)	N1—C1—C11—C16	113.39 (15)
C7A—N1—C2—C3	0.32 (15)	N1—C1—C11—C12	−65.13 (18)
C1—N1—C2—C3	−174.45 (12)	C16—C11—C12—C13	0.1 (2)
O2—C2—C3—C31	−2.0 (2)	C1—C11—C12—C13	178.62 (14)
N1—C2—C3—C31	177.30 (12)	C11—C12—C13—C14	0.1 (2)
O2—C2—C3—C3A	179.15 (14)	C12—C13—C14—C15	−0.4 (2)
N1—C2—C3—C3A	−1.56 (15)	C13—C14—C15—C16	0.7 (2)
C31—C3—C3A—C4	3.2 (3)	C12—C11—C16—C15	0.2 (2)
C2—C3—C3A—C4	−178.33 (15)	C1—C11—C16—C15	−178.37 (14)
C31—C3—C3A—C7A	−176.30 (17)	C14—C15—C16—C11	−0.5 (2)
C2—C3—C3A—C7A	2.19 (14)	C3A—C3—C31—C32	−2.7 (3)
C7A—C3A—C4—C5	0.6 (2)	C2—C3—C31—C32	178.91 (13)
C3—C3A—C4—C5	−178.82 (14)	C3—C31—C32—O32	2.6 (2)
C3A—C4—C5—C6	−0.1 (2)	C3—C31—C32—C322	−175.99 (14)
C4—C5—C6—C7	−0.2 (2)	O32—C32—C322—C323	168.3 (2)
C5—C6—C7—C7A	−0.1 (2)	C31—C32—C322—C323	−13.1 (3)
C6—C7—C7A—N1	−178.46 (14)	O32—C32—C322—S321	−11.11 (18)
C6—C7—C7A—C3A	0.7 (2)	C31—C32—C322—S321	167.55 (10)
C2—N1—C7A—C7	−179.64 (14)	C325—S321—C322—C323	0.66 (18)
C1—N1—C7A—C7	−5.0 (2)	C325—S321—C322—C32	−179.84 (13)
C2—N1—C7A—C3A	1.12 (16)	C32—C322—C323—C324	−179.6 (4)

C1—N1—C7A—C3A	175.74 (12)	S321—C322—C323—C324	−0.2 (4)
C4—C3A—C7A—C7	−1.0 (2)	C322—C323—C324—C325	−0.6 (6)
C3—C3A—C7A—C7	178.62 (13)	C323—C324—C325—S321	1.1 (6)
C4—C3A—C7A—N1	178.33 (12)	C322—S321—C325—C324	−1.0 (4)
C3—C3A—C7A—N1	−2.09 (15)	C423—C424—C425—S421	−6 (10)
C2—N1—C1—C11	94.94 (16)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O2^{i}	0.95	2.59	3.5058 (19)	161
C323—H323···*Cg*2^{ii}	0.95	2.93	3.744 (3)	145

Symmetry codes: (i) $x-1/2, y, -z+1/2$; (ii) $-x+1, y+1/2, -z+1/2$.

(*E*)-1-Benzyl-5-chloro-3-[2-(4-chlorophenyl)-2-oxoethylidene]indolin-2-one (IIh)

Crystal data

$C_{23}H_{15}Cl_2NO_2$
$M_r = 408.26$
Triclinic, $P\overline{1}$
$a = 8.2010$ (6) Å
$b = 9.7629$ (7) Å
$c = 12.1740$ (9) Å
$\alpha = 76.755$ (3)°
$\beta = 87.675$ (3)°
$\gamma = 76.211$ (3)°
$V = 921.34$ (12) Å^3
$Z = 2$
$F(000) = 420$
$D_x = 1.472$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4558 reflections
$\theta = 2.2$–$28.3°$
$\mu = 0.37$ mm^{-1}
$T = 100$ K
Needle, red
0.40 × 0.16 × 0.07 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)
$T_{min} = 0.924$, $T_{max} = 0.974$
51788 measured reflections
4558 independent reflections
3867 reflections with $I > 2\sigma(I)$
$R_{int} = 0.053$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.2°$
$h = -10\rightarrow10$
$k = -13\rightarrow13$
$l = -16\rightarrow16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.082$
$S = 1.11$
4558 reflections
253 parameters
0 restraints
Primary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0266P)^2 + 0.6446P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.44$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ 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	0.40211 (15)	0.82267 (12)	0.62793 (10)	0.0140 (2)
C2	0.31723 (17)	0.80095 (15)	0.54075 (11)	0.0146 (3)
O2	0.24859 (13)	0.89462 (11)	0.46090 (9)	0.0193 (2)
C3	0.32530 (17)	0.63951 (15)	0.56422 (11)	0.0141 (3)
C3A	0.41760 (16)	0.57654 (15)	0.67151 (11)	0.0132 (3)
C4	0.46454 (17)	0.43662 (15)	0.73834 (11)	0.0147 (3)
H4	0.4379	0.3570	0.7165	0.018*
C5	0.55211 (17)	0.41803 (15)	0.83839 (12)	0.0152 (3)
Cl5	0.61070 (5)	0.24487 (4)	0.92585 (3)	0.01917 (9)
C6	0.59452 (17)	0.53146 (15)	0.87280 (12)	0.0159 (3)
H6	0.6557	0.5135	0.9413	0.019*
C7	0.54707 (17)	0.67156 (15)	0.80651 (11)	0.0150 (3)
H7	0.5737	0.7508	0.8289	0.018*
C7A	0.45999 (17)	0.69159 (15)	0.70695 (11)	0.0134 (3)
C1	0.41607 (18)	0.96427 (15)	0.64222 (12)	0.0153 (3)
H1A	0.3970	1.0344	0.5683	0.018*
H1B	0.5312	0.9566	0.6686	0.018*
C11	0.29126 (18)	1.01998 (14)	0.72581 (12)	0.0156 (3)
C12	0.11934 (18)	1.04709 (15)	0.70389 (12)	0.0174 (3)
H12	0.0814	1.0336	0.6353	0.021*
C13	0.0037 (2)	1.09355 (16)	0.78175 (13)	0.0209 (3)
H13	−0.1130	1.1098	0.7672	0.025*
C14	0.0588 (2)	1.11640 (16)	0.88145 (13)	0.0230 (3)
H14	−0.0203	1.1473	0.9352	0.028*
C15	0.2293 (2)	1.09391 (17)	0.90210 (13)	0.0242 (3)
H15	0.2669	1.1118	0.9691	0.029*
C16	0.3451 (2)	1.04509 (16)	0.82469 (12)	0.0199 (3)
H16	0.4618	1.0288	0.8395	0.024*
C31	0.25083 (17)	0.59265 (15)	0.48826 (12)	0.0148 (3)
H31	0.1945	0.6651	0.4268	0.018*
C32	0.24591 (17)	0.44181 (15)	0.48870 (11)	0.0137 (3)
O32	0.32236 (13)	0.33852 (11)	0.55942 (8)	0.0186 (2)
C321	0.14826 (17)	0.41665 (15)	0.39646 (11)	0.0134 (3)
C322	0.15414 (17)	0.27330 (15)	0.39372 (12)	0.0152 (3)
H322	0.2117	0.1970	0.4523	0.018*
C323	0.07720 (18)	0.24067 (15)	0.30674 (12)	0.0166 (3)
H323	0.0828	0.1429	0.3048	0.020*
C324	−0.00812 (17)	0.35356 (16)	0.22254 (12)	0.0164 (3)
Cl34	−0.10252 (5)	0.31443 (4)	0.11194 (3)	0.02314 (9)

C325	−0.02016 (18)	0.49679 (16)	0.22460 (12)	0.0183 (3)
H325	−0.0814	0.5728	0.1673	0.022*
C326	0.05875 (18)	0.52767 (15)	0.31180 (12)	0.0166 (3)
H326	0.0516	0.6256	0.3138	0.020*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0166 (6)	0.0120 (5)	0.0140 (5)	−0.0036 (4)	−0.0012 (4)	−0.0035 (4)
C2	0.0148 (6)	0.0149 (6)	0.0149 (6)	−0.0036 (5)	0.0005 (5)	−0.0046 (5)
O2	0.0242 (5)	0.0155 (5)	0.0177 (5)	−0.0048 (4)	−0.0059 (4)	−0.0013 (4)
C3	0.0144 (6)	0.0136 (6)	0.0136 (6)	−0.0028 (5)	0.0013 (5)	−0.0025 (5)
C3A	0.0121 (6)	0.0163 (6)	0.0124 (6)	−0.0035 (5)	0.0007 (5)	−0.0051 (5)
C4	0.0147 (6)	0.0145 (6)	0.0152 (6)	−0.0030 (5)	0.0002 (5)	−0.0041 (5)
C5	0.0150 (6)	0.0137 (6)	0.0148 (6)	−0.0012 (5)	0.0005 (5)	−0.0015 (5)
Cl5	0.02428 (18)	0.01469 (16)	0.01655 (16)	−0.00253 (13)	−0.00516 (13)	−0.00074 (12)
C6	0.0155 (6)	0.0192 (7)	0.0130 (6)	−0.0033 (5)	−0.0003 (5)	−0.0046 (5)
C7	0.0151 (6)	0.0165 (7)	0.0146 (6)	−0.0042 (5)	0.0005 (5)	−0.0056 (5)
C7A	0.0126 (6)	0.0135 (6)	0.0137 (6)	−0.0022 (5)	0.0021 (5)	−0.0034 (5)
C1	0.0179 (7)	0.0126 (6)	0.0167 (6)	−0.0055 (5)	−0.0002 (5)	−0.0037 (5)
C11	0.0219 (7)	0.0101 (6)	0.0149 (6)	−0.0049 (5)	0.0009 (5)	−0.0022 (5)
C12	0.0209 (7)	0.0136 (6)	0.0180 (7)	−0.0050 (5)	−0.0001 (5)	−0.0033 (5)
C13	0.0230 (7)	0.0138 (7)	0.0253 (8)	−0.0045 (6)	0.0032 (6)	−0.0037 (6)
C14	0.0333 (8)	0.0141 (7)	0.0195 (7)	−0.0027 (6)	0.0079 (6)	−0.0039 (6)
C15	0.0366 (9)	0.0190 (7)	0.0162 (7)	−0.0025 (6)	−0.0021 (6)	−0.0063 (6)
C16	0.0248 (7)	0.0161 (7)	0.0191 (7)	−0.0039 (6)	−0.0031 (6)	−0.0049 (5)
C31	0.0140 (6)	0.0153 (6)	0.0147 (6)	−0.0023 (5)	−0.0005 (5)	−0.0038 (5)
C32	0.0131 (6)	0.0154 (6)	0.0133 (6)	−0.0038 (5)	0.0011 (5)	−0.0042 (5)
O32	0.0224 (5)	0.0164 (5)	0.0163 (5)	−0.0038 (4)	−0.0044 (4)	−0.0024 (4)
C321	0.0126 (6)	0.0143 (6)	0.0138 (6)	−0.0035 (5)	0.0009 (5)	−0.0040 (5)
C322	0.0139 (6)	0.0141 (6)	0.0168 (6)	−0.0027 (5)	−0.0007 (5)	−0.0025 (5)
C323	0.0171 (7)	0.0142 (6)	0.0200 (7)	−0.0049 (5)	−0.0001 (5)	−0.0056 (5)
C324	0.0153 (6)	0.0210 (7)	0.0150 (6)	−0.0058 (5)	−0.0006 (5)	−0.0067 (5)
Cl34	0.02808 (19)	0.02353 (19)	0.02023 (17)	−0.00608 (15)	−0.00785 (14)	−0.00842 (14)
C325	0.0194 (7)	0.0162 (7)	0.0182 (7)	−0.0031 (5)	−0.0050 (5)	−0.0020 (5)
C326	0.0188 (7)	0.0135 (6)	0.0177 (7)	−0.0034 (5)	−0.0019 (5)	−0.0040 (5)

Geometric parameters (Å, º)

N1—C2	1.3745 (17)	C12—H12	0.9500
N1—C7A	1.4043 (17)	C13—C14	1.394 (2)
N1—C1	1.4634 (17)	C13—H13	0.9500
C2—O2	1.2206 (17)	C14—C15	1.386 (2)
C2—C3	1.5217 (19)	C14—H14	0.9500
C3—C31	1.3456 (19)	C15—C16	1.391 (2)
C3—C3A	1.4654 (18)	C15—H15	0.9500
C3A—C4	1.3933 (19)	C16—H16	0.9500
C3A—C7A	1.4103 (19)	C31—C32	1.4815 (19)

C4—C5	1.3927 (19)	C31—H31	0.9500
C4—H4	0.9500	C32—O32	1.2230 (17)
C5—C6	1.3896 (19)	C32—C321	1.5020 (18)
C5—Cl5	1.7470 (14)	C321—C326	1.3951 (19)
C6—C7	1.393 (2)	C321—C322	1.3968 (19)
C6—H6	0.9500	C322—C323	1.3876 (19)
C7—C7A	1.3829 (18)	C322—H322	0.9500
C7—H7	0.9500	C323—C324	1.389 (2)
C1—C11	1.5109 (19)	C323—H323	0.9500
C1—H1A	0.9900	C324—C325	1.384 (2)
C1—H1B	0.9900	C324—Cl34	1.7377 (14)
C11—C16	1.3907 (19)	C325—C326	1.3899 (19)
C11—C12	1.396 (2)	C325—H325	0.9500
C12—C13	1.386 (2)	C326—H326	0.9500

C2—N1—C7A	110.46 (11)	C11—C12—H12	119.8
C2—N1—C1	124.77 (11)	C12—C13—C14	120.03 (15)
C7A—N1—C1	124.51 (11)	C12—C13—H13	120.0
O2—C2—N1	125.86 (13)	C14—C13—H13	120.0
O2—C2—C3	127.42 (12)	C15—C14—C13	119.90 (14)
N1—C2—C3	106.72 (11)	C15—C14—H14	120.1
C31—C3—C3A	137.31 (13)	C13—C14—H14	120.1
C31—C3—C2	117.11 (12)	C14—C15—C16	119.96 (14)
C3A—C3—C2	105.57 (11)	C14—C15—H15	120.0
C4—C3A—C7A	119.48 (12)	C16—C15—H15	120.0
C4—C3A—C3	133.79 (12)	C11—C16—C15	120.49 (14)
C7A—C3A—C3	106.73 (12)	C11—C16—H16	119.8
C5—C4—C3A	117.39 (13)	C15—C16—H16	119.8
C5—C4—H4	121.3	C3—C31—C32	127.92 (13)
C3A—C4—H4	121.3	C3—C31—H31	116.0
C6—C5—C4	123.02 (13)	C32—C31—H31	116.0
C6—C5—Cl5	118.04 (10)	O32—C32—C31	122.03 (12)
C4—C5—Cl5	118.94 (11)	O32—C32—C321	119.73 (12)
C5—C6—C7	119.76 (12)	C31—C32—C321	118.20 (12)
C5—C6—H6	120.1	C326—C321—C322	118.83 (12)
C7—C6—H6	120.1	C326—C321—C32	123.65 (12)
C7A—C7—C6	117.83 (13)	C322—C321—C32	117.47 (12)
C7A—C7—H7	121.1	C323—C322—C321	121.00 (13)
C6—C7—H7	121.1	C323—C322—H322	119.5
C7—C7A—N1	126.98 (12)	C321—C322—H322	119.5
C7—C7A—C3A	122.53 (13)	C322—C323—C324	118.76 (13)
N1—C7A—C3A	110.50 (11)	C322—C323—H323	120.6
N1—C1—C11	112.07 (11)	C324—C323—H323	120.6
N1—C1—H1A	109.2	C325—C324—C323	121.59 (13)
C11—C1—H1A	109.2	C325—C324—Cl34	119.10 (11)
N1—C1—H1B	109.2	C323—C324—Cl34	119.31 (11)
C11—C1—H1B	109.2	C324—C325—C326	118.95 (13)
H1A—C1—H1B	107.9	C324—C325—H325	120.5

C16—C11—C12	119.26 (14)	C326—C325—H325	120.5
C16—C11—C1	120.91 (13)	C325—C326—C321	120.84 (13)
C12—C11—C1	119.84 (12)	C325—C326—H326	119.6
C13—C12—C11	120.32 (14)	C321—C326—H326	119.6
C13—C12—H12	119.8		

C7A—N1—C2—O2	177.96 (14)	C7A—N1—C1—C11	−74.84 (16)
C1—N1—C2—O2	3.5 (2)	N1—C1—C11—C16	117.93 (14)
C7A—N1—C2—C3	−1.68 (15)	N1—C1—C11—C12	−62.18 (16)
C1—N1—C2—C3	−176.11 (12)	C16—C11—C12—C13	−2.3 (2)
O2—C2—C3—C31	1.0 (2)	C1—C11—C12—C13	177.82 (13)
N1—C2—C3—C31	−179.37 (12)	C11—C12—C13—C14	1.4 (2)
O2—C2—C3—C3A	−178.48 (14)	C12—C13—C14—C15	0.6 (2)
N1—C2—C3—C3A	1.16 (14)	C13—C14—C15—C16	−1.6 (2)
C31—C3—C3A—C4	−0.2 (3)	C12—C11—C16—C15	1.3 (2)
C2—C3—C3A—C4	179.12 (15)	C1—C11—C16—C15	−178.84 (13)
C31—C3—C3A—C7A	−179.53 (16)	C14—C15—C16—C11	0.7 (2)
C2—C3—C3A—C7A	−0.22 (14)	C3A—C3—C31—C32	−3.2 (3)
C7A—C3A—C4—C5	0.1 (2)	C2—C3—C31—C32	177.52 (13)
C3—C3A—C4—C5	−179.15 (14)	C3—C31—C32—O32	−4.6 (2)
C3A—C4—C5—C6	−0.5 (2)	C3—C31—C32—C321	177.57 (14)
C3A—C4—C5—Cl5	179.04 (10)	O32—C32—C321—C326	−179.40 (13)
C4—C5—C6—C7	0.8 (2)	C31—C32—C321—C326	−1.5 (2)
Cl5—C5—C6—C7	−178.71 (11)	O32—C32—C321—C322	−2.09 (19)
C5—C6—C7—C7A	−0.7 (2)	C31—C32—C321—C322	175.80 (12)
C6—C7—C7A—N1	−179.42 (13)	C326—C321—C322—C323	2.0 (2)
C6—C7—C7A—C3A	0.4 (2)	C32—C321—C322—C323	−175.42 (13)
C2—N1—C7A—C7	−178.55 (13)	C321—C322—C323—C324	−0.8 (2)
C1—N1—C7A—C7	−4.1 (2)	C322—C323—C324—C325	−1.1 (2)
C2—N1—C7A—C3A	1.61 (16)	C322—C323—C324—Cl34	179.06 (11)
C1—N1—C7A—C3A	176.06 (12)	C323—C324—C325—C326	1.6 (2)
C4—C3A—C7A—C7	−0.1 (2)	Cl34—C324—C325—C326	−178.54 (11)
C3—C3A—C7A—C7	179.36 (12)	C324—C325—C326—C321	−0.3 (2)
C4—C3A—C7A—N1	179.76 (12)	C322—C321—C326—C325	−1.5 (2)
C3—C3A—C7A—N1	−0.79 (15)	C32—C321—C326—C325	175.79 (13)
C2—N1—C1—C11	98.83 (15)		