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The crystal structures of four N-(4-halophenyl)-4-oxo-4H-chromene-3-carboxamides

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Four N-(4-halophenyl)-4-oxo-4H-chromene-3-carboxamides (halo = F, Cl, Br and I), N-(4-fluorophenyl)-4-oxo-4H-chromene-3-carboxamide, $C_{16}H_{10}FNO_3$, N-(4-chlorophenyl)-4-oxo-4H-chromene-3-carboxamide, $C_{16}H_{10}ClNO_3$, N-(4-bromophenyl)-4-oxo-4H-chromene-3-carboxamide, $C_{16}H_{10}BrNO_3$, N-(4-iodophenyl)-4-oxo-4H-chromene-3-carboxamide, $C_{16}H_{10}INO_3$, have been structurally characterized. The molecules are essentially planar and each exhibits an anti conformation with respect to the C—N rotamer of the amide and a cis geometry with respect to the relative positions of the C_{arom}—C_{arom} bond of the chromone ring and the carbonyl group of the amide. The structures each exhibit an intramolecular hydrogen-bonding network comprising an N—H⋯O hydrogen bond between the amide N atom and the O atom of the carbonyl group of the pyrone ring, forming an $S(6)$ ring, and a weak C_{arom}—H⋯O interaction with the O atom of the carbonyl group of the amide as acceptor, which forms another $S(6)$ ring. All four compounds have the same supramolecular structure, consisting of $R_2^2(13)$ rings that are propagated along the a-axis direction by unit translation. There is π–π stacking involving inversion-related molecules in each structure.
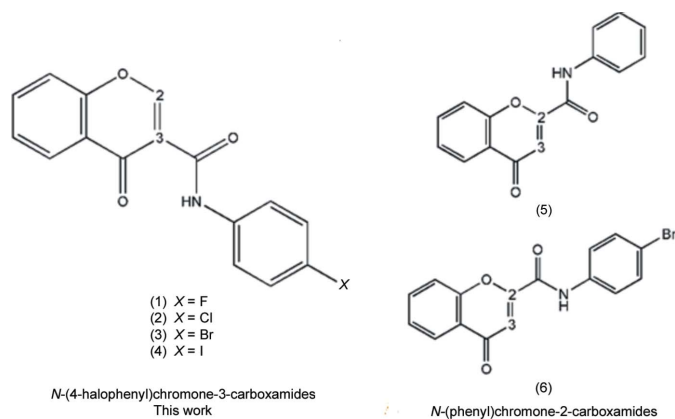
1. Chemical context

Chromones are a group of natural and synthetic oxygen heterocyclic compounds having a high degree of chemical diversity that is frequently linked to a broad array of biological activities (Gaspar et al. 2014). Parkinson's disease (PD) is a degenerative disorder of the central nervous system with an aetiology not yet completely clarified. There is no cure for PD, but medications, surgery and multidisciplinary management can provide relief from the symptoms. PD seems to be associated with a decrease in central levels of dopamine triggered by oxidative stress. These processes, among other factors, are mediated by the isoform B of the monoamino oxidase (MAO-B). Hence, the search for novel agents that can selectively inhibit MAO-B is of paramount relevance. In this context, the decoration of chromone, a privileged structure for the discovery and development of new chemical entities (NCEs), have led to the preparation of chromone carboxamides and to promising outcomes since preliminary data indicate that chromone-3-carboxamides are selective MAO-B inhibitors (Gaspar, Reis et al., 2011; Gaspar, Silva et al., 2011).

Previous results showed that the carbonyl group of the chromone moiety and the amide function play an important role in the establishment of hydrogen interactions with the MAO-B active pocket. In addition, the presence of a phenyl substituent attached to the amide seems to play a pivotal role in the potency conveyed by the ligand (Helguera et al., 2013).
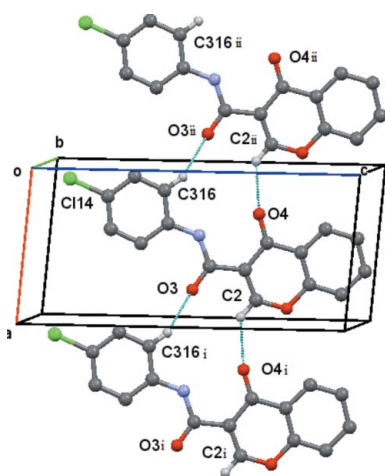
In this context, some *N*-(4-halophenyl)-4-oxo-4*H*-chromene-3-carboxamides (1)–(4), shown in the scheme, have been synthesized and structurally characterized in order to rationalize the structural factors that may affect the selectivity and the potency of their inhibitory activities towards MAO-B. These structures are compared with *N*-(4-phenyl)-4-oxo-4*H*-chromene-2-carboxamide and *N*-(4-bromophenyl)-4-oxo-4*H*-chromene-2-carboxamide, compounds (5) and (6) (Reis *et al.*, 2013; Gomes *et al.*, 2013), which do not show inhibitory activities against human MAO-B.

(1) *X* = F
(2) *X* = Cl
(3) *X* = Br
(4) *X* = I

N-(4-halophenyl)chromone-3-carboxamides
This work

(5)

(6)

N-(phenyl)chromone-2-carboxamides

2. Structural commentary

The structural analysis of (1)–(4) confirmed them to be *N*-(4-halophenyl)-4-oxo-4*H*-chromene-3-carboxamides with halo-substituents F (Fig. 1), Cl (Fig. 2), Br (Fig. 3) and I (Fig. 4), respectively, as depicted in the scheme. Figs. 1–4 show the displacement ellipsoid diagrams with the adopted labelling schemes. All compounds crystallize in the space group $P\bar{1}$. Compounds (1) and (2) are isostructural, as are compounds (3) and (4). The cell lengths are very similar in each pair of compounds.
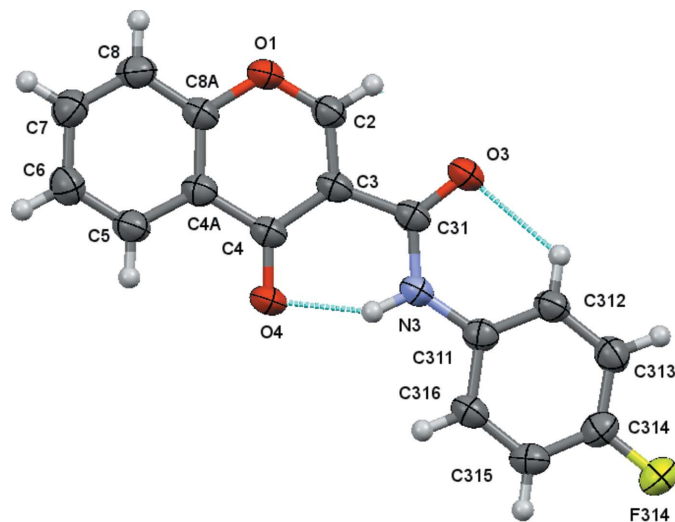
Figure 2
A view of the asymmetric unit of (2), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 80% probability level. Dashed lines indicate the intramolecular contacts.

Figure 3
A view of the asymmetric unit of (3), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 80% probability level. Dashed lines indicate the intramolecular contacts.

Figure 1
A view of the asymmetric unit of (1), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 80% probability level. Dashed lines indicate the intramolecular contacts.

Figure 4
A view of the asymmetric unit of (4), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 80% probability level. Dashed lines indicate the intramolecular contacts.
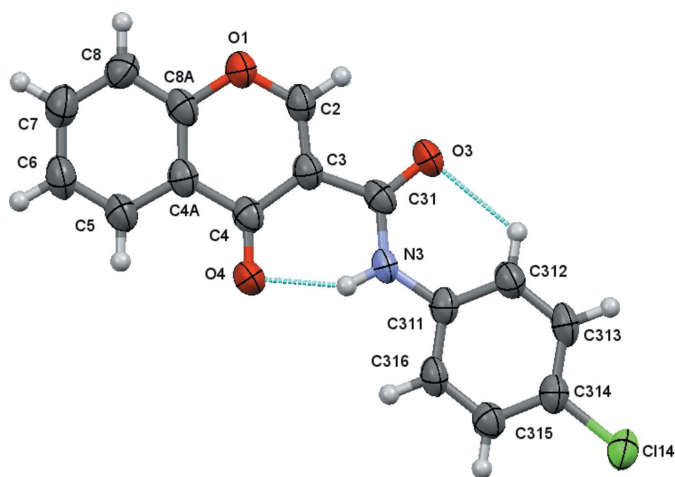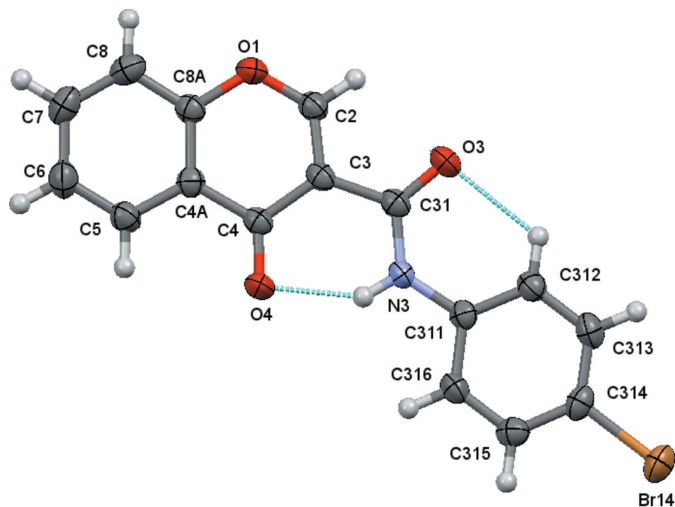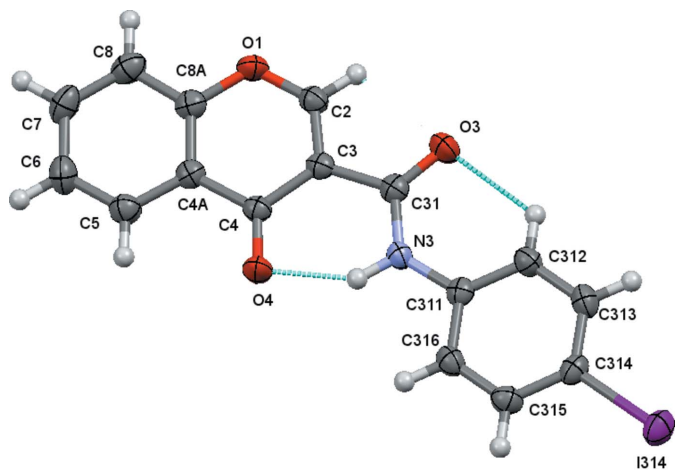
Table 1
Selected dihedral angles (°).

θ_1 is the dihedral angle between the mean planes of the chromene and phenyl rings and the phenyl ring. θ_2 is the dihedral angle between the mean plane of the chromene ring and the plane defined by atoms O2, C31 and N3. θ_3 is the dihedral angle between the mean planes of the phenyl ring and the plane defined by atoms O3, C31 and N3.

Compound	θ_1	θ_2	θ_3
(1)	2.51 (3)	5.51 (12)	5.05 (13)
(2)	1.95 (7)	5.7 (3)	4.4 (3)
(3)	4.90 (10)	2.0 (4)	2.9 (4)
(4)	5.37 (10)	1.8 (4)	3.6 (4)

The title compounds display similar structures, which are reflected in the molecular geometries and conformations; the values of the dihedral angles between the mean planes of the chromone ring and the exocyclic phenyl ring of the *N*-phenyl-4-oxo-4*H*-chromene-3-carboxamides are close to 2° in the case of the F, Cl pair [2.51 (3) and 1.95 (7)°, respectively,] and close to 5° for the Br, I pair [4.90 (10) and 5.37 (10)°, respectively]. In *N*-phenyl-4-oxo-4*H*-chromene-2-carboxamide (5) (Reis *et al.*, 2013), the dihedral angle between the mean planes of the chromone ring and the phenyl ring is 6.57° and in *N*-(4-bromophenyl)-4-oxo-4*H*-chromene-2-carboxamide (6), the structural isomer of (3) (Gomes *et al.*, 2013), the dihedral angle between the mean planes of the chromone ring and the phenyl ring is 5.0 (2)°. Selected dihedral angles are given in Table 1.

In (1) and (2), the maximum deviations from the mean plane of the 10 atoms of the chromone ring plus the three carboxamide atoms O3, C31 and N3, are 0.1220 (8) and 0.1319 (17) Å, respectively, both for atom O3 (r.m.s. deviations of fitted atoms = 0.0519 and 0.0571 Å, respectively). In (3) and (4), the deviations of O3 from the mean plane defined above are 0.0384 (14) and 0.0342 (15) Å, respectively (r.m.s. deviations of fitted atoms = 0.0314 Å in both compounds). In the case of (3) and (4), atom C2 shows the greatest deviation from the mean plane having deviations of 0.0569 (18) and 0.0596 (18) Å, respectively. These values indicate that the carboxamide groups are practically planar with the chromone ring, particularly in the case of the Br and I chromone carboxamide derivatives. This planarity may be related to the internal hydrogen-bond pattern in those molecules, which thus defines the molecular conformations.

The conformational features herein established are probably most relevant for the extrapolation of the inhibitory MAO-B activities of chromone carboxamides as they are related to the intermolecular forces responsible for enzyme–ligand binding affinity. The data can explain the MAO-B selectivity found for chromone-3-carboxamides (1)–(4), as opposed to the lack of activity presented by chromone-2-carboxamides (5) and (6). As seen in the scheme, (1)–(4) are *N*-(phenyl)-4-oxo-4*H*-chromene-**3**-carboxamides while (5) and (6) are *N*-(phenyl)-4-oxo-4*H*-chromene-**2**-carboxamides. As can be seen in Fig. 5, an *anti* conformation is adopted with respect to the C—N rotamer of the amide in all of the compounds. Nevertheless, due to the asymmetry of the chromone residue, the *anti* conformation can assume a *cis* (*a*) or *trans* (*b*) geometry with respect to the relative position of the

Table 2
Hydrogen-bond geometry (Å, °) for (1).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N3—H3⋯O4	0.896 (17)	1.901 (17)	2.7024 (13)	147.9 (15)
C312—H312⋯O3	0.95	2.26	2.8714 (15)	122
C2—H2⋯O4[i]	0.95	2.45	3.1645 (14)	132
C316—H316⋯O3[ii]	0.95	2.46	3.3160 (14)	149

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

Table 3
Hydrogen-bond geometry (Å, °) for (2).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N3—H3⋯O4	0.85 (3)	1.92 (3)	2.680 (3)	148 (3)
C312—H312⋯O3	0.95	2.29	2.892 (3)	121
C2—H2⋯O4[i]	0.95	2.47	3.194 (3)	133
C316—H316⋯O3[ii]	0.95	2.45	3.286 (3)	146

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

Table 4
Hydrogen-bond geometry (Å, °) for (3).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N3—H3⋯O4	0.86 (2)	1.95 (2)	2.695 (2)	145 (2)
C312—H312⋯O3	0.95	2.26	2.877 (2)	129
C2—H2⋯O4[i]	0.95	2.41	3.167 (2)	137
C316—H316⋯O3[ii]	0.95	2.47	3.314 (2)	148

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

Table 5
Hydrogen-bond geometry (Å, °) for (4).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N3—H3⋯O4	0.92 (2)	1.89 (2)	2.6977 (19)	145 (2)
C2—H2⋯O3	0.95	2.33	2.718 (2)	104
C312—H312⋯O3	0.95	2.27	2.881 (2)	122
C2—H2⋯O4[i]	0.95	2.44	3.185 (2)	136
C316—H316⋯O3[ii]	0.95	2.49	3.312 (2)	145

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

carbonyl O atom of the carboxamide and the C2$_{arom}$—C3$_{arom}$ bond of the chromone. Compounds (1)–(4) exhibit a *cis* relation between these bonds, as can be seen in the ellipsoid diagrams, Figs. 1–4. This molecular conformation permits the formation of two intramolecular hydrogen bonds, which generate a network that probably enhances their planarity. Details of the intramolecular hydrogen-bonding interactions are given in Tables 2 to 5. Specifically for each molecule, there is an intramolecular N—H⋯O hydrogen bond between the amide nitrogen and the oxygen atom of the carbonyl group, O4, of the chromone ring, forming an *S*(6) ring identified as ring *C*. In addition, the carbonyl oxygen of the amide, O3, acts as the acceptor for a weak interaction with an *ortho* hydrogen of the exocyclic phenyl ring, forming another S(6) ring, *B*. The corresponding *trans* structures (top right in Fig. 5) would probably only allow the formation of a weak hydrogen-bonding interaction with an *ortho* hydrogen atom of the exocyclic phenyl ring. It is interesting to compare the internal hydrogen-bonding network presented by the title compounds with those of the analogous 4-oxo-*N*-(substituted phenyl)-4*H*-
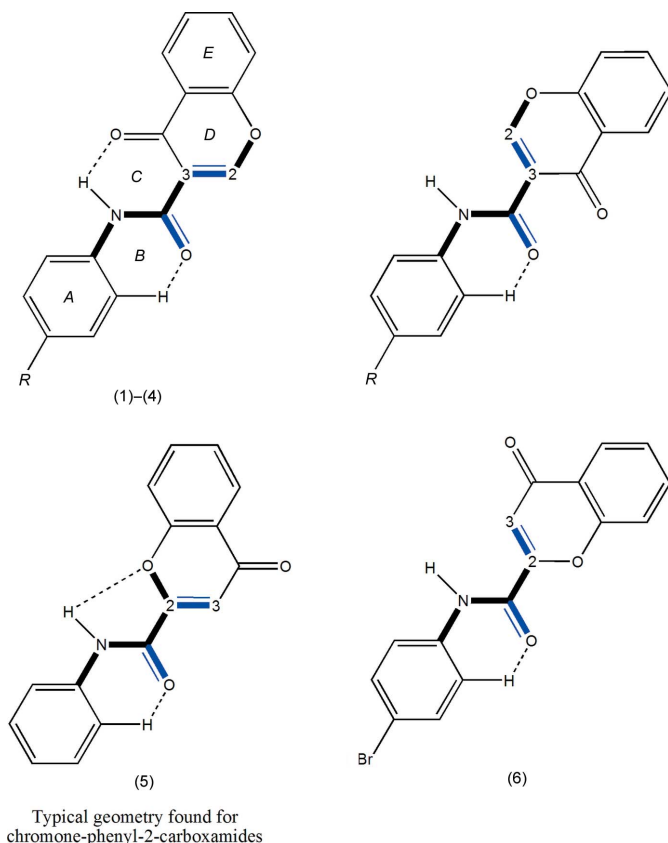
Figure 5
Anti-rotamer conformations around the C—N rotamer for the 3-carboxamides (top) and for the 2-carboxamide isomers (bottom), showing the relative positions of the $C3_{arom}$—$C2_{arom}$ bond of the chromone ring with respect to the carboxylic group of the amide: *cis* (right) or *trans* (left) geometries.

chromene-2-carboxamides (Reis *et al.*, 2013) and (Gomes *et al.*, 2013), compounds (5) and (6). Previous studies concerning the structures of the chromone-2-carboxamides show that the majority have geometries similar to compound (5), *e.g.* as in (1)–(4), they assume a *cis* conformation, but this is not the case for (6), the bromo isomer of (3), as shown in Fig. 5 (bottom right). In spite of this, none of this type of derivative displays inhibitory activity towards the MAO-B isoenzyme. When the geometries of the relative positions of rings *D* and *E* of the chromone residue with respect to rings *A* and *B* are compared, it can be seen that the effect of the 2/3 positional isomerism is to 'reflect' their relative positions while the effect of the *cis*/*trans* conformations is a 'twofold rotation' of the rings around the C_{amide}— $C_{chromone}$ bond. Those particular differences in conformation may condition the ability for docking when pharmacological activities are considered.

3. Supramolecular features

Intermolecular hydrogen-bonding information is given in Table 2 to 5. All compounds have the same supramolecular structure in which the C2—H2···O4($x + 1$, y, z) and C316—H316···O3($x − 1$, y, z) form $R_2^2(13)$ ring structures, which are
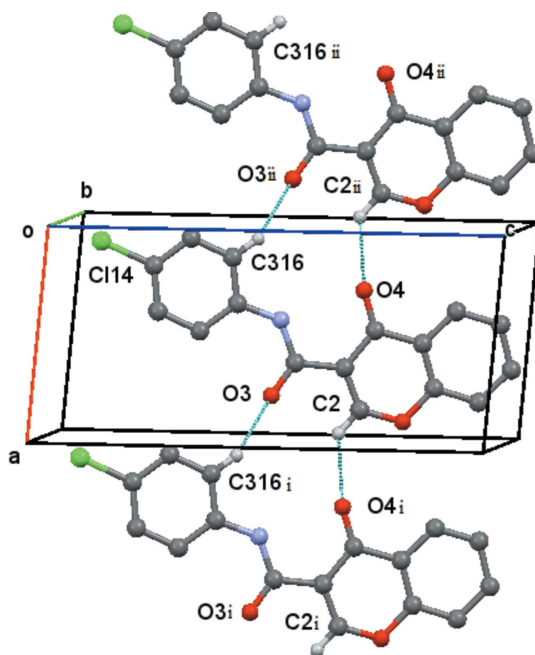
Figure 6
The distorted ladder formed by linked $R_2^2(13)$ rings in compound (3). The chain runs parallel to the *a* axis. Hydrogen bonds are indicated by blue dashed lines. Hydrogen atoms not involved in the hydrogen bonding have been omitted for clarity. A similar structure is found for compound (1) and all the halo-substituted compounds. [Symmetry codes: (i) $x + 1$, y, z; (ii) $x − 1$, y, x.]

propagated along the *a*-axis direction by unit translation. Fig. 6 shows the Cl compound, (3), as an example.

There is π–π stacking in each compound, involving inversion-related molecules in all compounds, Table 6.

4. Synthesis and crystallization

The title compounds were obtained by synthetic strategies described elsewhere (Cagide *et al.*, 2011). Chromone-3-carboxamides were synthesized using chromone-3-carboxylic acid as starting material which, after *in situ* activation with phosphorus(V) oxychloride (POCl₃) in dimethylformamide,

Table 6
π–π stacking (Å, °).

*Cg*1, *Cg*2, *Cg*3 and *Cg*7 [compound (6)] are the centroids of the rings containing atoms O1, C5, C311 and C211 [compound (6)], respectively. In contacts indicated *, the planes involved are inclined to each other, the perpendicular distance between the planes is an average value and the angle between the planes is given in place of a slippage. Only interplanar interactions with *Cg*···*Cg* distances ≤4.0 Å and with angles between the planes of <10° are included.

Compound	contact	distance	perp. dist.	angle between planes
(1)	*Cg*1···*Cg*3ⁱⁱⁱ	3.5187 (8)	3.3226*	1.77 (6)*
	*Cg*1···*Cg*3ⁱᵛ	3.543 (8)	3.3719*	1.77 (6)*
(2)	*Cg*1···*Cg*3ᵛ	3.5341 (17)	3.3573*	0.77 (13)*
	*Cg*2···*Cg*3ᵛⁱ	3.6691 (17)	3.3985*	3.14 (13)*
(3)	*Cg*1···*Cg*3ᵛ	3.5464 (11)	3.3342*	4.66 (9)*
(4)	*Cg*1···*Cg*3ⁱⁱⁱ	3.5721 (11)	3.3518*	5.37 (9)

Symmetry codes: (iii) $−x + 1$, $−y + 1$, $−z + 1$; (iv) $−x$, $−y + 2$, $−z$; (v) $−x + 1$, $−y$, $−z + 1$; (vi) $−x$, $−y$, $−z$.

Table 7
Experimental details.

	(1)	(2)	(3)	(4)
Crystal data				
Chemical formula	$C_{16}H_{10}FNO_3$	$C_{16}H_{10}ClNO_3$	$C_{16}H_{10}BrNO_3$	$C_{16}H_{10}INO_3$
M_r	283.25	299.70	344.16	391.15
Crystal system, space group	Triclinic, $P\bar{1}$	Triclinic, $P\bar{1}$	Triclinic, $P\bar{1}$	Triclinic, $P\bar{1}$
Temperature (K)	100	100	120	120
a, b, c (Å)	6.6213 (5), 7.0517 (5), 14.0864 (10)	6.6325 (12), 7.0577 (12), 14.671 (3)	6.6505 (5), 9.3580 (7), 11.0060 (8)	6.6750 (5), 9.4166 (7), 11.2673 (8)
α, β, γ (°)	101.957 (7), 90.047 (6), 106.657 (7)	103.536 (7), 89.714 (6), 105.589 (7)	100.280 (6), 90.461 (6), 100.884 (6)	100.974 (6), 90.769 (6), 100.062 (6)
V (Å^3)	615.17 (8)	641.9 (2)	661.24 (9)	683.77 (9)
Z	2	2	2	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.12	0.31	3.12	2.35
Crystal size (mm)	$0.46 \times 0.32 \times 0.02$	$0.17 \times 0.17 \times 0.04$	$0.58 \times 0.18 \times 0.06$	$0.46 \times 0.22 \times 0.05$
Data collection				
Diffractometer	Rigaku Saturn724+	Rigaku AFC12	Rigaku R-AXIS conversion	Rigaku R-AXIS conversion
Absorption correction	Multi-scan (*CrystalClear-SM Expert*; Rigaku, 2012)	Multi-scan (*CrystalClear-SM Expert*; Rigaku, 2012)	Multi-scan (*CrystalClear-SM Expert*; Rigaku, 2012)	Multi-scan (*CrystalClear-SM Expert*; Rigaku, 2012)
T_{min}, T_{max}	0.949, 0.998	0.950, 0.988	0.265, 0.835	0.411, 0.892
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8176, 2789, 2393	7435, 2265, 1668	9930, 3017, 2525	10032, 3095, 2819
R_{int}	0.056	0.078	0.045	0.026
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.649	0.598	0.649	0.649
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.044, 0.135, 1.06	0.056, 0.145, 0.99	0.027, 0.058, 0.94	0.018, 0.044, 1.03
No. of reflections	2789	2265	3017	3095
No. of parameters	194	194	194	194
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	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.41, −0.27	0.30, −0.65	0.53, −0.69	0.67, −0.32

Computer programs: *CrystalClear-SM Expert* (Rigaku, 2012), *SHELXS97* and *SHELXL2014* (Sheldrick, 2008), *PLATON* (Spek, 2009) *Flipper 25* (Oszlányi & Sütő, 2004), *OSCAIL* (McArdle *et al.*, 2004), *ShelXle* (Hübschle *et al.*, 2011), *Mercury* (Macrae *et al.*, 2006) and *OSCAIL* (McArdle *et al.*, 2004).

react with the different haloanilines. Recrystallization from dichloromethane afforded colourless plates whose dimensions are given in Table 7.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 7. Amino H atoms were located in difference Fourier maps and were refined isotropically. All other H atoms were treated as riding atoms with C—H(aromatic) = 0.95 Å, U_{iso}= $1.2U$eq(C).

Compounds (1) and (2), reduced cell: [a = 6.6325 (12), b = 0.0577 (12), c = 14.671 (3) Å, α = 76.464 (7), β = 89.714 (6), γ = 74.411 (7)°, V = 641.9 (2) Å^3], have different reduced cells in which the x and z coordinates are comparable and the y coordinate of (2) is close to 1 − y of (1). For ease of comparison of the structures of (1) and (2), the refinement reported here was carried out for the non-reduced cell of (2) in which the α and γ angles were given the supplementary values of those of the reduced unit cell. The coordinates of (1)

were used as starting values and the transformation matrix for the reduced to non-reduced cell was $\bar{1}\,0\,0\,0\,1\,0\,0\,0\,\bar{1}$. This gave the same final refinement values as those for the refinement with the reduced cell. Compounds (1) and (2) are therefore isostructural.

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supporting information

The crystal structures of four *N*-(4-halophenyl)-4-oxo-4*H*-chromene-3-carboxamides

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Computing details

For all compounds, data collection: *CrystalClear-SM Expert* (Rigaku, 2012); cell refinement: *CrystalClear-SM Expert* (Rigaku, 2012); data reduction: *CrystalClear-SM Expert* (Rigaku, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008), *PLATON* (Spek, 2009) and Flipper 25 (Oszlányi & Sütő, 2004); program(s) used to refine structure: *OSCAIL* (McArdle *et al.*, 2004), ShelXle (Hübschle *et al.*, 2011) and *SHELXL2014* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *OSCAIL* (McArdle *et al.*, 2004), *SHELXL2014* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

(1) *N*-(4-Fluorophenyl)-4-oxo-4*H*-chromene-3-carboxamide

Crystal data

$C_{16}H_{10}FNO_3$	$Z = 2$
$M_r = 283.25$	$F(000) = 292$
Triclinic, $P\overline{1}$	$D_x = 1.529$ Mg m^{-3}
$a = 6.6213$ (5) Å	Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å
$b = 7.0517$ (5) Å	Cell parameters from 7765 reflections
$c = 14.0864$ (10) Å	$\theta = 3.0–27.5°$
$\alpha = 101.957$ (7)°	$\mu = 0.12$ mm^{-1}
$\beta = 90.047$ (6)°	$T = 100$ K
$\gamma = 106.657$ (7)°	Plate, colourless
$V = 615.17$ (8) Å^3	$0.46 \times 0.32 \times 0.02$ mm

Data collection

Rigaku Saturn724+ (2x2 bin mode) diffractometer	8176 measured reflections
Radiation source: Sealed Tube	2789 independent reflections
Graphite Monochromator monochromator	2393 reflections with $I > 2\sigma(I)$
Detector resolution: 28.5714 pixels mm^{-1}	$R_{int} = 0.056$
profile data from ω–scans	$\theta_{max} = 27.5°$, $\theta_{min} = 3.0°$
Absorption correction: multi-scan	$h = -8 \rightarrow 8$
(*CrystalClear-SM Expert*; Rigaku, 2012)	$k = -8 \rightarrow 9$
$T_{min} = 0.949$, $T_{max} = 0.998$	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	2789 reflections
Least-squares matrix: full	194 parameters
$R[F^2 > 2\sigma(F^2)] = 0.044$	0 restraints
$wR(F^2) = 0.135$	Hydrogen site location: mixed
$S = 1.06$	

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0807P)^2 + 0.0803P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
F314	0.11483 (12)	0.26388 (12)	0.11344 (5)	0.0304 (2)
O1	0.94909 (13)	1.09267 (13)	0.74817 (6)	0.0234 (2)
O3	0.81282 (13)	0.75748 (13)	0.47173 (6)	0.0253 (2)
O4	0.32603 (13)	0.88444 (13)	0.64744 (6)	0.0246 (2)
N3	0.45748 (16)	0.72194 (15)	0.47646 (7)	0.0209 (2)
H3	0.367 (3)	0.754 (2)	0.5197 (12)	0.041 (4)*
C2	0.88961 (19)	0.98534 (18)	0.65678 (8)	0.0217 (3)
H2	0.9978	0.9610	0.6161	0.026*
C3	0.68710 (18)	0.90890 (17)	0.61796 (8)	0.0202 (3)
C4	0.51484 (18)	0.94372 (17)	0.67726 (8)	0.0204 (3)
C4A	0.58107 (18)	1.05466 (18)	0.77820 (8)	0.0209 (3)
C5	0.43206 (19)	1.09101 (18)	0.84541 (9)	0.0240 (3)
H5	0.2858	1.0448	0.8256	0.029*
C6	0.4975 (2)	1.19373 (19)	0.94021 (9)	0.0274 (3)
H6	0.3964	1.2184	0.9852	0.033*
C7	0.7134 (2)	1.26148 (19)	0.96985 (9)	0.0275 (3)
H7	0.7575	1.3313	1.0351	0.033*
C8	0.8630 (2)	1.22769 (19)	0.90507 (9)	0.0256 (3)
H8	1.0091	1.2737	0.9250	0.031*
C8A	0.79373 (19)	1.12433 (18)	0.80975 (8)	0.0215 (3)
C311	0.38044 (18)	0.60437 (17)	0.38276 (8)	0.0206 (3)
C312	0.51109 (19)	0.55259 (18)	0.30996 (8)	0.0224 (3)
H312	0.6601	0.5953	0.3223	0.027*
C313	0.41972 (19)	0.43732 (18)	0.21894 (8)	0.0243 (3)
H313	0.5061	0.4006	0.1688	0.029*
C314	0.2034 (2)	0.37765 (18)	0.20270 (8)	0.0240 (3)
C315	0.07065 (19)	0.42664 (18)	0.27332 (9)	0.0237 (3)
H315	−0.0782	0.3830	0.2602	0.028*
C316	0.16053 (19)	0.54118 (18)	0.36382 (8)	0.0223 (3)
H316	0.0724	0.5770	0.4133	0.027*
C31	0.66033 (18)	0.78951 (17)	0.51512 (8)	0.0205 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F314	0.0282 (4)	0.0355 (4)	0.0231 (4)	0.0070 (3)	−0.0036 (3)	0.0000 (3)
O1	0.0145 (4)	0.0289 (5)	0.0249 (4)	0.0044 (3)	0.0012 (3)	0.0043 (3)
O3	0.0164 (4)	0.0321 (5)	0.0268 (4)	0.0067 (3)	0.0048 (3)	0.0058 (4)
O4	0.0135 (4)	0.0326 (5)	0.0258 (4)	0.0056 (3)	0.0014 (3)	0.0037 (3)
N3	0.0149 (5)	0.0252 (5)	0.0215 (5)	0.0048 (4)	0.0027 (4)	0.0041 (4)
C2	0.0180 (6)	0.0239 (6)	0.0230 (5)	0.0055 (5)	0.0031 (4)	0.0058 (4)
C3	0.0155 (6)	0.0222 (6)	0.0236 (6)	0.0048 (4)	0.0030 (4)	0.0076 (4)
C4	0.0161 (6)	0.0217 (6)	0.0241 (6)	0.0048 (4)	0.0027 (4)	0.0072 (4)
C4A	0.0181 (6)	0.0225 (6)	0.0223 (6)	0.0051 (5)	0.0016 (4)	0.0069 (4)
C5	0.0178 (6)	0.0285 (6)	0.0259 (6)	0.0061 (5)	0.0022 (4)	0.0071 (5)
C6	0.0247 (6)	0.0333 (7)	0.0242 (6)	0.0086 (5)	0.0051 (5)	0.0064 (5)
C7	0.0274 (7)	0.0307 (7)	0.0221 (6)	0.0062 (5)	−0.0004 (5)	0.0038 (5)
C8	0.0196 (6)	0.0284 (6)	0.0266 (6)	0.0040 (5)	−0.0021 (5)	0.0056 (5)
C8A	0.0178 (6)	0.0232 (6)	0.0242 (6)	0.0058 (5)	0.0034 (4)	0.0072 (5)
C311	0.0190 (6)	0.0205 (6)	0.0222 (5)	0.0045 (4)	0.0019 (4)	0.0065 (4)
C312	0.0171 (6)	0.0256 (6)	0.0242 (6)	0.0052 (5)	0.0027 (4)	0.0063 (5)
C313	0.0227 (6)	0.0273 (6)	0.0237 (6)	0.0082 (5)	0.0050 (5)	0.0062 (5)
C314	0.0252 (6)	0.0243 (6)	0.0211 (6)	0.0055 (5)	−0.0019 (5)	0.0047 (4)
C315	0.0176 (6)	0.0259 (6)	0.0270 (6)	0.0046 (5)	−0.0005 (4)	0.0073 (5)
C316	0.0177 (6)	0.0252 (6)	0.0255 (6)	0.0066 (5)	0.0043 (4)	0.0080 (5)
C31	0.0168 (6)	0.0214 (6)	0.0241 (6)	0.0047 (4)	0.0038 (4)	0.0078 (4)

Geometric parameters (Å, º)

F314—C314	1.3708 (13)	C6—C7	1.4042 (19)
O1—C2	1.3453 (14)	C6—H6	0.9500
O1—C8A	1.3833 (14)	C7—C8	1.3841 (17)
O3—C31	1.2323 (14)	C7—H7	0.9500
O4—C4	1.2413 (15)	C8—C8A	1.3951 (17)
N3—C31	1.3609 (15)	C8—H8	0.9500
N3—C311	1.4116 (15)	C311—C312	1.4003 (16)
N3—H3	0.896 (17)	C311—C316	1.4029 (17)
C2—C3	1.3601 (16)	C312—C313	1.3984 (16)
C2—H2	0.9500	C312—H312	0.9500
C3—C4	1.4616 (15)	C313—C314	1.3769 (18)
C3—C31	1.4992 (16)	C313—H313	0.9500
C4—C4A	1.4722 (16)	C314—C315	1.3835 (17)
C4A—C8A	1.3905 (17)	C315—C316	1.3886 (16)
C4A—C5	1.4076 (16)	C315—H315	0.9500
C5—C6	1.3835 (16)	C316—H316	0.9500
C5—H5	0.9500		
C2—O1—C8A	118.36 (9)	C7—C8—H8	120.8
C31—N3—C311	128.07 (10)	C8A—C8—H8	120.8
C31—N3—H3	112.0 (11)	O1—C8A—C4A	121.60 (10)

C311—N3—H3	119.8 (11)	O1—C8A—C8	116.11 (10)
O1—C2—C3	125.29 (10)	C4A—C8A—C8	122.28 (11)
O1—C2—H2	117.4	C312—C311—C316	119.96 (11)
C3—C2—H2	117.4	C312—C311—N3	123.53 (10)
C2—C3—C4	119.54 (11)	C316—C311—N3	116.51 (10)
C2—C3—C31	115.37 (10)	C313—C312—C311	119.27 (11)
C4—C3—C31	125.08 (10)	C313—C312—H312	120.4
O4—C4—C3	124.13 (11)	C311—C312—H312	120.4
O4—C4—C4A	121.21 (10)	C314—C313—C312	119.34 (11)
C3—C4—C4A	114.66 (10)	C314—C313—H313	120.3
C8A—C4A—C5	118.29 (11)	C312—C313—H313	120.3
C8A—C4A—C4	120.46 (10)	F314—C314—C313	119.02 (11)
C5—C4A—C4	121.24 (11)	F314—C314—C315	118.40 (11)
C6—C5—C4A	120.32 (11)	C313—C314—C315	122.58 (11)
C6—C5—H5	119.8	C314—C315—C316	118.32 (11)
C4A—C5—H5	119.8	C314—C315—H315	120.8
C5—C6—C7	119.97 (11)	C316—C315—H315	120.8
C5—C6—H6	120.0	C315—C316—C311	120.53 (10)
C7—C6—H6	120.0	C315—C316—H316	119.7
C8—C7—C6	120.77 (11)	C311—C316—H316	119.7
C8—C7—H7	119.6	O3—C31—N3	124.54 (11)
C6—C7—H7	119.6	O3—C31—C3	121.15 (10)
C7—C8—C8A	118.36 (11)	N3—C31—C3	114.31 (10)
C8A—O1—C2—C3	2.09 (17)	C4—C4A—C8A—C8	−179.14 (10)
O1—C2—C3—C4	0.56 (18)	C7—C8—C8A—O1	−179.38 (10)
O1—C2—C3—C31	−178.42 (10)	C7—C8—C8A—C4A	−0.09 (19)
C2—C3—C4—O4	178.11 (11)	C31—N3—C311—C312	−5.88 (19)
C31—C3—C4—O4	−3.02 (19)	C31—N3—C311—C316	175.04 (11)
C2—C3—C4—C4A	−2.70 (16)	C316—C311—C312—C313	−0.21 (18)
C31—C3—C4—C4A	176.17 (10)	N3—C311—C312—C313	−179.26 (10)
O4—C4—C4A—C8A	−178.41 (10)	C311—C312—C313—C314	0.19 (18)
C3—C4—C4A—C8A	2.36 (17)	C312—C313—C314—F314	−179.70 (10)
O4—C4—C4A—C5	2.34 (18)	C312—C313—C314—C315	−0.21 (19)
C3—C4—C4A—C5	−176.88 (10)	F314—C314—C315—C316	179.74 (9)
C8A—C4A—C5—C6	0.05 (18)	C313—C314—C315—C316	0.25 (19)
C4—C4A—C5—C6	179.31 (10)	C314—C315—C316—C311	−0.26 (18)
C4A—C5—C6—C7	−0.27 (19)	C312—C311—C316—C315	0.25 (18)
C5—C6—C7—C8	0.32 (19)	N3—C311—C316—C315	179.37 (10)
C6—C7—C8—C8A	−0.13 (19)	C311—N3—C31—O3	0.53 (19)
C2—O1—C8A—C4A	−2.39 (17)	C311—N3—C31—C3	−179.13 (10)
C2—O1—C8A—C8	176.90 (9)	C2—C3—C31—O3	3.00 (17)
C5—C4A—C8A—O1	179.39 (10)	C4—C3—C31—O3	−175.91 (10)
C4—C4A—C8A—O1	0.12 (18)	C2—C3—C31—N3	−177.33 (10)
C5—C4A—C8A—C8	0.13 (18)	C4—C3—C31—N3	3.76 (17)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O4	0.896 (17)	1.901 (17)	2.7024 (13)	147.9 (15)
C312—H312···O3	0.95	2.26	2.8714 (15)	122
C2—H2···O4^i	0.95	2.45	3.1645 (14)	132
C316—H316···O3^{ii}	0.95	2.46	3.3160 (14)	149

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

(2) *N*-(4-Chlorophenyl)-4-oxo-4*H*-chromene-3-carboxamide

Crystal data

$C_{16}H_{10}ClNO_3$	$Z = 2$
$M_r = 299.70$	$F(000) = 308$
Triclinic, $P\bar{1}$	$D_x = 1.551$ Mg m^{-3}
$a = 6.6325$ (12) Å	Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å
$b = 7.0577$ (12) Å	Cell parameters from 5302 reflections
$c = 14.671$ (3) Å	$\theta = 3.1–25.1°$
$\alpha = 103.536$ (7)°	$\mu = 0.31$ mm^{-1}
$\beta = 89.714$ (6)°	$T = 100$ K
$\gamma = 105.589$ (7)°	Plate, colourless
$V = 641.9$ (2) Å^3	$0.17 \times 0.17 \times 0.04$ mm

Data collection

Rigaku AFC12 (Right) diffractometer	7435 measured reflections
Radiation source: Rotating Anode	2265 independent reflections
Detector resolution: 28.5714 pixels mm^{-1}	1668 reflections with $I > 2\sigma(I)$
profile data from ω–scans	$R_{int} = 0.078$
Absorption correction: multi-scan	$\theta_{max} = 25.1°$, $\theta_{min} = 3.1°$
(*CrystalClear-SM Expert*; Rigaku, 20112)	$h = -7 \rightarrow 7$
	$k = -8 \rightarrow 8$
$T_{min} = 0.950$, $T_{max} = 0.988$	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent
$R[F^2 > 2\sigma(F^2)] = 0.056$	and constrained refinement
$wR(F^2) = 0.145$	$w = 1/[\sigma^2(F_o^2) + (0.0834P)^2]$
$S = 0.99$	where $P = (F_o^2 + 2F_c^2)/3$
2265 reflections	$(\Delta/\sigma)_{max} < 0.001$
194 parameters	$\Delta\rho_{max} = 0.30$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.65$ e Å^{-3}

Special details

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

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

	x	y	z	$U_{iso}*/U_{eq}$
Cl14	0.07614 (10)	0.21374 (9)	0.10359 (4)	0.0313 (3)

O1	0.9406 (3)	1.1068 (3)	0.73477 (11)	0.0274 (5)
O3	0.8086 (3)	0.7526 (3)	0.46866 (11)	0.0288 (5)
O4	0.3241 (3)	0.8897 (3)	0.63834 (11)	0.0289 (5)
N3	0.4567 (4)	0.7165 (3)	0.47522 (15)	0.0244 (5)
H3	0.370 (5)	0.751 (4)	0.514 (2)	0.034 (8)*
C2	0.8826 (4)	0.9921 (4)	0.64698 (16)	0.0256 (6)
H2	0.9908	0.9643	0.6078	0.031*
C3	0.6843 (4)	0.9137 (4)	0.61023 (16)	0.0240 (6)
C4	0.5115 (4)	0.9506 (4)	0.66692 (16)	0.0240 (6)
C4A	0.5757 (4)	1.0672 (4)	0.76429 (16)	0.0244 (6)
C5	0.4267 (4)	1.1045 (4)	0.82901 (17)	0.0292 (6)
H5	0.2816	1.0564	0.8099	0.035*
C6	0.4897 (5)	1.2107 (4)	0.92032 (17)	0.0325 (7)
H6	0.3882	1.2337	0.9644	0.039*
C7	0.7028 (5)	1.2844 (4)	0.94793 (17)	0.0322 (7)
H7	0.7453	1.3590	1.0108	0.039*
C8	0.8523 (4)	1.2509 (4)	0.88568 (17)	0.0291 (6)
H8	0.9973	1.3011	0.9047	0.035*
C8A	0.7856 (4)	1.1414 (4)	0.79395 (17)	0.0264 (6)
C311	0.3776 (4)	0.5942 (4)	0.38578 (16)	0.0233 (6)
C312	0.5027 (4)	0.5348 (4)	0.31405 (17)	0.0268 (6)
H312	0.6510	0.5748	0.3243	0.032*
C313	0.4084 (4)	0.4162 (4)	0.22722 (17)	0.0272 (6)
H313	0.4924	0.3751	0.1779	0.033*
C314	0.1937 (4)	0.3590 (4)	0.21297 (16)	0.0253 (6)
C315	0.0677 (4)	0.4157 (4)	0.28384 (16)	0.0265 (6)
H315	−0.0806	0.3742	0.2733	0.032*
C316	0.1603 (4)	0.5332 (4)	0.36990 (16)	0.0259 (6)
H316	0.0749	0.5729	0.4189	0.031*
C31	0.6576 (4)	0.7865 (4)	0.51110 (17)	0.0238 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl14	0.0362 (5)	0.0322 (4)	0.0226 (4)	0.0117 (3)	0.0014 (3)	−0.0019 (3)
O1	0.0258 (10)	0.0280 (9)	0.0267 (9)	0.0103 (8)	0.0035 (7)	−0.0001 (7)
O3	0.0242 (10)	0.0335 (10)	0.0280 (9)	0.0129 (8)	0.0076 (8)	0.0008 (8)
O4	0.0223 (11)	0.0347 (10)	0.0272 (9)	0.0109 (9)	0.0035 (7)	−0.0007 (7)
N3	0.0225 (12)	0.0270 (12)	0.0220 (11)	0.0107 (10)	0.0052 (9)	−0.0018 (9)
C2	0.0285 (15)	0.0254 (13)	0.0242 (12)	0.0128 (12)	0.0058 (10)	0.0024 (10)
C3	0.0257 (15)	0.0231 (13)	0.0247 (13)	0.0112 (11)	0.0045 (10)	0.0032 (10)
C4	0.0257 (15)	0.0203 (13)	0.0269 (13)	0.0093 (12)	0.0053 (11)	0.0040 (10)
C4A	0.0286 (15)	0.0229 (13)	0.0243 (13)	0.0117 (12)	0.0054 (11)	0.0052 (10)
C5	0.0282 (15)	0.0330 (15)	0.0272 (13)	0.0134 (12)	0.0059 (11)	0.0034 (11)
C6	0.0379 (17)	0.0362 (15)	0.0260 (13)	0.0182 (13)	0.0097 (12)	0.0035 (11)
C7	0.0405 (17)	0.0313 (15)	0.0247 (13)	0.0149 (13)	0.0020 (12)	0.0006 (11)
C8	0.0294 (15)	0.0263 (14)	0.0277 (13)	0.0075 (12)	−0.0029 (11)	−0.0010 (10)
C8A	0.0277 (15)	0.0247 (13)	0.0294 (13)	0.0135 (12)	0.0069 (11)	0.0046 (11)

C311	0.0288 (15)	0.0199 (12)	0.0217 (12)	0.0105 (11)	0.0039 (10)	0.0019 (10)
C312	0.0262 (15)	0.0282 (14)	0.0269 (13)	0.0116 (12)	0.0066 (11)	0.0036 (10)
C313	0.0353 (16)	0.0245 (13)	0.0249 (13)	0.0161 (12)	0.0097 (11)	0.0027 (10)
C314	0.0305 (15)	0.0228 (13)	0.0222 (12)	0.0097 (12)	0.0042 (11)	0.0021 (10)
C315	0.0275 (15)	0.0270 (13)	0.0259 (13)	0.0114 (12)	0.0026 (11)	0.0036 (10)
C316	0.0307 (15)	0.0257 (13)	0.0229 (13)	0.0144 (12)	0.0068 (11)	0.0016 (10)
C31	0.0229 (15)	0.0226 (13)	0.0287 (13)	0.0105 (11)	0.0067 (11)	0.0067 (10)

Geometric parameters (Å, º)

Cl14—C314	1.745 (2)	C6—C7	1.394 (4)
O1—C2	1.346 (3)	C6—H6	0.9500
O1—C8A	1.377 (3)	C7—C8	1.374 (4)
O3—C31	1.225 (3)	C7—H7	0.9500
O4—C4	1.241 (3)	C8—C8A	1.392 (3)
N3—C31	1.352 (3)	C8—H8	0.9500
N3—C311	1.405 (3)	C311—C316	1.393 (4)
N3—H3	0.85 (3)	C311—C312	1.394 (3)
C2—C3	1.343 (4)	C312—C313	1.393 (3)
C2—H2	0.9500	C312—H312	0.9500
C3—C4	1.457 (4)	C313—C314	1.375 (4)
C3—C31	1.503 (3)	C313—H313	0.9500
C4—C4A	1.472 (3)	C314—C315	1.384 (4)
C4A—C8A	1.382 (4)	C315—C316	1.380 (3)
C4A—C5	1.400 (4)	C315—H315	0.9500
C5—C6	1.378 (3)	C316—H316	0.9500
C5—H5	0.9500		
C2—O1—C8A	118.2 (2)	C7—C8—H8	120.9
C31—N3—C311	128.7 (2)	C8A—C8—H8	120.9
C31—N3—H3	113.0 (19)	O1—C8A—C4A	121.7 (2)
C311—N3—H3	118 (2)	O1—C8A—C8	116.3 (2)
C3—C2—O1	125.4 (2)	C4A—C8A—C8	122.0 (2)
C3—C2—H2	117.3	C316—C311—C312	119.6 (2)
O1—C2—H2	117.3	C316—C311—N3	116.3 (2)
C2—C3—C4	119.9 (2)	C312—C311—N3	124.1 (2)
C2—C3—C31	115.8 (2)	C313—C312—C311	119.5 (2)
C4—C3—C31	124.3 (2)	C313—C312—H312	120.3
O4—C4—C3	124.6 (2)	C311—C312—H312	120.3
O4—C4—C4A	121.1 (2)	C314—C313—C312	119.9 (2)
C3—C4—C4A	114.4 (2)	C314—C313—H313	120.0
C8A—C4A—C5	118.5 (2)	C312—C313—H313	120.0
C8A—C4A—C4	120.4 (2)	C313—C314—C315	121.2 (2)
C5—C4A—C4	121.1 (2)	C313—C314—Cl14	119.82 (18)
C6—C5—C4A	120.3 (3)	C315—C314—Cl14	119.0 (2)
C6—C5—H5	119.9	C316—C315—C314	119.1 (2)
C4A—C5—H5	119.9	C316—C315—H315	120.4
C5—C6—C7	119.9 (2)	C314—C315—H315	120.4

C5—C6—H6	120.1	C315—C316—C311	120.7 (2)
C7—C6—H6	120.1	C315—C316—H316	119.6
C8—C7—C6	121.1 (2)	C311—C316—H316	119.6
C8—C7—H7	119.5	O3—C31—N3	124.6 (2)
C6—C7—H7	119.5	O3—C31—C3	121.2 (2)
C7—C8—C8A	118.3 (2)	N3—C31—C3	114.1 (2)
C8A—O1—C2—C3	2.8 (4)	C4—C4A—C8A—C8	−179.5 (2)
O1—C2—C3—C4	−0.1 (4)	C7—C8—C8A—O1	−178.3 (2)
O1—C2—C3—C31	−178.7 (2)	C7—C8—C8A—C4A	0.3 (4)
C2—C3—C4—O4	177.8 (3)	C31—N3—C311—C316	175.7 (2)
C31—C3—C4—O4	−3.8 (4)	C31—N3—C311—C312	−5.0 (4)
C2—C3—C4—C4A	−2.9 (3)	C316—C311—C312—C313	0.4 (4)
C31—C3—C4—C4A	175.6 (2)	N3—C311—C312—C313	−178.8 (2)
O4—C4—C4A—C8A	−177.2 (2)	C311—C312—C313—C314	0.0 (4)
C3—C4—C4A—C8A	3.4 (3)	C312—C313—C314—C315	−0.5 (4)
O4—C4—C4A—C5	3.2 (4)	C312—C313—C314—Cl14	179.03 (19)
C3—C4—C4A—C5	−176.1 (2)	C313—C314—C315—C316	0.6 (4)
C8A—C4A—C5—C6	−0.8 (4)	Cl14—C314—C315—C316	−178.97 (18)
C4—C4A—C5—C6	178.8 (2)	C314—C315—C316—C311	−0.1 (4)
C4A—C5—C6—C7	1.1 (4)	C312—C311—C316—C315	−0.4 (4)
C5—C6—C7—C8	−0.8 (4)	N3—C311—C316—C315	178.9 (2)
C6—C7—C8—C8A	0.1 (4)	C311—N3—C31—O3	0.4 (4)
C2—O1—C8A—C4A	−2.2 (3)	C311—N3—C31—C3	−179.8 (2)
C2—O1—C8A—C8	176.4 (2)	C2—C3—C31—O3	2.3 (4)
C5—C4A—C8A—O1	178.6 (2)	C4—C3—C31—O3	−176.3 (2)
C4—C4A—C8A—O1	−1.0 (4)	C2—C3—C31—N3	−177.6 (2)
C5—C4A—C8A—C8	0.1 (4)	C4—C3—C31—N3	3.9 (4)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O4	0.85 (3)	1.92 (3)	2.680 (3)	148 (3)
C312—H312···O3	0.95	2.29	2.892 (3)	121
C2—H2···O4[i]	0.95	2.47	3.194 (3)	133
C316—H316···O3[ii]	0.95	2.45	3.286 (3)	146

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

(3) *N*-(4-Bromophenyl)-4-oxo-4*H*-chromene-3-carboxamide

Crystal data

$C_{16}H_{10}BrNO_3$	$\gamma = 100.884\ (6)°$
$M_r = 344.16$	$V = 661.24\ (9)$ Å³
Triclinic, $P\overline{1}$	$Z = 2$
$a = 6.6505\ (5)$ Å	$F(000) = 344$
$b = 9.3580\ (7)$ Å	$D_x = 1.729$ Mg m⁻³
$c = 11.0060\ (8)$ Å	Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å
$\alpha = 100.280\ (6)°$	Cell parameters from 8172 reflections
$\beta = 90.461\ (6)°$	$\theta = 1.9–27.5°$

$\mu = 3.12$ mm^{-1}	Plate, colourless	
$T = 120$ K	$0.58 \times 0.18 \times 0.06$ mm	

Data collection

Rigaku RAXIS conversion diffractometer	9930 measured reflections
Radiation source: Sealed Tube	3017 independent reflections
Graphite Monochromator monochromator	2525 reflections with $I > 2\sigma(I)$
Detector resolution: 10.0000 pixels mm^{-1}	$R_{int} = 0.045$
profile data from ω–scans	$\theta_{max} = 27.5°$, $\theta_{min} = 2.3°$
Absorption correction: multi-scan	$h = -8 \rightarrow 8$
(*CrystalClear-SM Expert*; Rigaku, 20112)	$k = -12 \rightarrow 12$
$T_{min} = 0.265$, $T_{max} = 0.835$	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent
$R[F^2 > 2\sigma(F^2)] = 0.027$	and constrained refinement
$wR(F^2) = 0.058$	$w = 1/[\sigma^2(F_o^2) + (0.0254P)^2]$
$S = 0.94$	where $P = (F_o^2 + 2F_c^2)/3$
3017 reflections	$(\Delta/\sigma)_{max} < 0.001$
194 parameters	$\Delta\rho_{max} = 0.53$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.69$ e Å^{-3}

Special details

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

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

	x	y	z	$U_{iso}*/U_{eq}$
Br14	0.10149 (3)	0.28307 (2)	0.96928 (2)	0.02704 (8)
O1	0.88599 (18)	0.94245 (15)	0.35000 (13)	0.0205 (3)
O3	0.78338 (19)	0.68329 (16)	0.60891 (13)	0.0232 (3)
O4	0.28839 (18)	0.79112 (15)	0.42189 (13)	0.0217 (3)
N3	0.4338 (2)	0.64948 (18)	0.58599 (15)	0.0164 (3)
H3	0.341 (4)	0.674 (3)	0.545 (2)	0.032 (6)*
C2	0.8359 (3)	0.8522 (2)	0.43099 (18)	0.0179 (4)
H2	0.9450	0.8226	0.4701	0.021*
C3	0.6440 (3)	0.7994 (2)	0.46206 (17)	0.0157 (4)
C4	0.4703 (3)	0.8354 (2)	0.40029 (17)	0.0158 (4)
C4A	0.5260 (3)	0.9313 (2)	0.30911 (17)	0.0166 (4)
C5	0.3763 (3)	0.9747 (2)	0.24089 (18)	0.0194 (4)
H5	0.2352	0.9394	0.2516	0.023*
C6	0.4330 (3)	1.0678 (2)	0.15872 (18)	0.0231 (4)
H6	0.3307	1.0956	0.1120	0.028*
C7	0.6404 (3)	1.1223 (2)	0.14319 (19)	0.0231 (4)
H7	0.6778	1.1880	0.0870	0.028*
C8	0.7904 (3)	1.0813 (2)	0.20869 (19)	0.0221 (4)

H8	0.9313	1.1184	0.1989	0.027*
C8A	0.7304 (3)	0.9842 (2)	0.28949 (17)	0.0168 (4)
C311	0.3678 (3)	0.5624 (2)	0.67490 (17)	0.0156 (4)
C312	0.5008 (3)	0.5181 (2)	0.75335 (18)	0.0181 (4)
H312	0.6449	0.5456	0.7476	0.022*
C313	0.4212 (3)	0.4336 (2)	0.83976 (17)	0.0201 (4)
H313	0.5107	0.4026	0.8930	0.024*
C314	0.2110 (3)	0.3949 (2)	0.84817 (17)	0.0189 (4)
C315	0.0774 (3)	0.4369 (2)	0.77019 (18)	0.0197 (4)
H315	−0.0666	0.4085	0.7761	0.024*
C316	0.1558 (3)	0.5204 (2)	0.68385 (18)	0.0183 (4)
H316	0.0652	0.5497	0.6301	0.022*
C31	0.6284 (3)	0.7053 (2)	0.55924 (17)	0.0168 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br14	0.02998 (12)	0.03136 (13)	0.02276 (11)	0.00465 (9)	0.00436 (8)	0.01411 (9)
O1	0.0127 (6)	0.0238 (8)	0.0263 (7)	0.0026 (5)	0.0014 (5)	0.0094 (6)
O3	0.0147 (6)	0.0297 (8)	0.0280 (8)	0.0058 (6)	−0.0021 (6)	0.0113 (6)
O4	0.0123 (6)	0.0293 (8)	0.0266 (8)	0.0041 (6)	0.0009 (5)	0.0133 (6)
N3	0.0128 (7)	0.0191 (9)	0.0190 (8)	0.0038 (6)	−0.0008 (6)	0.0073 (7)
C2	0.0156 (9)	0.0185 (10)	0.0202 (10)	0.0045 (7)	−0.0007 (7)	0.0040 (8)
C3	0.0145 (9)	0.0150 (9)	0.0173 (9)	0.0039 (7)	−0.0009 (7)	0.0013 (8)
C4	0.0144 (8)	0.0159 (10)	0.0171 (9)	0.0042 (7)	−0.0003 (7)	0.0015 (8)
C4A	0.0177 (9)	0.0171 (10)	0.0151 (9)	0.0035 (7)	0.0002 (7)	0.0026 (8)
C5	0.0171 (9)	0.0228 (11)	0.0192 (9)	0.0058 (8)	0.0010 (8)	0.0038 (8)
C6	0.0260 (10)	0.0266 (11)	0.0198 (10)	0.0094 (9)	0.0010 (8)	0.0080 (9)
C7	0.0303 (10)	0.0206 (11)	0.0199 (10)	0.0055 (9)	0.0054 (8)	0.0071 (8)
C8	0.0204 (9)	0.0211 (11)	0.0247 (10)	0.0020 (8)	0.0062 (8)	0.0054 (9)
C8A	0.0158 (9)	0.0167 (10)	0.0181 (9)	0.0045 (7)	0.0009 (7)	0.0025 (8)
C311	0.0168 (9)	0.0142 (9)	0.0162 (9)	0.0042 (7)	0.0006 (7)	0.0024 (8)
C312	0.0169 (9)	0.0184 (10)	0.0190 (9)	0.0034 (7)	−0.0015 (7)	0.0033 (8)
C313	0.0222 (10)	0.0225 (11)	0.0168 (9)	0.0077 (8)	−0.0027 (8)	0.0038 (8)
C314	0.0253 (10)	0.0174 (10)	0.0143 (9)	0.0039 (8)	0.0027 (7)	0.0039 (8)
C315	0.0169 (9)	0.0207 (10)	0.0215 (10)	0.0030 (8)	0.0022 (8)	0.0040 (8)
C316	0.0164 (9)	0.0206 (10)	0.0196 (10)	0.0060 (8)	−0.0013 (7)	0.0053 (8)
C31	0.0154 (9)	0.0170 (10)	0.0181 (9)	0.0048 (7)	−0.0002 (7)	0.0020 (8)

Geometric parameters (Å, º)

Br14—C314	1.9045 (19)	C6—C7	1.401 (3)
O1—C2	1.337 (2)	C6—H6	0.9500
O1—C8A	1.379 (2)	C7—C8	1.376 (3)
O3—C31	1.231 (2)	C7—H7	0.9500
O4—C4	1.242 (2)	C8—C8A	1.390 (3)
N3—C31	1.356 (2)	C8—H8	0.9500
N3—C311	1.404 (2)	C311—C312	1.397 (2)

N3—H3	0.86 (2)	C311—C316	1.400 (2)
C2—C3	1.348 (3)	C312—C313	1.388 (3)
C2—H2	0.9500	C312—H312	0.9500
C3—C4	1.458 (2)	C313—C314	1.384 (3)
C3—C31	1.495 (3)	C313—H313	0.9500
C4—C4A	1.467 (3)	C314—C315	1.386 (3)
C4A—C8A	1.388 (3)	C315—C316	1.380 (3)
C4A—C5	1.403 (2)	C315—H315	0.9500
C5—C6	1.372 (3)	C316—H316	0.9500
C5—H5	0.9500		
C2—O1—C8A	118.38 (14)	C7—C8—H8	120.9
C31—N3—C311	128.47 (15)	C8A—C8—H8	120.9
C31—N3—H3	114.6 (16)	O1—C8A—C4A	121.24 (16)
C311—N3—H3	116.9 (16)	O1—C8A—C8	116.22 (16)
O1—C2—C3	125.75 (16)	C4A—C8A—C8	122.54 (17)
O1—C2—H2	117.1	C312—C311—C316	119.63 (17)
C3—C2—H2	117.1	C312—C311—N3	123.76 (16)
C2—C3—C4	119.38 (17)	C316—C311—N3	116.61 (15)
C2—C3—C31	115.58 (15)	C313—C312—C311	119.65 (17)
C4—C3—C31	125.04 (16)	C313—C312—H312	120.2
O4—C4—C3	124.05 (17)	C311—C312—H312	120.2
O4—C4—C4A	121.35 (16)	C314—C313—C312	119.81 (17)
C3—C4—C4A	114.59 (16)	C314—C313—H313	120.1
C8A—C4A—C5	117.93 (17)	C312—C313—H313	120.1
C8A—C4A—C4	120.55 (16)	C313—C314—C315	121.13 (18)
C5—C4A—C4	121.51 (17)	C313—C314—Br14	119.89 (14)
C6—C5—C4A	120.25 (18)	C315—C314—Br14	118.97 (15)
C6—C5—H5	119.9	C316—C315—C314	119.26 (17)
C4A—C5—H5	119.9	C316—C315—H315	120.4
C5—C6—C7	120.48 (18)	C314—C315—H315	120.4
C5—C6—H6	119.8	C315—C316—C311	120.50 (16)
C7—C6—H6	119.8	C315—C316—H316	119.7
C8—C7—C6	120.48 (19)	C311—C316—H316	119.7
C8—C7—H7	119.8	O3—C31—N3	124.65 (18)
C6—C7—H7	119.8	O3—C31—C3	120.84 (17)
C7—C8—C8A	118.28 (18)	N3—C31—C3	114.51 (15)
C8A—O1—C2—C3	−1.6 (3)	C4—C4A—C8A—C8	−176.67 (18)
O1—C2—C3—C4	3.0 (3)	C7—C8—C8A—O1	177.90 (17)
O1—C2—C3—C31	−177.47 (17)	C7—C8—C8A—C4A	−2.1 (3)
C2—C3—C4—O4	179.41 (19)	C31—N3—C311—C312	0.1 (3)
C31—C3—C4—O4	0.0 (3)	C31—N3—C311—C316	179.50 (18)
C2—C3—C4—C4A	−1.2 (3)	C316—C311—C312—C313	−0.3 (3)
C31—C3—C4—C4A	179.38 (17)	N3—C311—C312—C313	179.06 (17)
O4—C4—C4A—C8A	177.58 (18)	C311—C312—C313—C314	−0.5 (3)
C3—C4—C4A—C8A	−1.9 (3)	C312—C313—C314—C315	1.1 (3)
O4—C4—C4A—C5	−1.2 (3)	C312—C313—C314—Br14	−178.50 (15)

C3—C4—C4A—C5	179.33 (17)	C313—C314—C315—C316	−0.8 (3)
C8A—C4A—C5—C6	−0.7 (3)	Br14—C314—C315—C316	178.72 (15)
C4—C4A—C5—C6	178.16 (18)	C314—C315—C316—C311	0.0 (3)
C4A—C5—C6—C7	−0.9 (3)	C312—C311—C316—C315	0.6 (3)
C5—C6—C7—C8	1.0 (3)	N3—C311—C316—C315	−178.89 (17)
C6—C7—C8—C8A	0.4 (3)	C311—N3—C31—O3	2.3 (3)
C2—O1—C8A—C4A	−1.7 (3)	C311—N3—C31—C3	−177.52 (17)
C2—O1—C8A—C8	178.33 (17)	C2—C3—C31—O3	2.3 (3)
C5—C4A—C8A—O1	−177.77 (17)	C4—C3—C31—O3	−178.23 (18)
C4—C4A—C8A—O1	3.4 (3)	C2—C3—C31—N3	−177.91 (17)
C5—C4A—C8A—C8	2.2 (3)	C4—C3—C31—N3	1.6 (3)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O4	0.86 (2)	1.95 (2)	2.695 (2)	145 (2)
C312—H312···O3	0.95	2.26	2.877 (2)	129
C2—H2···O4[i]	0.95	2.41	3.167 (2)	137
C316—H316···O3[ii]	0.95	2.47	3.314 (2)	148

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

(4) *N*-(4-Iodophenyl)-4-oxo-4*H*-chromene-3-carboxamide

Crystal data

$C_{16}H_{10}INO_3$	$Z = 2$
$M_r = 391.15$	$F(000) = 380$
Triclinic, $P\overline{1}$	$D_x = 1.900$ Mg m^{-3}
$a = 6.6750$ (5) Å	Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å
$b = 9.4166$ (7) Å	Cell parameters from 9236 reflections
$c = 11.2673$ (8) Å	$\theta = 1.8–27.5°$
$\alpha = 100.974$ (6)°	$\mu = 2.35$ mm^{-1}
$\beta = 90.769$ (6)°	$T = 120$ K
$\gamma = 100.062$ (6)°	Plate, colourless
$V = 683.77$ (9) Å^3	0.46 × 0.22 × 0.05 mm

Data collection

Rigaku RAXIS conversion diffractometer	10032 measured reflections
	3095 independent reflections
Radiation source: Sealed Tube	2819 reflections with $I > 2\sigma(I)$
Graphite Monochromator monochromator	$R_{int} = 0.026$
Detector resolution: 10.0000 pixels mm^{-1}	$\theta_{max} = 27.5°$, $\theta_{min} = 2.2°$
profile data from ω–scans	$h = −7→8$
Absorption correction: multi-scan	$k = −12→11$
(*CrystalClear-SM Expert*; Rigaku, 20112)	$l = −14→14$
$T_{min} = 0.411$, $T_{max} = 0.892$	

Refinement

Refinement on F^2	3095 reflections
Least-squares matrix: full	194 parameters
$R[F^2 > 2\sigma(F^2)] = 0.018$	0 restraints
$wR(F^2) = 0.044$	Hydrogen site location: mixed
$S = 1.03$	

H atoms treated by a mixture of independent
  and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.026P)^2]$
  where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.67$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
I314	0.09431 (2)	0.28556 (2)	0.97093 (2)	0.02278 (5)
O1	0.88205 (19)	0.94124 (14)	0.35573 (11)	0.0188 (3)
O3	0.7838 (2)	0.68549 (15)	0.60794 (12)	0.0219 (3)
O4	0.28854 (19)	0.79085 (14)	0.42255 (11)	0.0199 (3)
N3	0.4362 (2)	0.65097 (16)	0.58280 (13)	0.0152 (3)
H3	0.337 (3)	0.678 (2)	0.5384 (19)	0.017 (5)*
C2	0.8340 (3)	0.85191 (19)	0.43445 (15)	0.0166 (3)
H2	0.9434	0.8230	0.4731	0.020*
C3	0.6432 (3)	0.79929 (17)	0.46363 (14)	0.0144 (3)
C4	0.4692 (3)	0.83443 (18)	0.40253 (14)	0.0144 (3)
C4A	0.5235 (3)	0.92875 (18)	0.31366 (14)	0.0149 (3)
C5	0.3733 (3)	0.9701 (2)	0.24576 (16)	0.0193 (4)
H5	0.2334	0.9352	0.2557	0.023*
C6	0.4280 (3)	1.0611 (2)	0.16480 (16)	0.0221 (4)
H6	0.3257	1.0865	0.1176	0.026*
C7	0.6338 (3)	1.1164 (2)	0.15168 (16)	0.0217 (4)
H7	0.6696	1.1811	0.0971	0.026*
C8	0.7853 (3)	1.0777 (2)	0.21751 (16)	0.0211 (4)
H8	0.9249	1.1154	0.2094	0.025*
C8A	0.7265 (3)	0.98182 (19)	0.29605 (15)	0.0167 (3)
C311	0.3707 (3)	0.56515 (18)	0.66915 (14)	0.0147 (3)
C312	0.5034 (3)	0.52301 (19)	0.74728 (15)	0.0170 (3)
H312	0.6466	0.5501	0.7426	0.020*
C313	0.4242 (3)	0.4409 (2)	0.83219 (15)	0.0183 (3)
H313	0.5134	0.4119	0.8857	0.022*
C314	0.2148 (3)	0.40150 (18)	0.83828 (15)	0.0167 (3)
C315	0.0823 (3)	0.44141 (19)	0.75977 (15)	0.0178 (3)
H315	−0.0608	0.4127	0.7637	0.021*
C316	0.1603 (3)	0.52325 (19)	0.67578 (15)	0.0171 (3)
H316	0.0702	0.5512	0.6221	0.020*
C31	0.6294 (3)	0.70661 (18)	0.55846 (15)	0.0154 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I314	0.02679 (8)	0.02415 (7)	0.01880 (6)	0.00303 (5)	0.00371 (4)	0.00890 (4)
O1	0.0120 (6)	0.0209 (6)	0.0242 (6)	0.0014 (5)	0.0022 (5)	0.0074 (5)
O3	0.0143 (6)	0.0284 (7)	0.0258 (7)	0.0053 (5)	−0.0013 (5)	0.0108 (5)
O4	0.0116 (6)	0.0256 (7)	0.0248 (6)	0.0024 (5)	0.0016 (5)	0.0116 (5)
N3	0.0136 (7)	0.0169 (7)	0.0164 (7)	0.0040 (6)	−0.0002 (5)	0.0057 (5)
C2	0.0139 (8)	0.0165 (8)	0.0192 (8)	0.0035 (6)	−0.0004 (6)	0.0027 (6)
C3	0.0138 (8)	0.0129 (8)	0.0158 (7)	0.0027 (6)	0.0005 (6)	0.0005 (6)
C4	0.0132 (8)	0.0146 (8)	0.0151 (7)	0.0027 (6)	0.0006 (6)	0.0018 (6)
C4A	0.0157 (9)	0.0141 (8)	0.0146 (7)	0.0031 (6)	0.0021 (6)	0.0017 (6)
C5	0.0169 (9)	0.0217 (9)	0.0195 (8)	0.0036 (7)	0.0019 (6)	0.0045 (6)
C6	0.0260 (10)	0.0246 (9)	0.0186 (8)	0.0088 (8)	0.0007 (7)	0.0078 (7)
C7	0.0283 (10)	0.0197 (9)	0.0198 (8)	0.0066 (7)	0.0080 (7)	0.0078 (6)
C8	0.0202 (10)	0.0193 (9)	0.0236 (9)	0.0024 (7)	0.0066 (7)	0.0047 (7)
C8A	0.0161 (9)	0.0155 (8)	0.0181 (8)	0.0035 (6)	0.0026 (6)	0.0016 (6)
C311	0.0166 (9)	0.0127 (8)	0.0145 (7)	0.0034 (6)	0.0011 (6)	0.0014 (6)
C312	0.0147 (9)	0.0183 (8)	0.0186 (8)	0.0041 (7)	−0.0002 (6)	0.0038 (6)
C313	0.0199 (9)	0.0198 (8)	0.0164 (8)	0.0065 (7)	−0.0023 (6)	0.0038 (6)
C314	0.0203 (9)	0.0148 (8)	0.0149 (7)	0.0018 (7)	0.0026 (6)	0.0039 (6)
C315	0.0148 (9)	0.0199 (9)	0.0183 (8)	0.0030 (7)	0.0018 (6)	0.0029 (6)
C316	0.0164 (9)	0.0171 (8)	0.0184 (8)	0.0055 (7)	−0.0014 (6)	0.0027 (6)
C31	0.0161 (9)	0.0140 (8)	0.0157 (7)	0.0036 (6)	0.0007 (6)	0.0010 (6)

Geometric parameters (Å, º)

I314—C314	2.1023 (17)	C6—C7	1.401 (3)
O1—C2	1.340 (2)	C6—H6	0.9500
O1—C8A	1.376 (2)	C7—C8	1.385 (3)
O3—C31	1.229 (2)	C7—H7	0.9500
O4—C4	1.243 (2)	C8—C8A	1.394 (2)
N3—C31	1.357 (2)	C8—H8	0.9500
N3—C311	1.406 (2)	C311—C312	1.397 (2)
N3—H3	0.92 (2)	C311—C316	1.398 (3)
C2—C3	1.352 (2)	C312—C313	1.395 (3)
C2—H2	0.9500	C312—H312	0.9500
C3—C4	1.460 (2)	C313—C314	1.388 (3)
C3—C31	1.497 (2)	C313—H313	0.9500
C4—C4A	1.469 (2)	C314—C315	1.389 (2)
C4A—C8A	1.390 (2)	C315—C316	1.383 (2)
C4A—C5	1.404 (2)	C315—H315	0.9500
C5—C6	1.377 (3)	C316—H316	0.9500
C5—H5	0.9500		
C2—O1—C8A	118.48 (14)	C7—C8—H8	121.0
C31—N3—C311	128.55 (15)	C8A—C8—H8	121.0
C31—N3—H3	114.0 (15)	O1—C8A—C4A	121.30 (15)

C311—N3—H3	117.4 (15)	O1—C8A—C8	116.04 (16)
O1—C2—C3	125.61 (16)	C4A—C8A—C8	122.66 (17)
O1—C2—H2	117.2	C312—C311—C316	119.80 (16)
C3—C2—H2	117.2	C312—C311—N3	123.60 (16)
C2—C3—C4	119.41 (15)	C316—C311—N3	116.59 (15)
C2—C3—C31	115.53 (15)	C313—C312—C311	119.53 (17)
C4—C3—C31	125.07 (15)	C313—C312—H312	120.2
O4—C4—C3	124.22 (16)	C311—C312—H312	120.2
O4—C4—C4A	121.28 (16)	C314—C313—C312	119.87 (16)
C3—C4—C4A	114.49 (15)	C314—C313—H313	120.1
C8A—C4A—C5	118.05 (16)	C312—C313—H313	120.1
C8A—C4A—C4	120.59 (16)	C313—C314—C315	120.84 (16)
C5—C4A—C4	121.35 (16)	C313—C314—I314	120.05 (13)
C6—C5—C4A	120.26 (18)	C315—C314—I314	119.08 (13)
C6—C5—H5	119.9	C316—C315—C314	119.42 (17)
C4A—C5—H5	119.9	C316—C315—H315	120.3
C5—C6—C7	120.37 (18)	C314—C315—H315	120.3
C5—C6—H6	119.8	C315—C316—C311	120.53 (16)
C7—C6—H6	119.8	C315—C316—H316	119.7
C8—C7—C6	120.65 (17)	C311—C316—H316	119.7
C8—C7—H7	119.7	O3—C31—N3	124.75 (16)
C6—C7—H7	119.7	O3—C31—C3	121.00 (16)
C7—C8—C8A	117.95 (17)	N3—C31—C3	114.25 (15)
C8A—O1—C2—C3	−1.7 (2)	C4—C4A—C8A—C8	−176.55 (16)
O1—C2—C3—C4	3.0 (3)	C7—C8—C8A—O1	177.28 (15)
O1—C2—C3—C31	−177.33 (15)	C7—C8—C8A—C4A	−2.4 (3)
C2—C3—C4—O4	179.64 (16)	C31—N3—C311—C312	0.3 (3)
C31—C3—C4—O4	0.0 (3)	C31—N3—C311—C316	179.48 (16)
C2—C3—C4—C4A	−0.9 (2)	C316—C311—C312—C313	−0.7 (2)
C31—C3—C4—C4A	179.46 (15)	N3—C311—C312—C313	178.37 (16)
O4—C4—C4A—C8A	177.21 (15)	C311—C312—C313—C314	0.1 (3)
C3—C4—C4A—C8A	−2.3 (2)	C312—C313—C314—C315	0.8 (3)
O4—C4—C4A—C5	−1.7 (3)	C312—C313—C314—I314	−177.36 (13)
C3—C4—C4A—C5	178.81 (15)	C313—C314—C315—C316	−1.0 (3)
C8A—C4A—C5—C6	−0.2 (3)	I314—C314—C315—C316	177.17 (12)
C4—C4A—C5—C6	178.65 (16)	C314—C315—C316—C311	0.3 (3)
C4A—C5—C6—C7	−1.7 (3)	C312—C311—C316—C315	0.5 (3)
C5—C6—C7—C8	1.6 (3)	N3—C311—C316—C315	−178.64 (15)
C6—C7—C8—C8A	0.4 (3)	C311—N3—C31—O3	2.5 (3)
C2—O1—C8A—C4A	−1.8 (2)	C311—N3—C31—C3	−177.41 (15)
C2—O1—C8A—C8	178.47 (15)	C2—C3—C31—O3	2.3 (2)
C5—C4A—C8A—O1	−177.34 (15)	C4—C3—C31—O3	−178.01 (15)
C4—C4A—C8A—O1	3.7 (2)	C2—C3—C31—N3	−177.74 (14)
C5—C4A—C8A—C8	2.4 (3)	C4—C3—C31—N3	1.9 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O4	0.92 (2)	1.89 (2)	2.6977 (19)	145 (2)
C2—H2···O3	0.95	2.33	2.718 (2)	104
C312—H312···O3	0.95	2.27	2.881 (2)	122
C2—H2···O4[i]	0.95	2.44	3.185 (2)	136
C316—H316···O3[ii]	0.95	2.49	3.312 (2)	145

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