research communications

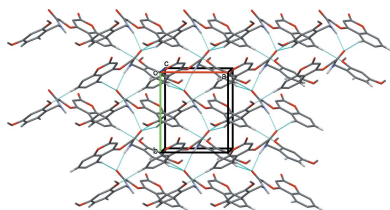# The crystal structures of two new coumarin derivatives: 2-(4-{2-[(2-oxo-2H-chromen-4-yl)-oxy]acetyl}piperazin-1-yl)acetamide and N-(2,4-dimethoxybenzyl)-2-[(2-oxo-2H-chromen-4-yl)oxy]-acetamide

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The title compounds, 2-(4-{2-[(2-oxo-2H-chromen-4-yl)oxy]acetyl}piperazin-1-yl)acetamide, $C_{17}H_{19}N_3O_5$, (I), and N-(2,4-dimethoxybenzyl)-2-[(2-oxo-2H-chromen-4-yl)oxy]acetamide, $C_{20}H_{19}NO_6$, (II), are new coumarin derivatives. In compound (I), the six-membered piperazine adopts a chair conformation. The dihedral angles between the mean planes of the chromene ring and amide plane is 82.65 (7)° in (I) and 26.2 (4)° in (II). The dihedral angles between the mean planes of the chromene ring and the four planar C atoms of the piperazine ring in (I) and the benzene ring in (II) are 87.66 (6) and 65.0 (4)°, respectively. There are short intramolecular contacts in both molecules forming $S(5)$ ring motifs, viz. N—H···N and C—H···O in (I), and N—H···O and C—H···N in (II). In the crystals of both compounds, molecules are linked by N—H···O hydrogen bonds, forming chains along [1$\bar{1}$0] in (I) and [010] in (II). The chains are linked by C—H···O hydrogen bonds, forming layers parallel to the ab plane in the crystals of both compounds. In the crystal of (I), there are also C—H···π and offset π–π interactions [intercentroid distance = 3.691 (1) Å] present within the layers. In the crystal of (II), there are only weak offset π–π interactions [intercentroid distance = 3.981 (6) Å] present within the layers. The intermolecular contacts in the crystals of both compounds have been analysed using Hirshfeld surface analysis and two-dimensional fingerprint plots.

1. Chemical context

Coumarin and its derivatives represent one of the most active classes of compounds possessing a wide spectrum of biological activity. The synthesis, and pharmacological and other properties of coumarin derivatives have been studied and reviewed (Kumar et al., 2015; Kubrak et al., 2017; Srikrishna et al., 2018; Venugopala et al., 2013). Many of these compounds have proven to be active as antibacterial, antifungal, anti-inflammatory, anticoagulant, anti-HIV and antitumor agents. One of the title compounds, 2-(4-{2-[(2-oxo-2H-chromen-4-yl)oxy]-acetyl}piperazin-1-yl)acetamide (I), has been shown to exhibit antimicrobial as well as antioxidant activity (Govindhan, Subramanian, Chennakesava Rao et al., 2015; Govindhan, Subramanian, Sridharan et al., 2015). In view of the importance of their natural occurrence, biological activities, pharmacological and medicinal activities, and utility as synthetic intermediates, we have synthesized the title 2-[(2-oxo-2H-

chromen-4-yl)oxy]acetamide derivatives, and report herein their crystal structures and Hirshfeld surface analysis.
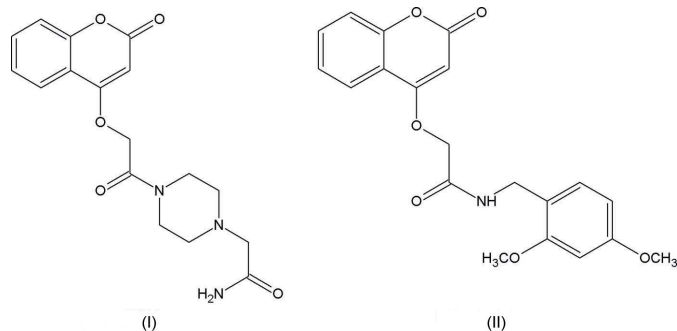
2. Structural commentary

The molecular structures of compounds (I) and (II) are illustrated in Figs. 1 and 2, respectively. In (I), the piperazine ring (N1/N2/C12–C15) is attached to the 2-[(2-oxochroman-4-yl)oxy]acetaldehyde moiety on atom N1 and to an acetamide moiety on atom N2. It has a chair conformation [puckering parameters: total amplitude $Q = 0.561$ (2) Å, $\theta = 0.67$ (2)° and $\varphi = 149$ (2)°], and is positioned *anti* with respect to the C—N rotamer of the amide. Nevertheless, because the asymmetry of the chromene residue, the *anti* conformation can assume a *cis* or *trans* geometry with respect to the relative position of the carbonyl O atom of the carboxamide and the C10—C11 and C16—C17 bonds. Both compounds exhibit a *cis* relation between these bonds, as can be seen in Figs. 1 and 2. This molecular conformation permits the formation of intramolecular hydrogen bonds (Tables 1 and 2), which enhance the relative planarity of each compound. Specifically for each
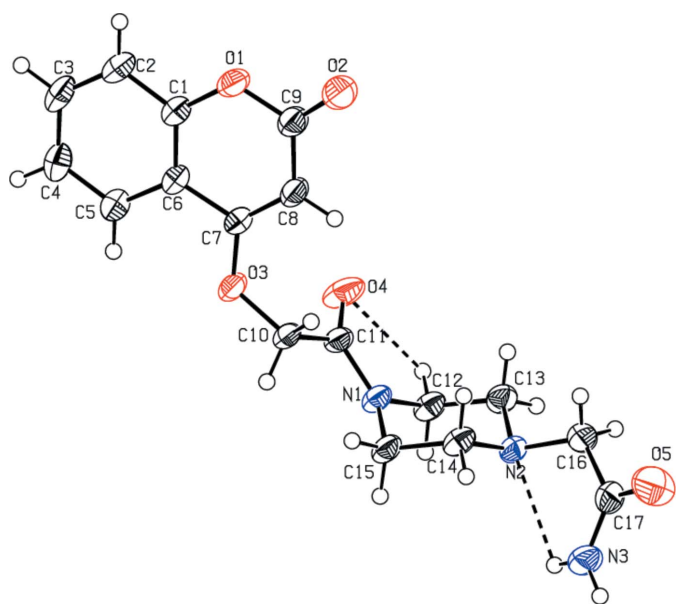

Figure 2
The molecular structure of compound (II), with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level. Intramolecular contacts (Table 2) are shown as dashed lines.
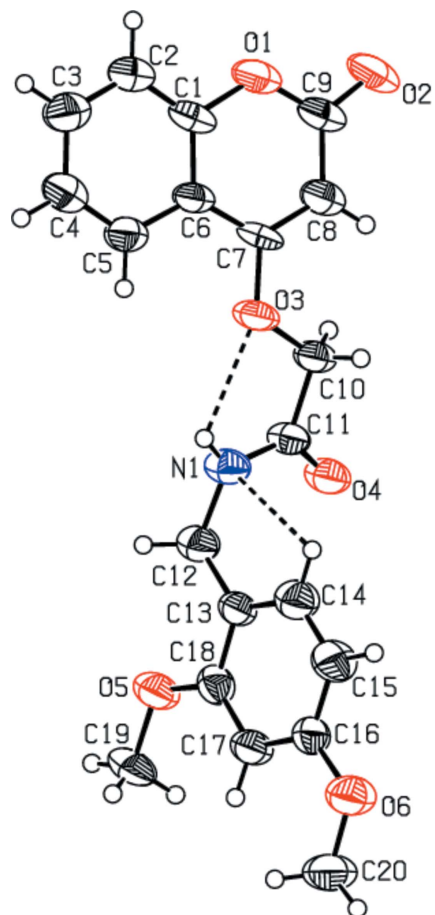
compound, as a result of the presence of the imidic nitrogen atom, the molecules display intramolecular N—H···N and N—H···O hydrogen bonds, between the amide nitrogen and the nitrogen atom N2 of the piperazine ring for compound (I), and oxygen atom O3 for (II), forming $S(5)$ ring motifs. In addition, the carbonyl oxygen atom O4 acts as the acceptor for a weak interaction with a hydrogen bond of the exocyclic piperazine ring, forming a second $S(5)$ ring motif in (I), and the amide nitrogen atom N1 acts as the acceptor for a weak interaction with a hydrogen bond of the exocyclic benzene ring, forming a second $S(5)$ ring motif in (II).

The values of the dihedral angles between the mean planes of the planar chromene ring system (O1/C1–C9; r.m.s. deviations = 0.008 Å for both compounds) and the amide plane (C10/C11/O4/N1) are 82.65 (7) and 26.2 (4)° in compounds (I) and (II), respectively. In (I), the dihedral angle between the mean planes of the chromene ring and the four C atoms (C12–C15) of the piperazine ring is 87.66 (6)°, while in (II) the benzene ring (C13–C18) is inclined to the mean plane of the chromene ring by 65.0 (4)°. Atom O2 deviates from the coumarin ring mean plane by 0.051 (1) Å in (I) and −0.043 (9) Å in (II).

It is interesting to compare the intramolecular hydrogen bonding present in the title compounds with that of the

Figure 1
The molecular structure of compound (I), with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level. Intramolecular contacts (Table 1) are shown as dashed lines.

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

$Cg1$ is the centroid of the C1–C6 ring.

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3B···N2	0.86	2.41	2.7716 (15)	106
C12—H12A···O4	0.97	2.35	2.7473 (15)	104
N3—H3A···O4[i]	0.86	2.05	2.8886 (15)	166
C8—H8···O2[ii]	0.93	2.56	3.3953 (16)	150
C10—H10A···O5[iii]	0.97	2.49	3.4506 (18)	173
C10—H10B···O2[ii]	0.97	2.42	3.3346 (16)	157
C14—H14A···O2[ii]	0.97	2.54	3.4012 (17)	148
C14—H14B···$Cg1$[i]	0.97	2.80	3.614 (2)	142

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

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3	0.86	2.31	2.669 (2)	105
C14—H14···N1	0.93	2.59	2.923 (2)	101
N1—H1···O4[i]	0.86	2.09	2.900 (2)	156
C3—H3···O5[ii]	0.93	2.49	3.419 (2)	175
C5—H5···O4[i]	0.93	2.43	3.307 (2)	157
C15—H15···O4[iii]	0.93	2.51	3.399 (2)	160

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

analogous 4-oxo-N-(substituted phenyl)-4H-chromene-2-carboxamides (Reis *et al.*, 2013; Gomes *et al.*, 2013). 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}—$C_{chromene}$ bond. These particular differences in conformation may condition the ability for docking when pharmacological activities are considered.

3. Supramolecular features

In the crystal of (I), molecules are linked by N3—H3A···O4[i] hydrogen bonds, forming chains along the [1$\bar{1}$0] direction, see Fig. 3 and Table 1. The chains are linked by C—H···O hydrogen bonds, forming layers lying parallel to the *ab* plane (Fig. 3 and Table 1). The C14—H14A···O2[ii] hydrogen bond generates an inversion dimer with an $R_2^2(22)$ ring motif; within the ring C8—H8···O2[ii] and C10—H10B···O2[ii] hydrogen bonds link the molecules into $R_2^2(8)$ and $R_2^2(14)$ rings, respectively. These rings are linked by $C(10)$ and $C(7)$ chains formed *via* the C10—H10A···O5[iii] and N3—H3A···O4[i] hydrogen bonds, respectively. A C—H···π interaction is also present within the layer (Table 1). An offset π–π contact between inversion-related chromene rings further stabilizes the crystal structure [$Cg2$···$Cg2$[iv] = 3.691 (1) Å, interplanar distance = 3.490 (1) Å, offset = 1.20 Å; $Cg2$ is the centroid of the O1/C1–C9 ring; symmetry code: (iv) $-x + 1, -y + 1, -z + 1$].

In the crystal of (II), molecules are linked by N1—H1···O4[i] hydrogen bonds, forming chains along the [010] direction, see Fig. 4 and Table 2. The chains are linked by C3—H3···O5[ii], C5—H5···O4[i] and C15—H15···O4[iii] hydrogen bonds, forming layers parallel to the *ab* plane (Fig. 4 and Table 2). Within the layer there are no C—H···π interactions present, only weak offset π–π interactions involving the benzene ring of the chromene ring system and the dimethoxybenzene ring [$Cg2$···$Cg3$[iv] = 3.981 (6) Å, interplanar distances = 3.638 (4) and 3.508 (4) Å, offset 0 1.88 Å; $Cg2$ and $Cg3$ are the centroids of rings C1–C6 and C13–C18, respectively; symmetry code: (iv) $-x + 1, y + \frac{1}{2}, -z + 1$].

4. Hirshfeld surface analysis

A recent article by Tiekink and collaborators (Tan *et al.*, 2019) reviews and describes the uses and utility of Hirshfeld surface analysis (Spackman & Jayatilaka, 2009), and the associated two-dimensional fingerprint plots (McKinnon *et al.*, 2007), to analyse intermolecular contacts in crystals. The various calculations were performed with *CrystalExplorer17* (Turner *et al.*, 2017).
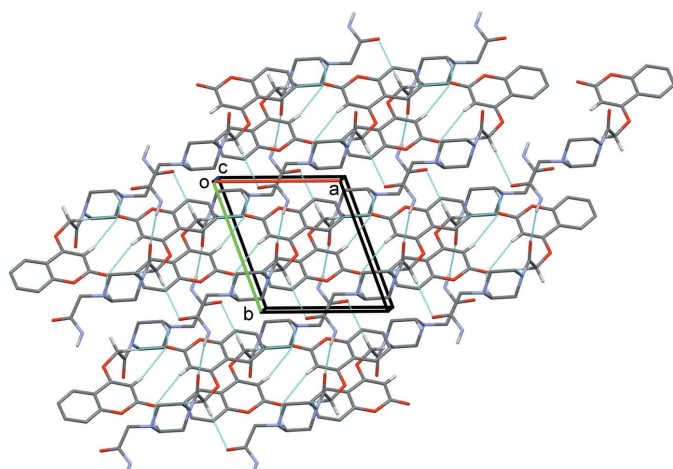

Figure 3
A view along the *c* axis of the crystal packing of compound (I). The hydrogen bonds (Table 1) are shown as dashed lines, and H atoms not involved in hydrogen bonding have been omitted.
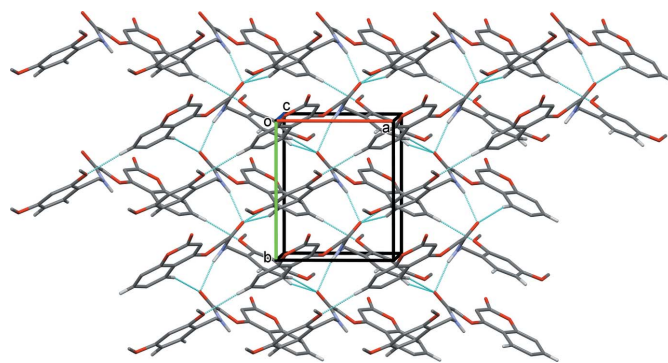

Figure 4
A view along the *a* axis of the crystal packing of compound (II). The hydrogen bonds (Table 2) are shown as dashed lines, and H atoms not involved in hydrogen bonding have been omitted.
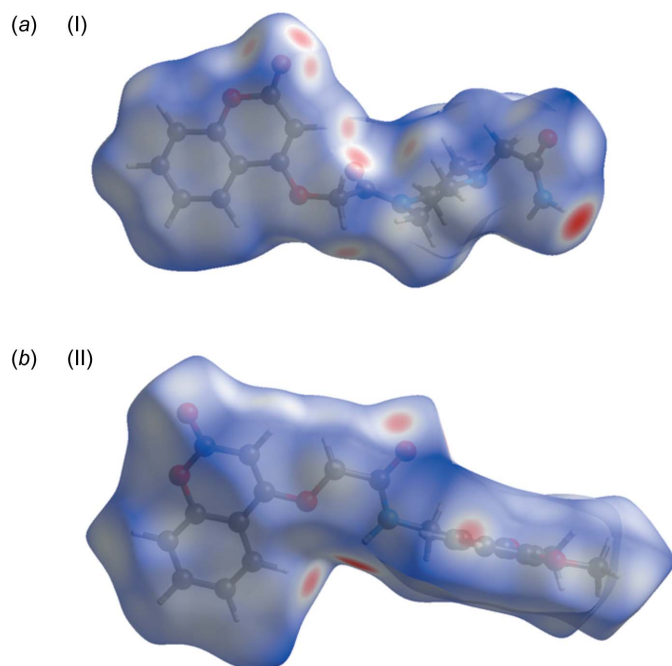
(a) (I)

(b) (II)

Figure 5
The Hirshfeld surfaces of compounds (a) (I) and (b) (II), mapped over d_{norm}
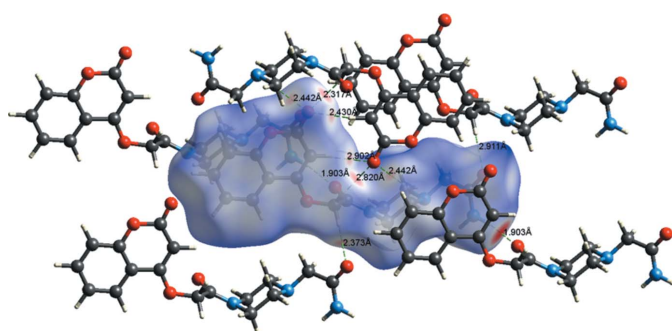

Figure 6
A view of the Hirshfeld surface mapped over d_{norm} of compound (I), showing the various intermolecular contacts in the crystal.
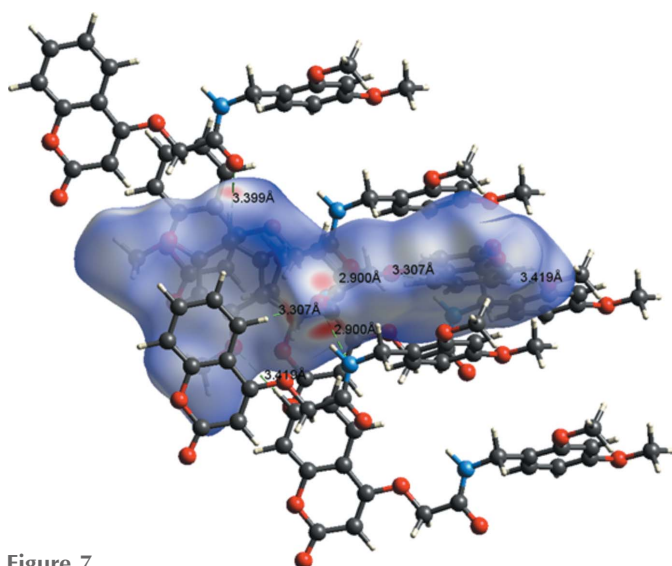

Figure 7
A view of the Hirshfeld surface mapped over d_{norm} of compound (II), showing the various intermolecular contacts in the crystal.
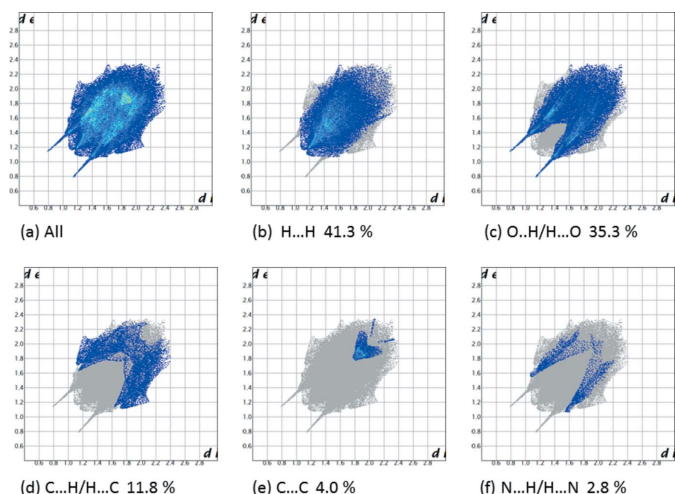

(a) All

(b) H···H 41.3 %

(c) O···H/H···O 35.3 %

(d) C···H/H···C 11.8 %

(e) C···C 4.0 %

(f) N···H/H···N 2.8 %

Figure 8
The full two-dimensional fingerprint plot for compound (I), and fingerprint plots delineated into (b) H···H, (c) O···H/H···O, (d) C···H/H···C, (e) C···C and (f) N···H/H···N contacts.

The Hirshfeld surfaces of compounds (I) and (II) mapped over d_{norm} are given in Fig. 5, and the intermolecular contacts are illustrated in Fig. 6 for (I) and Fig. 7 for (II). They are colour-mapped with the normalized contact distance, d_{norm}, from red (distances shorter than the sum of the van der Waals radii) through white to blue (distances longer than the sum of the van der Waals radii). The d_{norm} surface was mapped over a fixed colour scale of −0.544 (red) to 1.418 (blue) for compound (I) and −0.501 (red) to 1.672 (blue) for compound (II), where the red spots indicate the intermolecular contacts involved in the hydrogen bonding.

The fingerprint plots are given in Figs. 8 and 9. For compound (I), they reveal that the principal intermolecular contacts are H···H at 41.3% (Fig. 8b) and O···H/H···O at 35.3% (Fig. 8c), followed by the C···H/H···C contacts at

(a) All

(b) H···H 41.8 %

(c) O···H/H···O 32.4 %
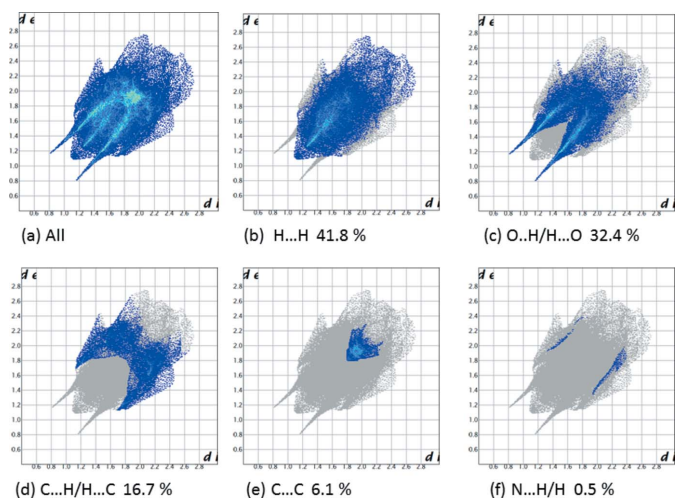
(d) C···H/H···C 16.7 %

(e) C···C 6.1 %

(f) N···H/H 0.5 %

Figure 9
The full two-dimensional fingerprint plot for compound (II), and fingerprint plots delineated into (b) H···H, (c) O···H/H···O, (d) C···H/H···C, (e) C···C and (f) N···H/H···N contacts.

Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_{17}H_{19}N_3O_5$	$C_{20}H_{19}NO_6$
M_r	345.35	369.36
Crystal system, space group	Triclinic, $P\bar{1}$	Monoclinic, $P2_1$
Temperature (K)	293	296
a, b, c (Å)	8.5260 (3), 8.8415 (3), 11.9462 (4)	7.2779 (2), 8.5759 (3), 14.4099 (5)
α, β, γ (°)	88.660 (2), 69.568 (2), 70.724 (2)	90, 93.796 (5), 90
V (Å^3)	792.27 (5)	897.41 (5)
Z	2	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.11	0.10
Crystal size (mm)	$0.25 \times 0.24 \times 0.20$	$0.30 \times 0.25 \times 0.20$
Data collection		
Diffractometer	Bruker Kappa APEXII CCD	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2008)	Multi-scan (*SADABS*; Bruker, 2008)
T_{min}, T_{max}	0.756, 0.824	0.763, 0.852
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	12022, 3382, 2947	4058, 2630, 1623
R_{int}	0.027	0.088
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.637	0.595
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.039, 0.110, 1.04	0.083, 0.243, 0.98
No. of reflections	3382	2630
No. of parameters	227	247
No. of restraints	0	1
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.30, −0.18	0.28, −0.29

Computer programs: *APEX2* and *SAINT* (Bruker, 2008), *SHELXS2018* (Sheldrick, 2008), *SHELXL2018* (Sheldrick, 2015), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008), *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

11.8% (Fig. 8*d*). For compound (II), they reveal a similar trend, with the principal intermolecular contacts being H···H at 41.8% (Fig. 9*b*) and O···H/H···O at 32.4% (Fig. 9*c*), followed by the C···H/H···C contacts at 16.7% (Fig. 9*d*). In both compounds, the H···H intermolecular contacts predominate, followed by the O···H/H···O contacts. However the C···H/H···C contacts are significantly different 11.8% *cf.* 16.7% for (I) and (II), respectively.

5. Database survey

A search of the Cambridge Structural Database (CSD, V5.40, update February 2019; Groom *et al.*, 2016) for 2-[(2-oxo-2*H*-chromen-4-yl)oxy]acetamide derivatives gave two hits. They include 2-[(2-oxo-2*H*-chromen-4-yl)oxy]-*N*-(1-phenylethyl)-acetamide (CSD refcode PUWMEB; Govindhan, Subramanian, Chennakesava Rao *et al.*, 2015) and *N*-(3,5-dimethyl-adamantan-1-yl)-2-[(2-oxo-2*H*-chromen-4-yl)oxy]propanamide (SEFRAY; Rambabu *et al.*, 2012).

A search for linear and angular pyranocoumarin (psoralene class) structures gave 35 hits. They include four reports, CSD refcodes AMYROL [Kato, 1970: seselin (smyrolin)]; AMYROL01 [Bauri *et al.*, 2006; seselin (redetermination)]; FUGVOS [Thailambal & Pattabhi, 1987: 2,3-dihydroxy-9-hydroxy-2(1-hydroxy-1-methylethyl)-7*H*-furo-[3,2-*g*]-[1]-benzo-pyran-7-one; bromohydroxyseselin (Bauri *et al.*, 2017*a*); dibromomomethoxyseselin (DMS) (Bauri *et al.*, 2017*b*)], and a

number of structures with various substituents at C3 and C4, many of which are natural products.

A CSD search found five coumarin ester structures with substituents at the 7 position (Ramasubbu *et al.*, 1982; Gnanaguru *et al.*, 1985; Parveen *et al.*, 2011; Zhuo *et al.*, 2014; Ji *et al.*, 2017). In these structures and those of *meta*-substituted coumarin esters (Abou *et al.*, 2012, 2013; Bibila Mayaya Bisseyou *et al.*, 2013; Zhang *et al.*, 2014; Gomes *et al.*, 2016; Ziki *et al.*, 2016, 2017), the pyrone rings all show three long (in the range 1.37–1.46 Å) and one short (1.32–1.34 Å) C—C distances, suggesting that the electronic density is preferentially located in the short C—C bond at the pyrone ring. This pattern is clearly repeated here with C1—C6 = 1.3883 (18) and 1.394 (11) Å, C6—C7 = 1.4538 (15) and 1.398 (12) Å, C7—C8 = 1.3444 (17) and 1.352 (12) Å and C8—C9 = 1.4338 (18) and 1.433 (12) Å.

Intramolecular C—H···O short contacts similar to that observed in the title compounds were found in five compounds in the CSD: LISLAB, 1-(1-pyrrolidinylcarbonyl)cyclopropyl sulfamate (Morin *et al.*, 2007), PEQHAU, 2-[30-(400-chloro-phenyl)-40,60-dimethoxyindol-70-yl]glyoxyl-1-pyrrolidine (Black *et al.*, 1997), QIBBEJ, [2-hydroxy-5-(2-hydroxybenzo-yl)phenyl](pyrrolidin-1-yl)-methanone (Holtz *et al.*, 2007), SINHAZ, 2-methoxy-1-(1-pyrrolidinylcarbonyl)naphthalene (Sakamoto *et al.*, 2007) and TAJDIR, (4S,5S)-4,5-bis-pyrrolidinylcarbonyl)-2,2-dimethyl-1,3-dioxolane (Garcia *et al.*, 1991).

6. Synthesis and crystallization

Compound (I) To a solution of 1 equiv. of 4-(2-(piperazine-1-yl)ethoxy)-2*H*-chromen-2-one (1.0 g) in dichloromethane (10 ml) at 273–278 K were added triethylamine (0.7 g, 2.0 equiv.) followed by iodoacetamide (1.0 g, 0.5 equiv.), and the reaction mixture was stirred at the same temperature for 1 h. On completion of the reaction (monitored by TLC), the reaction mixture was diluted with dichloromethane and water (10 ml). The organic layer was separated and washed with brine solution. It was then dried over anhydrous sodium sulfate, filtered and then evaporated under reduced pressure giving compound (I) as a white solid, which was then washed with hexane and dried under vacuum. Colourless block-like crystals of compound (I) were obtained by slow evaporation of a solution in chloroform (4 ml) and methanol (1 ml).

Compound (II) *N,N*-Diisopropylethylamine (DIPEA; 1.82 g, 3.1 equiv.) was added to a mixture of 2-(2-oxo-2*H*-chromen-4-yloxy)acetic acid (1.0 g, 1.0 equiv.), 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide (EDCI; 1.0 g, 1.2 equiv.), 1-hydroxybenzotriazole hydrate (HOBt; 0.61 g, 1.0 equiv.), 2,4-dimethoxybenzylamine (0.8 g, 1.0 equiv.) in *N,N*-dimethylformamide (5 ml) at 273–278 K. The temperature of the mixture was raised to ambient temperature and stirred for 8 h. Progress of the reaction was monitored by TLC (mobile phase: ethyl acetate/hexane). After completion of the reaction, the mixture was poured into ice–water and compound (II) was obtained as a white solid. It was then filtered, washed with hexane and dried under vacuum. Colourless block-like crystals of compound (II) were obtained by slow evaporation of a solution in chloroform (5 ml).

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. For both compounds the H atoms were positioned geometrically and constrained to ride on their parent atoms: N—H = 0.86 Å and C–H = 0.93—0.97Å with $U_{iso}(H) = 1.5U_{eq}$(C-methyl) and $1.2U_{eq}$(N, C) for other H atoms.

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supporting information

The crystal structures of two new coumarin derivatives: 2-(4-{2-[(2-oxo-2*H*-chromen-4-yl)oxy]acetyl}piperazin-1-yl)acetamide and *N*-(2,4-dimethoxy-benzyl)-2-[(2-oxo-2*H*-chromen-4-yl)oxy]acetamide

S. Syed Abuthahir, M. NizamMohideen, V. Viswanathan, M. Govindhan and K. Subramanian

Computing details

For both structures, data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008); program(s) used to solve structure: *SHELXS2018* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *WinGX* (Farrugia, 2012), *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

2-(4-{2-[(2-Oxo-2*H*-chromen-4-yl)oxy]acetyl}piperazin-1-yl)acetamide (I)

Crystal data

$C_{17}H_{19}N_3O_5$	$Z = 2$
$M_r = 345.35$	$F(000) = 364$
Triclinic, $P\overline{1}$	$D_x = 1.448$ Mg m^{-3}
$a = 8.5260$ (3) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 8.8415$ (3) Å	Cell parameters from 3382 reflections
$c = 11.9462$ (4) Å	$\theta = 1.8$–26.9°
$\alpha = 88.660$ (2)°	$\mu = 0.11$ mm^{-1}
$\beta = 69.568$ (2)°	$T = 293$ K
$\gamma = 70.724$ (2)°	Block, colourless
$V = 792.27$ (5) Å^3	$0.25 \times 0.24 \times 0.20$ mm

Data collection

Bruker Kappa APEXII CCD diffractometer	3382 independent reflections
	2947 reflections with $I > 2\sigma(I)$
ω and φ scans	$R_{int} = 0.027$
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)	$\theta_{max} = 26.9°$, $\theta_{min} = 1.8°$
	$h = -10 \rightarrow 10$
$T_{min} = 0.756$, $T_{max} = 0.824$	$k = -11 \rightarrow 11$
12022 measured reflections	$l = -15 \rightarrow 15$

Refinement

Refinement on F^2	227 parameters
Least-squares matrix: full	0 restraints
$R[F^2 > 2\sigma(F^2)] = 0.039$	Primary atom site location: structure-invariant direct methods
$wR(F^2) = 0.110$	
$S = 1.04$	Secondary atom site location: difference Fourier map
3382 reflections	

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0556P)^2 + 0.2007P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.30$ e Å^{-3}

$\Delta\rho_{min} = -0.18$ e Å^{-3}

Extinction correction: (SHELXL2018; Sheldrick, 2015), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.020 (4)

Special details

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

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

	x	*y*	*z*	$U_{iso}*/U_{eq}$
C1	0.57138 (16)	0.24849 (15)	0.44898 (11)	0.0326 (3)
C2	0.72086 (18)	0.15454 (16)	0.47279 (13)	0.0396 (3)
H2	0.713439	0.076848	0.526334	0.047*
C3	0.87983 (18)	0.17916 (17)	0.41538 (13)	0.0426 (3)
H3	0.980829	0.116794	0.430161	0.051*
C4	0.89217 (18)	0.29534 (18)	0.33584 (13)	0.0419 (3)
H4	1.000730	0.310220	0.297651	0.050*
C5	0.74271 (16)	0.38890 (16)	0.31349 (12)	0.0355 (3)
H5	0.750512	0.467720	0.260957	0.043*
C6	0.57975 (15)	0.36540 (14)	0.36965 (11)	0.0297 (3)
C7	0.41622 (16)	0.45736 (14)	0.35108 (11)	0.0298 (3)
C8	0.26528 (16)	0.42648 (15)	0.40841 (12)	0.0359 (3)
H8	0.162437	0.483635	0.393790	0.043*
C9	0.25981 (17)	0.30714 (16)	0.49166 (13)	0.0385 (3)
C10	0.28057 (16)	0.68340 (15)	0.26066 (12)	0.0328 (3)
H10A	0.295441	0.787474	0.249025	0.039*
H10B	0.176835	0.696178	0.332561	0.039*
C11	0.25178 (15)	0.62523 (14)	0.15331 (11)	0.0316 (3)
C12	0.11451 (17)	0.69477 (15)	0.00279 (11)	0.0342 (3)
H12A	0.191175	0.585566	−0.031236	0.041*
H12B	0.137190	0.766053	−0.059147	0.041*
C13	−0.07778 (17)	0.70594 (14)	0.04377 (13)	0.0358 (3)
H13A	−0.104008	0.680104	−0.024677	0.043*
H13B	−0.097577	0.627835	0.100571	0.043*
C14	−0.15445 (17)	0.91151 (15)	0.20136 (11)	0.0349 (3)
H14A	−0.176974	0.839746	0.262958	0.042*
H14B	−0.231186	1.020451	0.236057	0.042*
C15	0.03821 (17)	0.90093 (14)	0.16159 (12)	0.0348 (3)
H15A	0.058654	0.980127	0.105913	0.042*
H15B	0.064622	0.924109	0.230622	0.042*
C16	−0.38171 (17)	0.87031 (15)	0.14408 (15)	0.0423 (3)
H16A	−0.401742	0.814610	0.215885	0.051*
H16B	−0.396724	0.810638	0.083757	0.051*

C17	−0.52246 (17)	1.03707 (16)	0.17290 (14)	0.0419 (3)
N1	0.15528 (13)	0.73936 (12)	0.10337 (9)	0.0319 (2)
N2	−0.19807 (13)	0.86796 (11)	0.10060 (9)	0.0308 (2)
N3	−0.47248 (16)	1.15212 (14)	0.11445 (12)	0.0465 (3)
H3A	−0.547778	1.248448	0.125737	0.056*
H3B	−0.364686	1.130611	0.065147	0.056*
O1	0.41560 (12)	0.22028 (11)	0.50832 (9)	0.0416 (2)
O2	0.12892 (14)	0.27595 (14)	0.55044 (11)	0.0566 (3)
O3	0.43529 (11)	0.57057 (11)	0.27551 (9)	0.0382 (2)
O4	0.31673 (15)	0.48319 (11)	0.11304 (10)	0.0529 (3)
O5	−0.67279 (15)	1.05727 (14)	0.24444 (14)	0.0753 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0309 (6)	0.0317 (6)	0.0322 (6)	−0.0043 (5)	−0.0139 (5)	0.0023 (5)
C2	0.0411 (7)	0.0344 (6)	0.0401 (7)	−0.0007 (5)	−0.0225 (6)	0.0044 (5)
C3	0.0347 (7)	0.0414 (7)	0.0480 (8)	0.0031 (5)	−0.0252 (6)	−0.0034 (6)
C4	0.0283 (6)	0.0502 (8)	0.0437 (8)	−0.0067 (6)	−0.0150 (6)	−0.0027 (6)
C5	0.0309 (6)	0.0402 (7)	0.0330 (7)	−0.0080 (5)	−0.0128 (5)	0.0042 (5)
C6	0.0275 (6)	0.0309 (6)	0.0280 (6)	−0.0032 (5)	−0.0132 (5)	0.0006 (5)
C7	0.0298 (6)	0.0305 (6)	0.0285 (6)	−0.0061 (5)	−0.0141 (5)	0.0057 (5)
C8	0.0282 (6)	0.0379 (7)	0.0415 (7)	−0.0068 (5)	−0.0173 (5)	0.0110 (5)
C9	0.0312 (6)	0.0395 (7)	0.0431 (7)	−0.0089 (5)	−0.0146 (5)	0.0109 (6)
C10	0.0287 (6)	0.0318 (6)	0.0394 (7)	−0.0072 (5)	−0.0177 (5)	0.0130 (5)
C11	0.0277 (6)	0.0270 (6)	0.0371 (7)	−0.0049 (4)	−0.0129 (5)	0.0088 (5)
C12	0.0354 (6)	0.0293 (6)	0.0328 (6)	−0.0015 (5)	−0.0153 (5)	0.0001 (5)
C13	0.0385 (7)	0.0252 (6)	0.0436 (7)	−0.0057 (5)	−0.0195 (6)	−0.0003 (5)
C14	0.0347 (6)	0.0295 (6)	0.0336 (7)	−0.0003 (5)	−0.0142 (5)	0.0009 (5)
C15	0.0372 (7)	0.0236 (5)	0.0443 (7)	−0.0008 (5)	−0.0243 (6)	−0.0003 (5)
C16	0.0325 (7)	0.0316 (6)	0.0635 (9)	−0.0104 (5)	−0.0192 (6)	0.0099 (6)
C17	0.0281 (6)	0.0363 (7)	0.0591 (9)	−0.0070 (5)	−0.0171 (6)	0.0049 (6)
N1	0.0322 (5)	0.0250 (5)	0.0363 (6)	−0.0012 (4)	−0.0179 (4)	0.0017 (4)
N2	0.0278 (5)	0.0250 (5)	0.0388 (6)	−0.0047 (4)	−0.0153 (4)	0.0032 (4)
N3	0.0340 (6)	0.0324 (6)	0.0650 (8)	−0.0031 (5)	−0.0166 (6)	0.0110 (5)
O1	0.0351 (5)	0.0414 (5)	0.0469 (6)	−0.0093 (4)	−0.0178 (4)	0.0197 (4)
O2	0.0375 (5)	0.0654 (7)	0.0673 (7)	−0.0209 (5)	−0.0181 (5)	0.0328 (6)
O3	0.0280 (4)	0.0439 (5)	0.0448 (5)	−0.0100 (4)	−0.0190 (4)	0.0205 (4)
O4	0.0659 (7)	0.0272 (5)	0.0597 (7)	0.0043 (4)	−0.0346 (6)	0.0017 (4)
O5	0.0340 (6)	0.0528 (7)	0.1128 (11)	−0.0086 (5)	−0.0018 (6)	0.0128 (7)

Geometric parameters (Å, º)

C1—O1	1.3718 (16)	C11—N1	1.3482 (15)
C1—C6	1.3883 (18)	C12—N1	1.4574 (16)
C1—C2	1.3922 (17)	C12—C13	1.5073 (18)
C2—C3	1.376 (2)	C12—H12A	0.9700
C2—H2	0.9300	C12—H12B	0.9700

C3—C4	1.387 (2)	C13—N2	1.4677 (15)
C3—H3	0.9300	C13—H13A	0.9700
C4—C5	1.3823 (18)	C13—H13B	0.9700
C4—H4	0.9300	C14—N2	1.4712 (16)
C5—C6	1.3990 (17)	C14—C15	1.5127 (18)
C5—H5	0.9300	C14—H14A	0.9700
C6—C7	1.4538 (15)	C14—H14B	0.9700
C7—O3	1.3439 (15)	C15—N1	1.4632 (15)
C7—C8	1.3444 (17)	C15—H15A	0.9700
C8—C9	1.4338 (18)	C15—H15B	0.9700
C8—H8	0.9300	C16—N2	1.4604 (16)
C9—O2	1.2089 (16)	C16—C17	1.5164 (18)
C9—O1	1.3774 (15)	C16—H16A	0.9700
C10—O3	1.4328 (13)	C16—H16B	0.9700
C10—C11	1.5177 (18)	C17—O5	1.2232 (18)
C10—H10A	0.9700	C17—N3	1.3226 (19)
C10—H10B	0.9700	N3—H3A	0.8600
C11—O4	1.2241 (15)	N3—H3B	0.8600
O1—C1—C6	121.80 (11)	C13—C12—H12B	109.6
O1—C1—C2	116.65 (12)	H12A—C12—H12B	108.1
C6—C1—C2	121.54 (12)	N2—C13—C12	111.22 (10)
C3—C2—C1	118.55 (13)	N2—C13—H13A	109.4
C3—C2—H2	120.7	C12—C13—H13A	109.4
C1—C2—H2	120.7	N2—C13—H13B	109.4
C2—C3—C4	121.20 (12)	C12—C13—H13B	109.4
C2—C3—H3	119.4	H13A—C13—H13B	108.0
C4—C3—H3	119.4	N2—C14—C15	111.67 (10)
C5—C4—C3	119.83 (13)	N2—C14—H14A	109.3
C5—C4—H4	120.1	C15—C14—H14A	109.3
C3—C4—H4	120.1	N2—C14—H14B	109.3
C4—C5—C6	120.20 (13)	C15—C14—H14B	109.3
C4—C5—H5	119.9	H14A—C14—H14B	107.9
C6—C5—H5	119.9	N1—C15—C14	109.84 (10)
C1—C6—C5	118.66 (11)	N1—C15—H15A	109.7
C1—C6—C7	117.34 (11)	C14—C15—H15A	109.7
C5—C6—C7	123.99 (11)	N1—C15—H15B	109.7
O3—C7—C8	126.49 (11)	C14—C15—H15B	109.7
O3—C7—C6	113.36 (10)	H15A—C15—H15B	108.2
C8—C7—C6	120.14 (11)	N2—C16—C17	114.84 (10)
C7—C8—C9	121.26 (11)	N2—C16—H16A	108.6
C7—C8—H8	119.4	C17—C16—H16A	108.6
C9—C8—H8	119.4	N2—C16—H16B	108.6
O2—C9—O1	116.18 (12)	C17—C16—H16B	108.6
O2—C9—C8	125.71 (12)	H16A—C16—H16B	107.5
O1—C9—C8	118.11 (11)	O5—C17—N3	124.21 (13)
O3—C10—C11	110.44 (10)	O5—C17—C16	119.73 (13)
O3—C10—H10A	109.6	N3—C17—C16	116.04 (12)

C11—C10—H10A	109.6	C11—N1—C12	120.29 (10)
O3—C10—H10B	109.6	C11—N1—C15	125.01 (11)
C11—C10—H10B	109.6	C12—N1—C15	111.98 (9)
H10A—C10—H10B	108.1	C16—N2—C13	109.10 (10)
O4—C11—N1	122.28 (12)	C16—N2—C14	109.88 (10)
O4—C11—C10	121.53 (11)	C13—N2—C14	109.94 (9)
N1—C11—C10	116.17 (10)	C17—N3—H3A	120.0
N1—C12—C13	110.40 (10)	C17—N3—H3B	120.0
N1—C12—H12A	109.6	H3A—N3—H3B	120.0
C13—C12—H12A	109.6	C1—O1—C9	121.30 (10)
N1—C12—H12B	109.6	C7—O3—C10	119.29 (9)
O1—C1—C2—C3	−179.85 (11)	N2—C16—C17—O5	−155.82 (15)
C6—C1—C2—C3	−0.22 (19)	N2—C16—C17—N3	25.9 (2)
C1—C2—C3—C4	0.4 (2)	O4—C11—N1—C12	4.18 (19)
C2—C3—C4—C5	0.2 (2)	C10—C11—N1—C12	−177.62 (10)
C3—C4—C5—C6	−0.8 (2)	O4—C11—N1—C15	163.96 (13)
O1—C1—C6—C5	179.20 (11)	C10—C11—N1—C15	−17.84 (18)
C2—C1—C6—C5	−0.42 (18)	C13—C12—N1—C11	105.42 (13)
O1—C1—C6—C7	−0.25 (18)	C13—C12—N1—C15	−56.81 (14)
C2—C1—C6—C7	−179.87 (11)	C14—C15—N1—C11	−105.06 (14)
C4—C5—C6—C1	0.93 (19)	C14—C15—N1—C12	56.17 (14)
C4—C5—C6—C7	−179.66 (12)	C17—C16—N2—C13	−163.63 (12)
C1—C6—C7—O3	178.34 (10)	C17—C16—N2—C14	75.77 (15)
C5—C6—C7—O3	−1.09 (17)	C12—C13—N2—C16	−177.13 (11)
C1—C6—C7—C8	−0.84 (18)	C12—C13—N2—C14	−56.57 (14)
C5—C6—C7—C8	179.74 (12)	C15—C14—N2—C16	176.60 (10)
O3—C7—C8—C9	−176.93 (12)	C15—C14—N2—C13	56.50 (13)
C6—C7—C8—C9	2.1 (2)	C6—C1—O1—C9	0.03 (18)
C7—C8—C9—O2	176.98 (14)	C2—C1—O1—C9	179.66 (12)
C7—C8—C9—O1	−2.3 (2)	O2—C9—O1—C1	−178.15 (12)
O3—C10—C11—O4	21.02 (17)	C8—C9—O1—C1	1.20 (19)
O3—C10—C11—N1	−157.19 (10)	C8—C7—O3—C10	6.90 (19)
N1—C12—C13—N2	56.80 (14)	C6—C7—O3—C10	−172.21 (10)
N2—C14—C15—N1	−56.02 (13)	C11—C10—O3—C7	−94.52 (13)

Hydrogen-bond geometry (Å, °)

*Cg*1 is the centroid of the C1–C6 ring.

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3B···N2	0.86	2.41	2.7716 (15)	106
C12—H12A···O4	0.97	2.35	2.7473 (15)	104
N3—H3A···O4ⁱ	0.86	2.05	2.8886 (15)	166
C8—H8···O2ⁱⁱ	0.93	2.56	3.3953 (16)	150
C10—H10A···O5ⁱⁱⁱ	0.97	2.49	3.4506 (18)	173
C10—H10B···O2ⁱⁱ	0.97	2.42	3.3346 (16)	157

C14—H14A⋯O2^{ii}	0.97	2.54	3.4012 (17)	148
C14—H14B⋯Cg1^{i}	0.97	2.80	3.614 (2)	142

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

N-(2,4-Dimethoxybenzyl)-2-[(2-oxo-2H-chromen-4-yl)oxy]acetamide (II)

Crystal data

$C_{20}H_{19}NO_6$	$F(000) = 388$
$M_r = 369.36$	$D_x = 1.367$ Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 7.2779$ (2) Å	Cell parameters from 2630 reflections
$b = 8.5759$ (3) Å	$\theta = 1.4$–25.0°
$c = 14.4099$ (5) Å	$\mu = 0.10$ mm^{-1}
$\beta = 93.796$ (5)°	$T = 296$ K
$V = 897.41$ (5) Å^3	Block, colourless
$Z = 2$	0.30 × 0.25 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer	2630 independent reflections
	1623 reflections with $I > 2\sigma(I)$
ω and φ scans	$R_{int} = 0.088$
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)	$\theta_{max} = 25.0°$, $\theta_{min} = 1.4°$
$T_{min} = 0.763$, $T_{max} = 0.852$	$h = -8 \rightarrow 8$
	$k = -9 \rightarrow 9$
4058 measured reflections	$l = -17 \rightarrow 16$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.083$	H-atom parameters constrained
$wR(F^2) = 0.243$	$w = 1/[\sigma^2(F_o^2) + (0.1336P)^2]$
$S = 0.98$	where $P = (F_o^2 + 2F_c^2)/3$
2630 reflections	$(\Delta/\sigma)_{max} < 0.001$
247 parameters	$\Delta\rho_{max} = 0.28$ e Å^{-3}
1 restraint	$\Delta\rho_{min} = -0.29$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: (SHELXL2018; Sheldrick, 2015), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.042 (15)

Special details

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

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

	x	y	z	$U_{iso}*/U_{eq}$
C9	0.7608 (13)	0.3941 (14)	0.8943 (6)	0.063 (3)
C1	0.9935 (12)	0.5490 (11)	0.8258 (6)	0.053 (2)
C2	1.1588 (13)	0.6195 (14)	0.8397 (7)	0.064 (3)
H2	1.225084	0.612966	0.897037	0.077*

C3	1.2277 (13)	0.7016 (13)	0.7673 (7)	0.067 (3)
H3	1.340590	0.752129	0.775680	0.080*
C4	1.1281 (13)	0.7085 (14)	0.6818 (7)	0.065 (3)
H4	1.175985	0.762353	0.632809	0.078*
C5	0.9635 (12)	0.6383 (12)	0.6689 (6)	0.056 (3)
H5	0.898157	0.645409	0.611398	0.067*
C6	0.8876 (11)	0.5536 (11)	0.7417 (6)	0.048 (2)
C7	0.7177 (12)	0.4769 (10)	0.7354 (5)	0.047 (2)
C8	0.6529 (13)	0.3977 (13)	0.8075 (7)	0.060 (3)
H8	0.540283	0.346313	0.800782	0.072*
C10	0.4698 (11)	0.3848 (12)	0.6345 (6)	0.052 (2)
H10A	0.365667	0.432162	0.662342	0.062*
H10B	0.493567	0.284583	0.664029	0.062*
C11	0.4238 (11)	0.3613 (11)	0.5313 (6)	0.046 (2)
C12	0.4617 (11)	0.4368 (12)	0.3725 (6)	0.053 (2)
H12A	0.461193	0.326030	0.358746	0.064*
H12B	0.563467	0.482854	0.341984	0.064*
C13	0.2869 (10)	0.5056 (10)	0.3312 (5)	0.043 (2)
C14	0.1620 (12)	0.5896 (12)	0.3804 (7)	0.059 (3)
H14	0.190916	0.609186	0.443136	0.071*
C15	−0.0013 (12)	0.6448 (14)	0.3410 (7)	0.062 (3)
H15	−0.081969	0.699614	0.376388	0.074*
C16	−0.0442 (11)	0.6175 (12)	0.2473 (6)	0.052 (2)
C17	0.0765 (11)	0.5380 (11)	0.1954 (6)	0.052 (2)
H17	0.048794	0.523211	0.132044	0.062*
C18	0.2389 (11)	0.4798 (11)	0.2368 (6)	0.048 (2)
C19	0.3259 (14)	0.3671 (17)	0.0943 (6)	0.080 (4)
H19A	0.212442	0.310013	0.086580	0.120*
H19B	0.423474	0.306460	0.070819	0.120*
H19C	0.313972	0.463616	0.060635	0.120*
C20	−0.2598 (13)	0.6447 (17)	0.1157 (7)	0.085 (4)
H20A	−0.162895	0.679847	0.078717	0.127*
H20B	−0.371543	0.699158	0.097180	0.127*
H20C	−0.278034	0.534745	0.106668	0.127*
N1	0.4962 (10)	0.4564 (9)	0.4721 (5)	0.051 (2)
H1	0.565201	0.531787	0.492909	0.061*
O1	0.9285 (9)	0.4669 (9)	0.9009 (4)	0.068 (2)
O2	0.7213 (10)	0.3219 (10)	0.9634 (5)	0.084 (3)
O3	0.6246 (8)	0.4805 (7)	0.6494 (4)	0.0562 (18)
O4	0.3165 (8)	0.2542 (8)	0.5078 (4)	0.0560 (18)
O5	0.3671 (8)	0.3989 (9)	0.1907 (4)	0.0641 (19)
O6	−0.2109 (8)	0.6749 (9)	0.2111 (5)	0.072 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C9	0.079 (6)	0.082 (8)	0.028 (5)	−0.001 (6)	−0.004 (4)	−0.001 (5)
C1	0.074 (6)	0.060 (6)	0.025 (5)	0.007 (5)	−0.004 (4)	0.000 (4)

C2	0.065 (6)	0.087 (9)	0.039 (5)	−0.006 (6)	−0.009 (4)	0.004 (6)
C3	0.063 (6)	0.079 (8)	0.056 (7)	−0.010 (6)	−0.011 (5)	0.002 (6)
C4	0.073 (6)	0.080 (8)	0.042 (6)	−0.006 (6)	0.002 (4)	0.007 (6)
C5	0.057 (5)	0.066 (7)	0.042 (5)	−0.008 (5)	−0.008 (4)	0.009 (5)
C6	0.053 (5)	0.058 (6)	0.029 (5)	−0.002 (4)	−0.007 (4)	0.001 (4)
C7	0.071 (5)	0.049 (6)	0.018 (4)	0.002 (5)	−0.008 (4)	0.003 (4)
C8	0.068 (6)	0.075 (7)	0.036 (5)	−0.007 (5)	−0.003 (4)	0.004 (5)
C10	0.059 (5)	0.065 (7)	0.031 (5)	−0.012 (5)	−0.007 (4)	0.003 (5)
C11	0.057 (5)	0.046 (5)	0.033 (5)	−0.004 (5)	−0.007 (4)	0.004 (5)
C12	0.056 (5)	0.063 (6)	0.040 (5)	0.000 (5)	−0.002 (4)	−0.006 (5)
C13	0.049 (4)	0.048 (6)	0.032 (4)	−0.003 (4)	0.001 (3)	0.001 (4)
C14	0.059 (5)	0.072 (7)	0.048 (6)	0.006 (5)	0.000 (4)	−0.006 (5)
C15	0.063 (5)	0.077 (7)	0.046 (5)	0.012 (5)	0.007 (4)	−0.021 (6)
C16	0.043 (4)	0.073 (7)	0.041 (5)	0.011 (5)	0.005 (4)	0.001 (5)
C17	0.055 (5)	0.066 (6)	0.035 (5)	0.011 (5)	0.000 (4)	−0.001 (5)
C18	0.049 (4)	0.053 (6)	0.042 (5)	0.004 (4)	0.009 (4)	−0.003 (5)
C19	0.082 (7)	0.130 (11)	0.029 (5)	0.008 (7)	0.005 (4)	−0.006 (7)
C20	0.067 (6)	0.137 (11)	0.048 (6)	0.033 (7)	−0.012 (5)	−0.014 (7)
N1	0.065 (4)	0.046 (5)	0.041 (4)	−0.010 (4)	−0.009 (3)	0.001 (4)
O1	0.080 (4)	0.090 (5)	0.033 (4)	−0.009 (4)	−0.009 (3)	0.007 (4)
O2	0.105 (6)	0.115 (7)	0.030 (4)	−0.014 (5)	0.001 (3)	0.012 (4)
O3	0.070 (4)	0.068 (4)	0.029 (3)	−0.017 (3)	−0.012 (3)	0.007 (3)
O4	0.070 (4)	0.060 (4)	0.038 (4)	−0.012 (3)	−0.006 (3)	0.002 (3)
O5	0.067 (4)	0.090 (5)	0.035 (4)	0.015 (4)	0.002 (3)	−0.002 (4)
O6	0.065 (4)	0.102 (6)	0.048 (4)	0.026 (4)	−0.003 (3)	−0.011 (4)

Geometric parameters (Å, º)

C9—O2	1.223 (11)	C12—N1	1.451 (11)
C9—O1	1.369 (11)	C12—C13	1.490 (11)
C9—C8	1.433 (12)	C12—H12A	0.9700
C1—C2	1.350 (13)	C12—H12B	0.9700
C1—C6	1.394 (11)	C13—C14	1.391 (12)
C1—O1	1.399 (11)	C13—C18	1.400 (11)
C2—C3	1.381 (14)	C14—C15	1.367 (12)
C2—H2	0.9300	C14—H14	0.9300
C3—C4	1.389 (12)	C15—C16	1.386 (12)
C3—H3	0.9300	C15—H15	0.9300
C4—C5	1.343 (13)	C16—C17	1.372 (12)
C4—H4	0.9300	C16—O6	1.379 (10)
C5—C6	1.417 (12)	C17—C18	1.382 (11)
C5—H5	0.9300	C17—H17	0.9300
C6—C7	1.398 (12)	C18—O5	1.369 (10)
C7—C8	1.352 (12)	C19—O5	1.428 (11)
C7—O3	1.373 (9)	C19—H19A	0.9600
C8—H8	0.9300	C19—H19B	0.9600
C10—O3	1.399 (10)	C19—H19C	0.9600
C10—C11	1.516 (11)	C20—O6	1.421 (11)

C10—H10A	0.9700	C20—H20A	0.9600
C10—H10B	0.9700	C20—H20B	0.9600
C11—O4	1.239 (10)	C20—H20C	0.9600
C11—N1	1.315 (11)	N1—H1	0.8600
O2—C9—O1	115.5 (8)	C13—C12—H12B	108.3
O2—C9—C8	125.3 (10)	H12A—C12—H12B	107.4
O1—C9—C8	119.0 (9)	C14—C13—C18	116.6 (7)
C2—C1—C6	123.5 (9)	C14—C13—C12	124.8 (8)
C2—C1—O1	117.0 (7)	C18—C13—C12	118.5 (7)
C6—C1—O1	119.4 (8)	C15—C14—C13	123.2 (9)
C1—C2—C3	118.7 (8)	C15—C14—H14	118.4
C1—C2—H2	120.6	C13—C14—H14	118.4
C3—C2—H2	120.6	C14—C15—C16	118.7 (8)
C2—C3—C4	119.9 (9)	C14—C15—H15	120.7
C2—C3—H3	120.0	C16—C15—H15	120.7
C4—C3—H3	120.0	C17—C16—O6	123.4 (7)
C5—C4—C3	120.8 (10)	C17—C16—C15	120.3 (8)
C5—C4—H4	119.6	O6—C16—C15	116.4 (7)
C3—C4—H4	119.6	C16—C17—C18	120.3 (8)
C4—C5—C6	121.1 (8)	C16—C17—H17	119.9
C4—C5—H5	119.5	C18—C17—H17	119.9
C6—C5—H5	119.5	O5—C18—C17	124.4 (8)
C1—C6—C7	118.6 (8)	O5—C18—C13	114.6 (7)
C1—C6—C5	116.0 (8)	C17—C18—C13	120.9 (8)
C7—C6—C5	125.4 (7)	O5—C19—H19A	109.5
C8—C7—O3	121.9 (8)	O5—C19—H19B	109.5
C8—C7—C6	122.6 (7)	H19A—C19—H19B	109.5
O3—C7—C6	115.4 (7)	O5—C19—H19C	109.5
C7—C8—C9	118.9 (9)	H19A—C19—H19C	109.5
C7—C8—H8	120.6	H19B—C19—H19C	109.5
C9—C8—H8	120.6	O6—C20—H20A	109.5
O3—C10—C11	110.6 (7)	O6—C20—H20B	109.5
O3—C10—H10A	109.5	H20A—C20—H20B	109.5
C11—C10—H10A	109.5	O6—C20—H20C	109.5
O3—C10—H10B	109.5	H20A—C20—H20C	109.5
C11—C10—H10B	109.5	H20B—C20—H20C	109.5
H10A—C10—H10B	108.1	C11—N1—C12	121.3 (7)
O4—C11—N1	123.7 (7)	C11—N1—H1	119.4
O4—C11—C10	117.5 (8)	C12—N1—H1	119.4
N1—C11—C10	118.8 (8)	C9—O1—C1	121.4 (7)
N1—C12—C13	115.9 (8)	C7—O3—C10	118.0 (6)
N1—C12—H12A	108.3	C18—O5—C19	117.5 (7)
C13—C12—H12A	108.3	C16—O6—C20	117.3 (7)
N1—C12—H12B	108.3		
C6—C1—C2—C3	0.3 (16)	C13—C14—C15—C16	−0.7 (17)
O1—C1—C2—C3	179.9 (9)	C14—C15—C16—C17	−0.7 (16)

C1—C2—C3—C4	−0.9 (17)	C14—C15—C16—O6	179.7 (10)
C2—C3—C4—C5	1.1 (17)	O6—C16—C17—C18	−178.1 (9)
C3—C4—C5—C6	−0.8 (17)	C15—C16—C17—C18	2.2 (15)
C2—C1—C6—C7	−179.6 (10)	C16—C17—C18—O5	179.9 (9)
O1—C1—C6—C7	0.8 (13)	C16—C17—C18—C13	−2.5 (15)
C2—C1—C6—C5	0.0 (14)	C14—C13—C18—O5	179.0 (8)
O1—C1—C6—C5	−179.6 (9)	C12—C13—C18—O5	−3.5 (12)
C4—C5—C6—C1	0.3 (15)	C14—C13—C18—C17	1.1 (13)
C4—C5—C6—C7	179.8 (10)	C12—C13—C18—C17	178.6 (9)
C1—C6—C7—C8	−0.6 (14)	O4—C11—N1—C12	−3.4 (13)
C5—C6—C7—C8	179.9 (10)	C10—C11—N1—C12	178.1 (8)
C1—C6—C7—O3	−177.3 (8)	C13—C12—N1—C11	83.3 (11)
C5—C6—C7—O3	3.2 (14)	O2—C9—O1—C1	177.8 (9)
O3—C7—C8—C9	177.7 (9)	C8—C9—O1—C1	2.3 (15)
C6—C7—C8—C9	1.1 (14)	C2—C1—O1—C9	178.7 (10)
O2—C9—C8—C7	−177.0 (11)	C6—C1—O1—C9	−1.7 (13)
O1—C9—C8—C7	−2.0 (15)	C8—C7—O3—C10	−7.3 (12)
O3—C10—C11—O4	164.9 (8)	C6—C7—O3—C10	169.5 (8)
O3—C10—C11—N1	−16.4 (11)	C11—C10—O3—C7	−160.9 (7)
N1—C12—C13—C14	2.3 (14)	C17—C18—O5—C19	−2.8 (13)
N1—C12—C13—C18	−175.0 (8)	C13—C18—O5—C19	179.4 (10)
C18—C13—C14—C15	0.5 (15)	C17—C16—O6—C20	1.9 (14)
C12—C13—C14—C15	−176.8 (10)	C15—C16—O6—C20	−178.4 (11)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3	0.86	2.31	2.669 (2)	105
C14—H14···N1	0.93	2.59	2.923 (2)	101
N1—H1···O4[i]	0.86	2.09	2.900 (2)	156
C3—H3···O5[ii]	0.93	2.49	3.419 (2)	175
C5—H5···O4[i]	0.93	2.43	3.307 (2)	157
C15—H15···O4[iii]	0.93	2.51	3.399 (2)	160

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