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Polarized molecular–electronic structures and supramolecular aggregation in 1-(6-amino-1,3-benzodioxol-5-yl)-3-arylprop-2-en-1-ones

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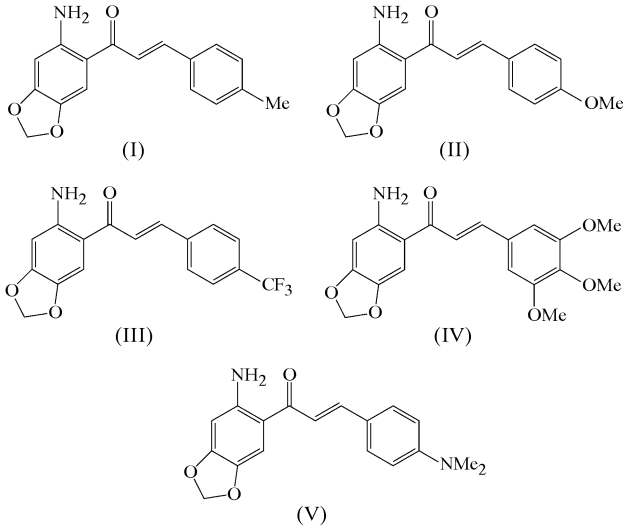
Molecules of 1-(6-amino-1,3-benzodioxol-5-yl)-3-(4-methylphenyl)prop-2-en-1-one, $C_{17}H_{15}NO_3$, (I), 1-(6-amino-1,3-benzodioxol-5-yl)-3-(4-methoxyphenyl)prop-2-en-1-one, $C_{17}H_{15}NO_4$, (II), and 1-(6-amino-1,3-benzodioxol-5-yl)-3-[4-(trifluoromethyl)phenyl]prop-2-en-1-one, $C_{17}H_{12}F_3NO_3$, (III), all contain an intramolecular N—H···O hydrogen bond and all exhibit polarized molecular–electronic structures. The molecules of (I) are linked into simple sheets, generated by translation, by means of one N—H···O and one C—H···π(arene) hydrogen bond. Compound (II) crystallizes as two concomitant polymorphs, *viz*. (II*a*), with $Z' = 1$ in $P2_1/c$, and (II*b*), with $Z' = 2$ in $P\overline{1}$. In (II*a*), intra- and intermolecular N—H···O hydrogen bonds generate a helical chain of rings, and these chains are linked into sheets by simple helical chains built from a C—H···π(arene) hydrogen bond, while in (II*b*), the molecules are linked into simple chains by a C—H···O hydrogen bond. In (III), where $Z' = 2$, each type of molecule forms a simple N—H···O hydrogen-bonded chain generated by translation and the two types of chain are linked by a single π–π stacking interaction.

Comment

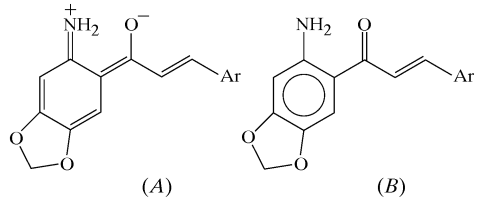
A range of 2-aminochalcone derivatives have been prepared for use as intermediates in the synthesis of new 6,7-methylenedioxytetrahydroquinolin-4-ones, compounds with interesting biological and pharmacological properties (Donnelly & Farell, 1990; Prager & Thredgold, 1968; Kurasawa *et al.*, 2002). We report here the molecular and supramolecular structures of three such compounds, (I)–(III), and compare them with two further examples, (IV) and (V) (Low *et al.*, 2002).

Compounds (I) and (III) crystallize with Z' values of 1 and 2, respectively, while compound (II) forms two polymorphs, *viz*. monoclinic and triclinic, denoted (II*a*) and (II*b*), respectively, which crystallize concomitantly from dimethylformamide, with Z' values of 1 and 2, respectively (Figs. 1–4). Of the two polymorphs of (II), the monoclinic polymorph has a significantly higher density than the triclinic polymorph and hence is probably the thermodynamically more stable form (Burger & Ramberger, 1979).



(I) (II) (III) (IV) (V)

There is significant bond fixation within the amino-substituted aryl rings of (I)–(III) (Table 1). In particular, the C3a—C4 and C7—C7a bonds are both short, while the C5—C6 and C6—C7 bonds are long. In addition, the C6—C8 bond is short for its type (mean value 1.488 Å; Allen *et al.*, 1987), while C8—O8 is long (mean value 1.231 Å). These values point to the charge-separated form, (*A*) (see scheme below), as an important contributor to the overall molecular–electronic structure, alongside the delocalized form, (*B*). An entirely similar pattern of distances (Table 1) is observed in the analogous compounds (IV) and (V), the structures of which have recently been reported (Low *et al.*, 2002), although this was not discussed or noted in the original report, which focused exclusively on the supramolecular aggregation of (IV) and (V).



(*A*) (*B*)

In all cases, the molecular skeletons are fairly close to being planar but, as shown by the key torsion angles (Table 2), there are some significant deviations in most of the independent examples. The sole exception is the type 1 molecule (containing atom O11, *etc*.; Fig. 3*a*) of compound (III). The five-membered rings show some flexibility of conformational behaviour. Thus, this ring is planar in (II*a*) [although not in (II*b*)] and in the type 2 molecule of compound (III), but it

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adopts an envelope conformation, with a folding across the O···O line, in (I), in both molecules of (II*b*) and in the type 1 molecule of (III). For these rings, the ring-puckering parameter φ_2 (Cremer & Pople, 1975) takes the values 31.1 (15) and 30.5 (9)° in (I) and (III), respectively, and 30.9 (5) and 213.7 (5)° in the two independent molecules of (II*b*). The two independent molecules in (II*b*) exhibit different conformations at the methoxy substituent (Table 2), and this alone is sufficient to preclude the possibility of any additional symmetry

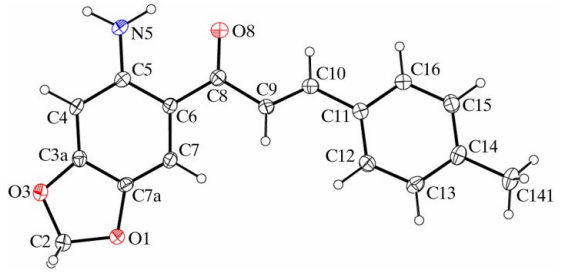

Figure 1
The molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

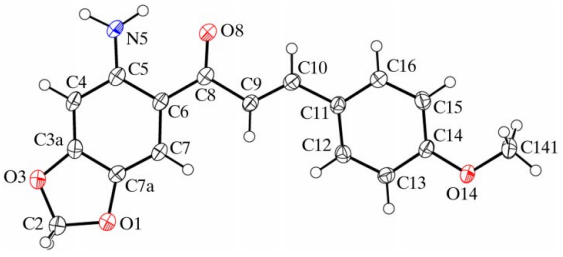

Figure 2
The molecule of (II*a*), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

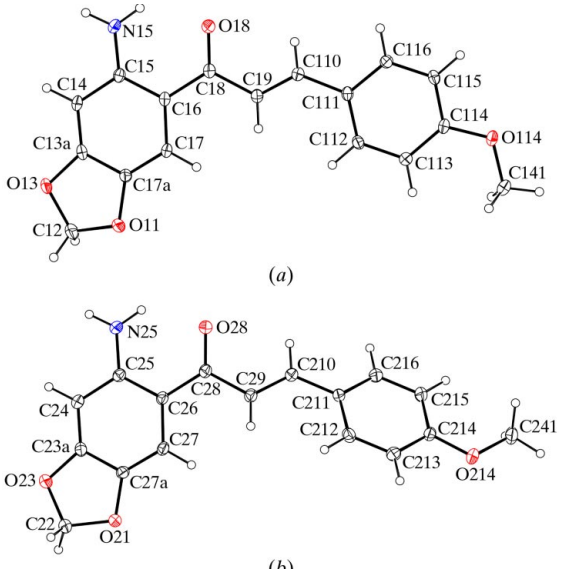

Figure 3
The two independent molecules of (II*b*), showing the atom-labelling scheme for (*a*) the type 1 molecule and (*b*) the type 2 molecule. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

All of the molecules contain an intramolecular N—H···O hydrogen bond (Tables 3–6), in each case generating an *S*(6) motif (Bernstein *et al.*, 1995), and these may have some influence on the overall molecular conformations. The supramolecular structures of (I) and (II*a*) both depend upon a combination of N—H···O and C—H···π(arene) hydrogen bonds to generate sheets, but the structures differ considerably in detail. In compound (I), the amine atom N5 in the molecule at (*x*, *y*, *z*) acts as hydrogen-bond donor, *via* atom H5*B*, to ring atom O1 in the molecule at (*x*, *y* − 1, *z*), so generating by translation a *C*(7) chain running parallel to the

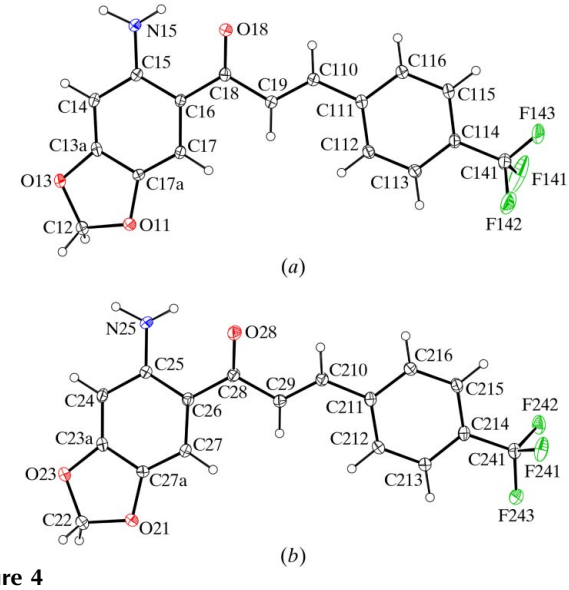

Figure 4
The two independent molecules of (III), showing the atom-labelling scheme for (*a*) the type 1 molecule and (*b*) the type 2 molecule. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

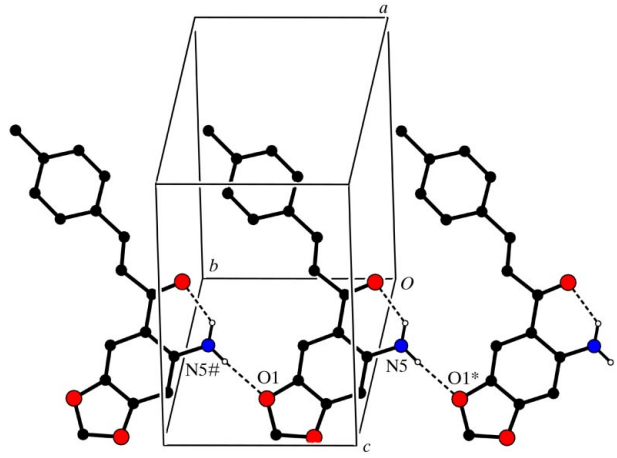

Figure 5
Part of the crystal structure of (I), showing the formation of a chain parallel to [010]. For the sake of clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions (*x*, *y* − 1, *z*) and (*x*, 1 + *y*, *z*), respectively.

[010] direction (Fig. 5). In addition, atom C2 in the molecule at (x, y, z) acts as hydrogen-bond donor, *via* atom H2*A*, to the C11–C16 ring in the molecule at ($x-1$, y, z), so generating by translation a chain running parallel to the [100] direction (Fig. 6). The combination of the [100] and [010] chains generates a sheet parallel to (001), lying in the domain $\frac{1}{2} < z < \frac{3}{4}$ (Fig. 7). Four sheets of this type pass through each unit cell, but there are no direction-specific interactions between adjacent sheets.

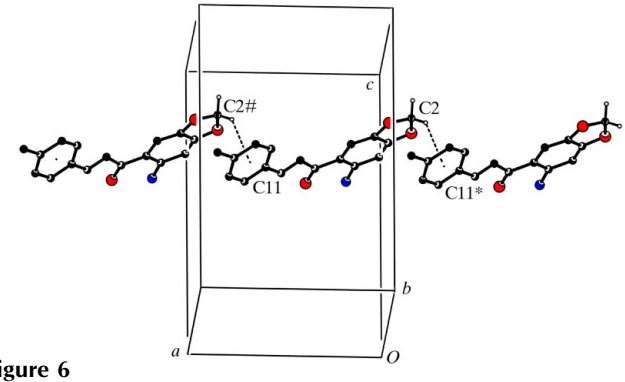

Figure 6
Part of the crystal structure of (I), showing the formation of a chain parallel to [100]. For the sake of clarity, H atoms bonded to C atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions ($x-1$, y, z) and ($1+x$, y, z), respectively.

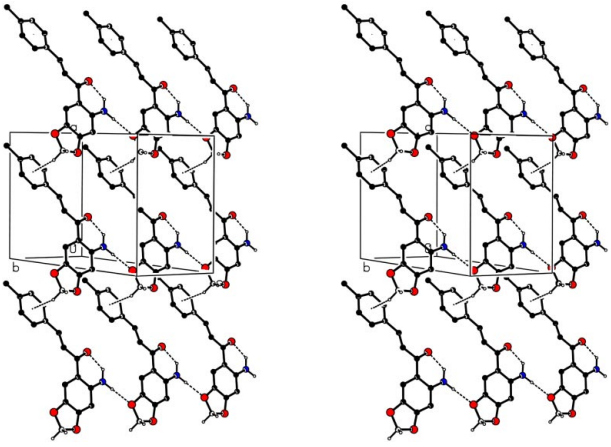

Figure 7
A stereoview of part of the crystal structure of (I), showing the formation of a sheet parallel to (001). For the sake of clarity, H atoms bonded to C atoms not involved in the motif shown have been omitted.

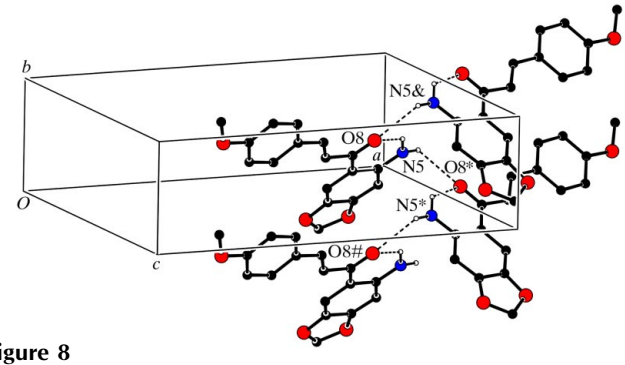

Figure 8
Part of the crystal structure of polymorph (II*a*), showing the formation of a chain of rings parallel to [010]. For the sake of clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*), a hash (#) or an ampersand (&) are at the symmetry positions ($2-x$, $y-\frac{1}{2}$, $\frac{1}{2}-z$), (x, $y-1$, z) and ($2-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$), respectively.

The monoclinic polymorph (II*a*) of compound (II) exhibits two C—H$\cdots\pi$(arene) hydrogen bonds in addition to the two N—H$\cdots$O interactions (Table 4). Amine atom N5 in the molecule at (x, y, z) acts as donor, again *via* atom H5*B*, but this time to carbonyl atom O8 in the molecule at ($2-x$, $y-\frac{1}{2}$, $\frac{1}{2}-z$), so producing a helical $C_2^1(4)C(6)[S(6)]$ chain of rings running parallel to the [010] direction and generated by the 2_1 screw axis along (1, y, $\frac{1}{4}$) (Fig. 8). This chain of rings may be contrasted with the very simple chain formed by the N—H$\cdots$O hydrogen bonds in compound (I) (Fig. 5). Of the two C—H$\cdots\pi$(arene) hydrogen bonds, that having atom C2 as the donor simply reinforces the foregoing [010] chain. However, that involving atom C13 in the molecule at (x, y, z) as donor to

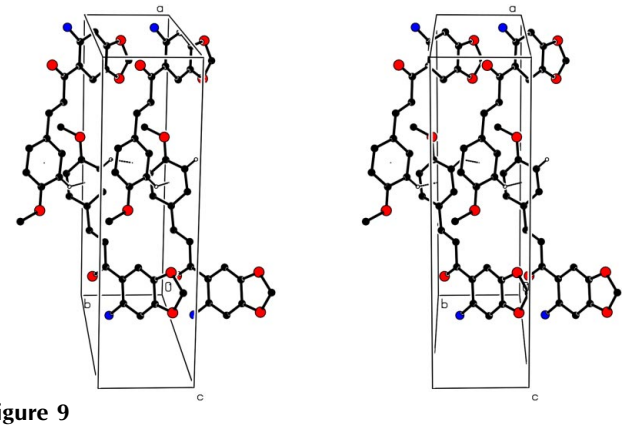

Figure 9
A stereoview of part of the crystal structure of polymorph (II*a*), showing the formation of an [010] chain generated by C—H$\cdots\pi$(arene) hydrogen bonds. For the sake of clarity, H atoms not involved in the motif shown have been omitted.

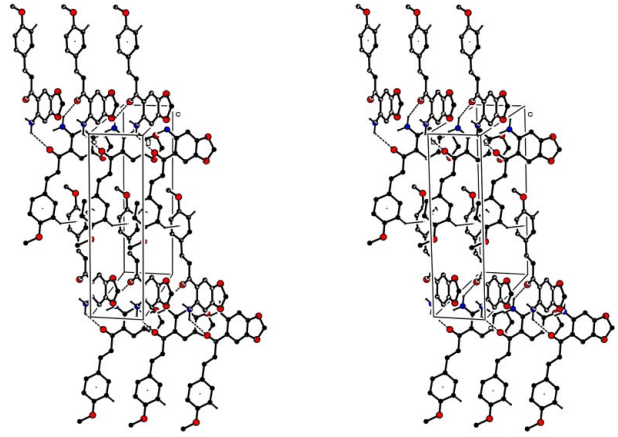

Figure 10
A stereoview of part of the crystal structure of polymorph (II*a*), showing the formation of a sheet parallel to (001). For the sake of clarity, the intramolecular hydrogen bond and H atoms bonded to C atoms and not involved in the motif shown have been omitted.

the C11–C16 ring in the molecule at $(1-x, y-\frac{1}{2}, \frac{1}{2}-z)$ not only generates a second chain running parallel to [010], this time generated by the 2_1 axis along $(\frac{1}{2}, y, \frac{1}{4})$ (Fig. 9), but also serves to link all of the chain of rings into an (001) sheet (Fig. 10). In the triclinic polymorph (II*b*), the type 1 molecules (Fig. 3*a*) are linked by means of a single C—H···O hydrogen bond into chains generated by translation, while the type 2 molecules (Fig. 3*b*) are pendent from these chains and linked to them by N—H···O hydrogen bonds (Fig. 11)

Each of the two independent molecules in compound (III) forms a simple *C*(7) chain. The amine atoms N15 and N25 in the molecules at (x, y, z) act as donors to, respectively, the ring atoms O11 and O21 in the molecules at $(x-1, y, z)$, so generating *C*(7) chains by translation (Table 5 and Fig. 12). These two chains are linked by an aromatic π–π stacking interaction between the C111–C116 and C211–C216 rings within the asymmetric unit. The dihedral angle between the planes of these two rings is only 4.5 (2)°, the interplanar spacing is *ca* 3.5 Å and the centroid–centroid separation is 3.618 (2) Å. Propagation of this interaction then links the two independent translational chains (Fig. 12)

The simple and complex sheets in (I) and (II*a*), the single chains in (II*b*) and the paired chains in (III) may be briefly compared with the supramolecular structures of the analogues (IV) and (V) (Low *et al.*, 2002). In (IV), where $Z' = 1$, the sole significant intermolecular interactions are a C—H···O hydrogen bond with a ring O atom as acceptor, which generates zigzag *C*(10) chains, and a π–π stacking interaction linking these chains into sheets. In (V), where $Z' = 2$, two N—H···O hydrogen bonds generate centrosymmetric $R_8^4(16)$ tetramers, which are weakly linked into chains by two rather long C—H···O hydrogen bonds. Hence, for the five compounds (I)–(V), while their intramolecular properties are all very similar, their supramolecular aggregation patterns are all different. For no single example in this series could the supramolecular structure be predicted from a knowledge of the supramolecular structures of all the others.

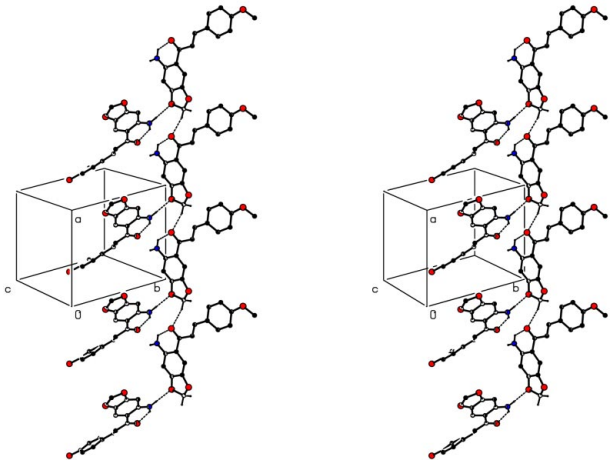

Figure 11
A stereoview of part of the crystal structure of polymorph (II*b*), showing the formation of a *C*(8) chain along [100]. For the sake of clarity, the intramolecular hydrogen bond and H atoms bonded to C atoms and not involved in the motif shown have been omitted.

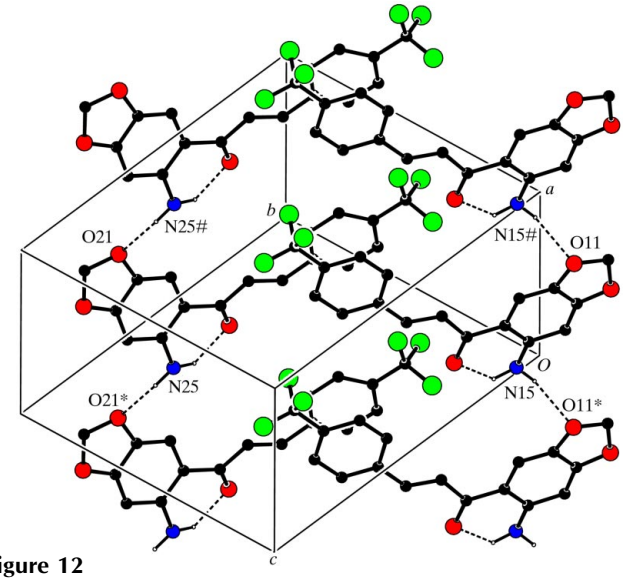

Figure 12
Part of the crystal structure of (III), showing the formation of a π-stacked pair of chains parallel to [100]. For the sake of clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(x-1, y, z)$ and $(1+x, y, z)$, respectively.

Experimental

For the synthesis of (I), a solution of 6-amino-3,4-methylenedioxyacetophenone (0.5 g, 2.79 mmol), 4-tolualdehyde (0.33 g, 2.75 mmol), ethanol (10 ml) and aqueous NaOH (0.5 ml, 20%) was heated under reflux for 20 min. After cooling the mixture, the resulting precipitate was filtered off and washed with ethanol, yielding (I) as a yellow solid (yield 91%, m.p. 401 K). Spectroscopic analysis, IR (KBr disc, ν, cm^{-1}): 3454, 3278 (NH_2), 1646 (C═O), 1606 (C═C), 1224 (OCH_2O); ^{1}H NMR (DMSO-d_6, δ): 2.33 (3H, *s*, CH_3), 5.96 (2H, *s*, OCH_2O), 6.35 (1H, *s*), 7.23 (2H, *d*, J = 8.0 Hz), 7.53 (1H, *d*, J = 15.4 Hz), 7.65 (1H, *s*), 7.67 (2H, *br s*, NH_2), 7.73 (2H, *d*, J = 8.0 Hz), 7.81 (1H, *d*, J = 15.4 Hz); ^{13}C NMR (DMSO-d_6, δ): 21.0 (CH_3), 95.8, 101.1 (OCH_2O), 108.0, 109.9, 122.7, 128.5, 129.4, 132.5, 137.7, 139.6, 141.0, 151.7, 152.7, 187.7 (C═O). MS (70 eV): *m*/e (%) 281 (41, [M^+]), 190 (100, [M–C_7H_7]). Crystals of (I) suitable for single-crystal X-ray diffraction were grown from a solution in ethanol. For the synthesis of (II), a solution of 6-amino-3,4-methylenedioxyacetophenone (0.5 g, 2.79 mmol), 4-methoxybenzaldehyde (0.38 g, 2.79 mmol), ethanol (10 ml) and aqueous NaOH (0.5 ml, 20%) was heated under reflux for 30 min. After cooling the mixture, the resulting precipitate was filtered off and crystallized from ethanol, giving (II) as an orange solid (yield 50%, m.p. 405 K). Spectroscopic analysis, IR (KBr disc, ν, cm^{-1}): 3461, 3303 (NH_2), 1644 (C═O), 1603 (C═C), 1223 (OCH_2O); ^{1}H NMR ($CDCl_3$, δ): 3.89 (3H, *s*, OCH_3), 5.93 (2H, *s*, OCH_2O), 6.19 (1H, *s*), 6.57 (2H, *br s*, NH_2), 6.91 (2H, *d*, J = 8.0 Hz), 7.26 (1H, *s*), 7.35 (1H, *d*, J = 15.4 Hz), 7.47 (2H, *d*, J = 8.0 Hz), 7.71 (1H, *d*, J = 15.4 Hz); ^{13}C NMR ($CDCl_3$, δ): 55.2 (OCH_3), 96.8, 101.5 (OCH_2O), 108.2, 112.0, 114.5, 121.2, 128.3, 130.0, 138.9, 142.1, 150.0, 153.5, 161.2, 189.0 (C═O). MS (70 eV): *m*/e (%) 297 (27, [M^+]), 190 (100, [M–C_7H_7O]). Crystallization from dimethylformamide gave a mixture of the monoclinic polymorph (II*a*) as red crystals (m.p. 382 K) and the triclinic polymorph (II*b*) as yellow crystals (m.p. 389 K). For the

Table 1
Selected bond distances (Å) for compounds (I)–(V).

Bond	(I)	(II*a*)	(II*b*) Mol 1	(II*b*) Mol 2	(III) Mol 1	(III) Mol 2	(IV)	(V) Mol 1	(V) Mol 2
x	nil	nil	1	2	1	2	nil	1	2
C*x*3a—C*x*4	1.368 (3)	1.357 (2)	1.357 (2)	1.359 (2)	1.358 (3)	1.355 (3)	1.361 (2)	1.358 (2)	1.351 (2)
C*x*4—C*x*5	1.402 (3)	1.416 (2)	1.418 (2)	1.418 (2)	1.415 (3)	1.417 (3)	1.417 (2)	1.419 (2)	1.418 (2)
C*x*5—C*x*6	1.424 (3)	1.430 (2)	1.423 (2)	1.422 (2)	1.429 (3)	1.419 (3)	1.426 (2)	1.426 (2)	1.431 (2)
C*x*6—C*x*7	1.428 (3)	1.430 (2)	1.420 (2)	1.435 (2)	1.428 (3)	1.421 (3)	1.428 (2)	1.426 (2)	1.423 (2)
C*x*7—C*x*7a	1.339 (3)	1.354 (2)	1.353 (2)	1.350 (2)	1.350 (3)	1.355 (3)	1.355 (2)	1.354 (2)	1.353 (2)
C*x*7a—C*x*3a	1.386 (3)	1.394 (2)	1.387 (2)	1.391 (2)	1.390 (3)	1.390 (3)	1.394 (2)	1.390 (2)	1.393 (2)
C*x*5—N*x*5	1.364 (3)	1.353 (2)	1.364 (2)	1.361 (2)	1.368 (2)	1.369 (3)	1.359 (2)	1.370 (2)	1.360 (2)
C*x*6—C*x*8	1.459 (3)	1.461 (2)	1.470 (2)	1.462 (2)	1.468 (2)	1.468 (3)	1.470 (2)	1.473 (2)	1.463 (2)
C*x*8-O*x*8	1.249 (3)	1.246 (2)	1.240 (2)	1.244 (2)	1.244 (4)	1.237 (2)	1.243 (2)	1.253 (2)	1.250 (2)

Table 2
Selected torsion angles (°) for compounds (I)–(III).

Parameter	(I)	(II*a*)	(II*b*) Mol 1	(II*b*) Mol 2	(III) Mol 1	(III) Mol 2
x	nil	nil	1	2	1	2
C*x*5—C*x*6—C*x*8—C*x*9	−177.8 (2)	179.63 (13)	−168.15 (13)	−175.23 (13)	−171.93 (18)	−156.36 (19)
C*x*6—C*x*8—C*x*9—C*x*10	−159.1 (2)	−175.03 (14)	−170.48 (13)	153.13 (14)	171.9 (2)	176.7 (2)
C*x*8—C*x*9—C*x*10—C*x*11	178.4 (2)	179.77 (14)	−178.12 (13)	−176.05 (13)	−177.59 (19)	−177.65 (19)
C*x*9—C*x*10—C*x*11—C*x*12	−9.0 (2)	5.1 (2)	−11.3 (2)	−16.8 (2)	0.7 (3)	−8.6 (3)
C*x*13—C*x*14—O*x*14—C*x*41		−175.91 (13)	1.4 (2)	−176.09 (14)		

synthesis of (III), a solution of 6-amino-3,4-methylenedioxyacetophenone (0.5 g, 2.79 mmol), 4-(trifluoromethyl)benzaldehyde (0.49 g, 2.79 mmol), ethanol (10 ml) and aqueous NaOH (0.5 ml, 20%) was heated under reflux for 25 min. After cooling the mixture, the resulting precipitate was filtered off and washed with ethanol, yielding (III) as an orange solid (yield 75%, m.p. 417 K). Spectroscopic analysis, IR (KBr disc, ν, cm^{-1}): 3468, 3305 (NH_2), 1646 (C═O), 1606 (C═C), 1228 (OCH_2O); ^{1}H NMR (DMSO-d_6, δ): 5.94 (1H, *s*, H2), 5.98 (2H, *s*, OCH_2O), 6.38 (1H, *s*, H6), 7.6 (1H, *d*, H8, J = 15.0 Hz), 7.69 (2H, *br s*, NH_2), 7.76 (2H, *d*, J = 8.0 Hz), 8.02 (1H, *d*, J = 15.4 Hz), 8.15 (2H, *d*, J = 8.0 Hz); ^{13}C NMR (DMSO-d_6, δ): 95.8, 101.2 (OCH_2O), 108.0, 108.9, 113.5 (CF_3), 125.5, 126.7, 129.3, 138.0, 139.1, 139.4, 152.2, 153.2, 187.1 (C═O). MS (70 eV): *m*/e (%) 335 (100, [M^+]). Crystals of (III) suitable for single-crystal X-ray diffraction were grown from a solution in ethanol.

Compound (I)

Crystal data

$C_{17}H_{15}NO_3$
M_r = 281.30
Monoclinic, $P2_1/n$
a = 10.530 (5) Å
b = 7.362 (5) Å
c = 17.546 (5) Å
β = 91.719 (5)°
V = 1359.6 (12) Å^3
Z = 4
D_x = 1.374 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3062 reflections
θ = 5.3–27.5°
μ = 0.10 mm^{-1}
T = 120 (2) K
Block, yellow
0.40 × 0.30 × 0.20 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*EvalCCD*; Duisenberg *et al.*, 2003)
T_{min} = 0.958, T_{max} = 0.981
17 073 measured reflections
3062 independent reflections
2024 reflections with $I > 2\sigma(I)$
R_{int} = 0.065
θ_{max} = 27.5°
$h = -13 \rightarrow 12$
$k = -9 \rightarrow 9$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.135
S = 1.11
3062 reflections
191 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0198P)^2 + 1.4934P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 3
Hydrogen-bonding geometry (Å, °) for (I).

*Cg*1 is the centroid of the C11–C16 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5*A*···O8	0.96	1.88	2.612 (3)	131
N5—H5*B*···O1^{i}	0.96	2.07	3.032 (3)	178
C2—H2*A*···*Cg*1^{ii}	0.99	2.86	3.644 (4)	137

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

Polymorph (II*a*)

Crystal data

$C_{17}H_{15}NO_4$
M_r = 297.30
Monoclinic, $P2_1/c$
a = 17.5560 (4) Å
b = 5.0914 (2) Å
c = 15.4869 (4) Å
β = 91.9240 (16)°
V = 1383.51 (7) Å^3
Z = 4
D_x = 1.427 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3170 reflections
θ = 2.9–27.5°
μ = 0.10 mm^{-1}
T = 120 (2) K
Block, red
0.04 × 0.02 × 0.02 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.959, T_{max} = 0.998
14 325 measured reflections
3170 independent reflections
2271 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 27.5°
$h = -21 \rightarrow 22$
$k = -6 \rightarrow 6$
$l = -20 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0701P)^2 + 0.2374P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.045$	$(\Delta/\sigma)_{max} = 0.002$
$wR(F^2) = 0.128$	$\Delta\rho_{max} = 0.26$ e Å^{-3}
$S = 1.04$	$\Delta\rho_{min} = -0.21$ e Å^{-3}
3169 reflections	Extinction correction: *SHELXL*97 (Sheldrick, 1997)
201 parameters	Extinction coefficient: 0.007 (2)
H-atom parameters constrained	

Table 4
Hydrogen-bonding geometry (Å, °) for polymorph (II*a*).

*Cg*1 is the centroid of the C11–C16 ring and *Cg*2 is the centroid of the C3a/C4–C7/C7a ring.

D—H···A	D—H	H···A	D···A	D—H···A
N5—H5*A*···O8	0.96	1.95	2.6301 (15)	126
N5—H5*B*···O8^{i}	0.96	2.49	3.1232 (15)	123
C2—H2*B*···*Cg*2^{ii}	0.99	2.84	3.640 (2)	138
C13—H13···*Cg*1^{iii}	0.95	2.81	3.488 (2)	130

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

Polymorph (II*b*)

Crystal data

$C_{17}H_{15}NO_4$	Mo $K\alpha$ radiation
$M_r = 297.30$	Cell parameters from 6517 reflections
Triclinic, $P\overline{1}$	$\theta = 3.1$–$27.5°$
$a = 9.5352$ (2) Å	$\mu = 0.10$ mm^{-1}
$b = 10.6193$ (3) Å	$T = 120$ (2) K
$c = 14.7611$ (4) Å	Block, yellow
$\alpha = 89.1400$ (14)°	0.45 × 0.30 × 0.20 mm
$\beta = 81.0970$ (17)°	
$\gamma = 75.7540$ (14)°	
$V = 1430.83$ (6) Å^3	
$Z = 4$	
$D_x = 1.380$ Mg m^{-3}	

Data collection

Nonius KappaCCD area-detector diffractometer	4940 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.035$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	$\theta_{max} = 27.5°$
$T_{min} = 0.951$, $T_{max} = 0.981$	$h = -12 \rightarrow 12$
23 999 measured reflections	$k = -13 \rightarrow 13$
6517 independent reflections	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0778P)^2 + 0.1431P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.049$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.137$	$\Delta\rho_{max} = 0.30$ e Å^{-3}
$S = 1.10$	$\Delta\rho_{min} = -0.37$ e Å^{-3}
6512 reflections	
399 parameters	
H-atom parameters constrained	

Table 5
Hydrogen-bonding geometry (Å, °) for polymorph (II*b*).

D—H···A	D—H	H···A	D···A	D—H···A
N15—H15*B*···O18	0.88	1.99	2.6451 (17)	130
N25—H25*A*···O28	0.88	1.95	2.6069 (19)	131
N25—H25*B*···O13	0.88	2.19	3.0586 (17)	170
C12—H12*A*···O18^{i}	0.99	2.27	3.221 (2)	161

Symmetry code: (i) $x-1, y, z$.

Compound (III)

Crystal data

$C_{17}H_{12}F_3NO_3$	$Z = 4$
$M_r = 335.28$	$D_x = 1.544$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 7.3420$ (2) Å	Cell parameters from 6611 reflections
$b = 10.9241$ (3) Å	$\theta = 3.0$–$27.6°$
$c = 18.7176$ (5) Å	$\mu = 0.13$ mm^{-1}
$\alpha = 85.0180$ (11)°	$T = 120$ (2) K
$\beta = 83.2280$ (14)°	Block, red
$\gamma = 75.8180$ (14)°	0.60 × 0.60 × 0.50 mm
$V = 1442.71$ (7) Å^3	

Data collection

Nonius KappaCCD area-detector diffractometer	6611 independent reflections
φ and ω scans	4510 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	$R_{int} = 0.038$
$T_{min} = 0.920$, $T_{max} = 0.937$	$\theta_{max} = 27.6°$
20 172 measured reflections	$h = -9 \rightarrow 6$
	$k = -14 \rightarrow 13$
	$l = -24 \rightarrow 24$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0874P)^2 + 0.3568P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.052$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.157$	$\Delta\rho_{max} = 0.77$ e Å^{-3}
$S = 1.04$	$\Delta\rho_{min} = -0.52$ e Å^{-3}
6611 reflections	
433 parameters	
H-atom parameters constrained	

Table 6
Hydrogen-bonding geometry (Å, °) for (III).

D—H···A	D—H	H···A	D···A	D—H···A
N15—H15*A*···O18	0.96	1.91	2.637 (2)	131
N15—H15*B*···O11^{i}	0.96	2.26	3.161 (2)	156
N25—H25*A*···O28	0.96	1.91	2.662 (2)	134
N25—H25*B*···O21^{i}	0.96	2.09	3.024 (2)	165

Symmetry code: (i) $x-1, y, z$.

For (I) and (II*a*), the space groups $P2_1/n$ and $P2_1/c$, respectively, were uniquely determined from the systematic absences. Crystals of (II*b*) and (III) are triclinic, and the space group $P\overline{1}$ was selected and then confirmed by the structure analysis. All H atoms were located from difference maps and subsequently treated as riding atoms, with C—H = 0.95 (CH), 0.98 (CH_3) or 0.99 Å (CH_2) and N—H = 0.96 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N), or 1.5U_{eq}(C) for the methyl groups. In compound (III), the highest residual peak (0.77 e Å^{-3}) is 1.26 Å from F141 and the deepest hole (−0.52 e Å^{-3}) is 0.97 Å from F141. Careful inspection of electron-density maps indicated some libration of the CF_3 groups about the adjacent C—C bonds, but gave no grounds for modelling these groups with more than three F-atom sites per group.

For all four compounds, data collection: *COLLECT* (Nonius, 1998). For compound (I), cell refinement: *DIRAX*/*LSQ* (Duisenberg *et al.*, 2000); data reduction: *EvalCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *OSCAIL* (McArdle, 2003) and *SHELXL*97 (Sheldrick, 1997). For compounds (II*a*) and (III), cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97. For compound (II*b*), cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* and *SHELXS*97;

program(s) used to refine structure: *OSCAIL* and *SHELXL*97. For all four compounds, molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PRPKAPPA* (Ferguson, 1999).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1762). Services for accessing these data are described at the back of the journal.

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Polarized molecular–electronic structures and supramolecular aggregation in 1-(6-amino-1,3-benzodioxol-5-yl)-3-arylprop-2-en-1-ones

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Computing details

For all compounds, data collection: *COLLECT* (Nonius, 1998). Cell refinement: *DIRAX/LSQ* (Duisenberg *et al.*, 2000) for (I); *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* for (IIa), (IIb), (III). Data reduction: *EVALCCD* (Duisenberg *et al.*, 2003) for (I); *DENZO* and *COLLECT* for (IIa), (IIb), (III). Program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999) for (I); *SHELXS97* (Sheldrick, 1997) for (IIa), (III); *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997) for (IIb). Program(s) used to refine structure: *OSCAIL* (McArdle, 2003) and *SHELXL97* (Sheldrick, 1997) for (I); *SHELXL97* (Sheldrick, 1997) for (IIa), (III); *OSCAIL* and *SHELXL97* (Sheldrick, 1997) for (IIb). For all compounds, molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

(I) 1-(6-Amino-1,3-benzodioxol-5-yl)-3-(4-methylphenyl)prop-2-en-1-one

Crystal data

$C_{17}H_{15}NO_3$
$M_r = 281.30$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 10.530\ (5)$ Å
$b = 7.362\ (5)$ Å
$c = 17.546\ (5)$ Å
$\beta = 91.719\ (5)°$
$V = 1359.6\ (12)$ Å^3
$Z = 4$
$F(000) = 592$
$D_x = 1.374$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3062 reflections
$\theta = 5.3–27.5°$
$\mu = 0.10$ mm^{-1}
$T = 120$ K
Block, yellow
0.40 × 0.30 × 0.20 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Radiation source: Finefocus Sealed Tube
Graphite monochromator
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*EVALCCD*; Duisenberg et al., 2003)
$T_{min} = 0.958$, $T_{max} = 0.981$
17073 measured reflections
3062 independent reflections
2024 reflections with $I > 2\sigma(I)$
$R_{int} = 0.065$
$\theta_{max} = 27.5°$, $\theta_{min} = 5.3°$
$h = -13\rightarrow12$
$k = -9\rightarrow9$
$l = -22\rightarrow22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.135$
$S = 1.11$
3062 reflections
191 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0198P)^2 + 1.4934P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.00920 (16)	0.5248 (2)	0.70572 (10)	0.0377 (4)
C2	−0.1286 (2)	0.4763 (4)	0.73506 (16)	0.0395 (6)
O3	−0.14191 (15)	0.2809 (2)	0.72870 (10)	0.0376 (4)
O8	0.38327 (17)	−0.0352 (2)	0.61985 (11)	0.0420 (5)
N5	0.1656 (2)	−0.1693 (3)	0.66004 (12)	0.0349 (5)
C3a	−0.0267 (2)	0.2171 (3)	0.70827 (13)	0.0287 (5)
C4	0.0092 (2)	0.0401 (3)	0.69898 (13)	0.0296 (5)
C5	0.1324 (2)	0.0062 (3)	0.67436 (12)	0.0283 (5)
C6	0.2172 (2)	0.1534 (3)	0.66237 (12)	0.0272 (5)
C7	0.1736 (2)	0.3349 (3)	0.67335 (13)	0.0290 (5)
C7a	0.0542 (2)	0.3615 (3)	0.69493 (13)	0.0291 (5)
C8	0.3452 (2)	0.1205 (3)	0.63584 (13)	0.0305 (5)
C9	0.4315 (2)	0.2774 (3)	0.62687 (13)	0.0305 (5)
C10	0.5305 (2)	0.2676 (3)	0.58262 (13)	0.0316 (5)
C11	0.6208 (2)	0.4135 (3)	0.56747 (13)	0.0294 (5)
C12	0.6041 (2)	0.5939 (3)	0.59041 (13)	0.0332 (6)
C13	0.6932 (2)	0.7254 (3)	0.57474 (13)	0.0356 (6)
C14	0.8011 (2)	0.6835 (4)	0.53535 (13)	0.0342 (6)
C15	0.8158 (2)	0.5059 (4)	0.51085 (14)	0.0374 (6)
C16	0.7271 (2)	0.3723 (3)	0.52632 (14)	0.0351 (6)
C141	0.8975 (3)	0.8287 (4)	0.51910 (16)	0.0445 (7)
H2A	−0.1979	0.5376	0.7057	0.047*
H2B	−0.1329	0.5139	0.7891	0.047*
H4	−0.0476	−0.0568	0.7089	0.036*
H5A	0.2528	−0.1925	0.6488	0.042*
H5B	0.1103	−0.2648	0.6759	0.042*
H7	0.2285	0.4352	0.6655	0.035*
H9	0.4153	0.3870	0.6534	0.037*
H10	0.5446	0.1545	0.5582	0.038*
H12	0.5304	0.6263	0.6172	0.040*
H13	0.6801	0.8467	0.5913	0.043*
H15	0.8884	0.4747	0.4828	0.045*
H16	0.7394	0.2519	0.5085	0.042*
H14A	0.8545	0.9461	0.5134	0.067*

H14B	0.9406	0.7991	0.4719	0.067*
H14C	0.9602	0.8352	0.5614	0.067*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0316 (9)	0.0259 (9)	0.0564 (11)	0.0009 (7)	0.0126 (8)	0.0042 (8)
C2	0.0351 (13)	0.0329 (14)	0.0510 (16)	−0.0017 (11)	0.0112 (12)	0.0024 (12)
O3	0.0311 (9)	0.0317 (10)	0.0504 (10)	−0.0033 (8)	0.0093 (8)	0.0047 (8)
O8	0.0394 (10)	0.0289 (10)	0.0584 (12)	0.0014 (8)	0.0118 (9)	−0.0034 (8)
N5	0.0359 (11)	0.0233 (11)	0.0456 (12)	−0.0033 (9)	0.0042 (9)	0.0013 (9)
C3a	0.0272 (11)	0.0316 (13)	0.0273 (11)	−0.0020 (10)	0.0023 (9)	0.0038 (10)
C4	0.0313 (12)	0.0254 (13)	0.0322 (12)	−0.0081 (10)	0.0025 (10)	0.0037 (9)
C5	0.0318 (12)	0.0270 (13)	0.0259 (11)	−0.0018 (10)	−0.0020 (9)	0.0015 (9)
C6	0.0278 (11)	0.0263 (12)	0.0275 (11)	−0.0034 (10)	0.0019 (9)	0.0007 (9)
C7	0.0295 (12)	0.0266 (12)	0.0311 (12)	−0.0060 (10)	0.0033 (9)	0.0009 (9)
C7a	0.0340 (12)	0.0227 (12)	0.0307 (12)	−0.0011 (10)	0.0024 (9)	0.0016 (9)
C8	0.0314 (12)	0.0298 (13)	0.0301 (12)	−0.0017 (10)	−0.0007 (9)	0.0006 (10)
C9	0.0294 (12)	0.0289 (13)	0.0332 (12)	−0.0013 (10)	0.0023 (10)	−0.0041 (10)
C10	0.0324 (12)	0.0277 (13)	0.0347 (12)	0.0017 (10)	0.0014 (10)	−0.0015 (10)
C11	0.0285 (12)	0.0318 (13)	0.0280 (11)	0.0002 (10)	0.0006 (9)	0.0006 (10)
C12	0.0337 (13)	0.0346 (14)	0.0315 (12)	0.0005 (11)	0.0047 (10)	−0.0019 (10)
C13	0.0454 (14)	0.0292 (13)	0.0324 (12)	−0.0060 (11)	0.0008 (11)	−0.0020 (10)
C14	0.0353 (13)	0.0383 (15)	0.0287 (12)	−0.0085 (11)	−0.0035 (10)	0.0064 (11)
C15	0.0311 (13)	0.0415 (15)	0.0400 (14)	0.0012 (11)	0.0052 (11)	0.0040 (12)
C16	0.0344 (13)	0.0295 (13)	0.0418 (14)	0.0004 (11)	0.0054 (10)	−0.0019 (11)
C141	0.0408 (14)	0.0494 (17)	0.0429 (15)	−0.0157 (13)	−0.0030 (12)	0.0048 (13)

Geometric parameters (Å, º)

O1—C7a	1.391 (3)	C8—C9	1.480 (3)
O1—C2	1.418 (3)	C9—C10	1.321 (3)
C2—O3	1.449 (3)	C9—H9	0.95
C2—H2A	0.99	C10—C11	1.464 (3)
C2—H2B	0.99	C10—H10	0.95
O3—C3a	1.359 (3)	C11—C16	1.383 (3)
C3a—C4	1.368 (3)	C11—C12	1.400 (3)
C3a—C7a	1.386 (3)	C12—C13	1.381 (3)
C4—C5	1.402 (3)	C12—H12	0.95
C4—H4	0.95	C13—C14	1.383 (4)
C5—N5	1.364 (3)	C13—H13	0.95
C5—C6	1.424 (3)	C14—C15	1.387 (4)
N5—H5A	0.96	C14—C141	1.507 (3)
N5—H5B	0.96	C141—H14A	0.98
C6—C7	1.428 (3)	C141—H14B	0.98
C6—C8	1.459 (3)	C141—H14C	0.98
C7—C7a	1.339 (3)	C15—C16	1.389 (4)
C7—H7	0.95	C15—H15	0.95

C8—O8	1.249 (3)	C16—H16	0.95
C7a—O1—C2	105.41 (18)	C6—C8—C9	118.7 (2)
O1—C2—O3	107.88 (19)	C10—C9—C8	121.2 (2)
O1—C2—H2A	110.1	C10—C9—H9	119.4
O3—C2—H2A	110.1	C8—C9—H9	119.4
O1—C2—H2B	110.1	C9—C10—C11	126.5 (2)
O3—C2—H2B	110.1	C9—C10—H10	116.7
H2A—C2—H2B	108.4	C11—C10—H10	116.7
C3a—O3—C2	106.14 (18)	C16—C11—C12	117.9 (2)
O3—C3a—C4	127.8 (2)	C16—C11—C10	118.3 (2)
O3—C3a—C7a	109.7 (2)	C12—C11—C10	123.8 (2)
C4—C3a—C7a	122.5 (2)	C13—C12—C11	121.0 (2)
C3a—C4—C5	117.9 (2)	C13—C12—H12	119.5
C3a—C4—H4	121.1	C11—C12—H12	119.5
C5—C4—H4	121.1	C12—C13—C14	121.1 (2)
N5—C5—C4	118.0 (2)	C12—C13—H13	119.5
N5—C5—C6	121.9 (2)	C14—C13—H13	119.5
C4—C5—C6	120.0 (2)	C13—C14—C15	117.8 (2)
C5—N5—H5A	117.3	C13—C14—C141	120.3 (2)
C5—N5—H5B	118.6	C15—C14—C141	121.9 (2)
H5A—N5—H5B	121.4	C14—C141—H14A	109.5
C5—C6—C7	119.1 (2)	C14—C141—H14B	109.5
C5—C6—C8	120.7 (2)	H14A—C141—H14B	109.5
C7—C6—C8	120.1 (2)	C14—C141—H14C	109.5
C7a—C7—C6	118.9 (2)	H14A—C141—H14C	109.5
C7a—C7—H7	120.5	H14B—C141—H14C	109.5
C6—C7—H7	120.5	C14—C15—C16	121.7 (2)
C7—C7a—C3a	121.5 (2)	C14—C15—H15	119.2
C7—C7a—O1	128.6 (2)	C16—C15—H15	119.2
C3a—C7a—O1	109.9 (2)	C11—C16—C15	120.4 (2)
O8—C8—C6	122.0 (2)	C11—C16—H16	119.8
O8—C8—C9	119.3 (2)	C15—C16—H16	119.8
C7a—O1—C2—O3	−9.7 (3)	C2—O1—C7a—C3a	6.7 (3)
O1—C2—O3—C3a	9.4 (3)	C5—C6—C8—O8	2.9 (3)
C2—O3—C3a—C4	176.6 (2)	C7—C6—C8—O8	−174.3 (2)
C2—O3—C3a—C7a	−5.3 (3)	C5—C6—C8—C9	−177.8 (2)
O3—C3a—C4—C5	177.4 (2)	C7—C6—C8—C9	5.0 (3)
C7a—C3a—C4—C5	−0.6 (3)	O8—C8—C9—C10	20.2 (3)
C3a—C4—C5—N5	−175.6 (2)	C6—C8—C9—C10	−159.1 (2)
C3a—C4—C5—C6	2.1 (3)	C8—C9—C10—C11	178.4 (2)
N5—C5—C6—C7	175.7 (2)	C9—C10—C11—C16	172.8 (2)
C4—C5—C6—C7	−2.0 (3)	C9—C10—C11—C12	−9.0 (4)
N5—C5—C6—C8	−1.6 (3)	C16—C11—C12—C13	−2.2 (3)
C4—C5—C6—C8	−179.2 (2)	C10—C11—C12—C13	179.7 (2)
C5—C6—C7—C7a	0.3 (3)	C11—C12—C13—C14	0.6 (4)
C8—C6—C7—C7a	177.5 (2)	C12—C13—C14—C15	1.2 (4)

C6—C7—C7a—C3a	1.3 (3)	C12—C13—C14—C141	−179.7 (2)
C6—C7—C7a—O1	−177.1 (2)	C13—C14—C15—C16	−1.3 (4)
O3—C3a—C7a—C7	−179.5 (2)	C141—C14—C15—C16	179.6 (2)
C4—C3a—C7a—C7	−1.2 (4)	C12—C11—C16—C15	2.1 (3)
O3—C3a—C7a—O1	−0.8 (3)	C10—C11—C16—C15	−179.6 (2)
C4—C3a—C7a—O1	177.4 (2)	C14—C15—C16—C11	−0.4 (4)
C2—O1—C7a—C7	−174.8 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5*A*···O8	0.96	1.88	2.612 (3)	131
N5—H5*B*···O1^{i}	0.96	2.07	3.032 (3)	178
C2—H2*A*···*Cg*1^{ii}	0.99	2.86	3.644 (4)	137

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

(IIa) 1-(6-Amino-1,3-benzodioxol-5-yl)-3-(4-methoxyphenyl)prop-2-en-1-one, monoclinic polymorph

Crystal data

$C_{17}H_{15}NO_4$
M_r = 297.30
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 17.5560 (4) Å
b = 5.0914 (2) Å
c = 15.4869 (4) Å
β = 91.9240 (16)°
V = 1383.51 (7) Å^3
Z = 4
$F(000)$ = 624
D_x = 1.427 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3170 reflections
θ = 2.9–27.5°
μ = 0.10 mm^{-1}
T = 120 K
Block, red
0.04 × 0.02 × 0.02 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Radiation source: Bruker-Nonius FR591 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.959, T_{max} = 0.998
14325 measured reflections
3170 independent reflections
2271 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 27.5°, θ_{min} = 2.9°
h = −21→22
k = −6→6
l = −20→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.128
S = 1.04
3169 reflections
201 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0701P)^2 + 0.2374P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.007 (2)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.78208 (6)	−0.4220 (2)	0.01174 (7)	0.0337 (3)
C2	0.83733 (8)	−0.5459 (4)	−0.03950 (11)	0.0340 (4)
O3	0.91051 (6)	−0.4401 (2)	−0.01723 (7)	0.0356 (3)
C3a	0.89963 (8)	−0.2603 (3)	0.04676 (9)	0.0252 (4)
C4	0.95265 (8)	−0.1077 (3)	0.08815 (10)	0.0267 (4)
C5	0.92780 (8)	0.0730 (3)	0.15079 (9)	0.0232 (3)
N5	0.98106 (6)	0.2294 (3)	0.18954 (8)	0.0277 (3)
C6	0.84914 (8)	0.0844 (3)	0.17162 (9)	0.0222 (3)
C7	0.79644 (8)	−0.0847 (3)	0.12632 (9)	0.0242 (3)
C7a	0.82251 (8)	−0.2480 (3)	0.06499 (9)	0.0244 (3)
C8	0.82376 (8)	0.2649 (3)	0.23813 (9)	0.0240 (3)
O8	0.86837 (5)	0.4110 (2)	0.28000 (7)	0.0303 (3)
C9	0.74153 (8)	0.2741 (3)	0.25819 (9)	0.0252 (4)
C10	0.71444 (8)	0.4477 (3)	0.31353 (9)	0.0248 (3)
C11	0.63473 (8)	0.4739 (3)	0.33750 (9)	0.0224 (3)
C12	0.57815 (8)	0.2974 (3)	0.30779 (9)	0.0240 (3)
C13	0.50314 (8)	0.3286 (3)	0.32865 (9)	0.0240 (3)
C14	0.48187 (8)	0.5373 (3)	0.38070 (9)	0.0228 (3)
O14	0.40540 (5)	0.5557 (2)	0.39519 (6)	0.0269 (3)
C141	0.38014 (8)	0.7774 (3)	0.44357 (10)	0.0302 (4)
C15	0.53670 (8)	0.7104 (3)	0.41239 (9)	0.0257 (4)
C16	0.61226 (8)	0.6771 (3)	0.39075 (10)	0.0261 (4)
H2A	0.8250	−0.5151	−0.1015	0.041*
H2B	0.8372	−0.7377	−0.0290	0.041*
H4	1.0051	−0.1217	0.0755	0.032*
H5A	0.9664	0.3721	0.2260	0.033*
H5B	1.0317	0.2275	0.1681	0.033*
H7	0.7439	−0.0824	0.1391	0.029*
H9	0.7075	0.1527	0.2308	0.030*
H10	0.7501	0.5657	0.3400	0.030*
H12	0.5920	0.1535	0.2726	0.029*
H13	0.4658	0.2076	0.3075	0.029*
H14A	0.4036	0.7724	0.5019	0.045*
H14B	0.3246	0.7715	0.4473	0.045*
H14C	0.3950	0.9397	0.4146	0.045*
H15	0.5228	0.8514	0.4488	0.031*
H16	0.6496	0.7966	0.4130	0.031*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0224 (6)	0.0406 (7)	0.0384 (6)	−0.0021 (5)	0.0042 (5)	−0.0136 (6)
C2	0.0247 (8)	0.0368 (10)	0.0407 (9)	0.0012 (7)	0.0044 (7)	−0.0109 (8)
O3	0.0232 (6)	0.0460 (8)	0.0380 (7)	0.0004 (5)	0.0057 (5)	−0.0161 (6)
C3a	0.0231 (7)	0.0292 (9)	0.0237 (8)	0.0048 (6)	0.0042 (6)	0.0003 (7)

C4	0.0159 (7)	0.0346 (9)	0.0299 (8)	0.0030 (6)	0.0046 (6)	0.0020 (7)
C5	0.0195 (7)	0.0258 (8)	0.0243 (7)	0.0011 (6)	0.0017 (6)	0.0059 (7)
N5	0.0159 (6)	0.0320 (8)	0.0354 (7)	−0.0018 (5)	0.0034 (5)	−0.0038 (6)
C6	0.0169 (7)	0.0252 (8)	0.0245 (7)	0.0017 (6)	0.0014 (6)	0.0043 (6)
C7	0.0160 (7)	0.0284 (8)	0.0283 (8)	−0.0005 (6)	0.0037 (6)	0.0014 (7)
C7a	0.0201 (7)	0.0272 (9)	0.0260 (8)	−0.0011 (6)	0.0003 (6)	0.0012 (7)
C8	0.0189 (7)	0.0281 (9)	0.0250 (7)	0.0001 (6)	0.0015 (6)	0.0037 (7)
O8	0.0200 (5)	0.0361 (7)	0.0350 (6)	−0.0021 (5)	0.0022 (4)	−0.0073 (5)
C9	0.0179 (7)	0.0327 (9)	0.0250 (7)	−0.0007 (6)	0.0014 (6)	−0.0024 (7)
C10	0.0210 (7)	0.0269 (8)	0.0263 (8)	−0.0010 (6)	0.0004 (6)	0.0000 (7)
C11	0.0192 (7)	0.0263 (8)	0.0217 (7)	0.0025 (6)	0.0010 (6)	0.0002 (6)
C12	0.0242 (7)	0.0271 (8)	0.0208 (7)	0.0022 (6)	0.0020 (6)	−0.0008 (6)
C13	0.0209 (7)	0.0286 (9)	0.0223 (7)	−0.0023 (6)	−0.0002 (6)	−0.0012 (6)
C14	0.0172 (7)	0.0297 (9)	0.0216 (7)	0.0024 (6)	0.0018 (6)	0.0025 (6)
O14	0.0183 (5)	0.0344 (7)	0.0282 (6)	0.0015 (4)	0.0050 (4)	−0.0047 (5)
C15	0.0255 (8)	0.0263 (8)	0.0253 (8)	0.0037 (6)	0.0026 (6)	−0.0035 (7)
C16	0.0218 (7)	0.0272 (8)	0.0292 (8)	−0.0014 (6)	0.0014 (6)	−0.0026 (7)
C141	0.0241 (8)	0.0348 (10)	0.0322 (8)	0.0051 (7)	0.0074 (6)	−0.0033 (7)

Geometric parameters (Å, º)

O1—C7a	1.3890 (18)	C9—C10	1.330 (2)
O1—C2	1.4214 (18)	C9—H9	0.95
C2—O3	1.4248 (19)	C10—C11	1.4659 (19)
C2—H2A	0.99	C10—H10	0.95
C2—H2B	0.99	C11—C16	1.388 (2)
O3—C3a	1.3673 (18)	C11—C12	1.406 (2)
C3a—C4	1.357 (2)	C12—C13	1.376 (2)
C3a—C7a	1.3937 (19)	C12—H12	0.95
C4—C5	1.416 (2)	C13—C14	1.392 (2)
C4—H4	0.95	C13—H13	0.95
C5—N5	1.3531 (19)	C14—O14	1.3718 (17)
C5—C6	1.4297 (19)	C14—C15	1.383 (2)
N5—H5A	0.96	O14—C141	1.4332 (18)
N5—H5B	0.9599	C141—H14A	0.98
C6—C7	1.430 (2)	C141—H14B	0.98
C6—C8	1.461 (2)	C141—H14C	0.98
C7—C7a	1.354 (2)	C15—C16	1.3894 (19)
C7—H7	0.95	C15—H15	0.95
C8—O8	1.2460 (18)	C16—H16	0.95
C8—C9	1.4877 (19)		
C7a—O1—C2	105.65 (11)	C10—C9—C8	121.71 (14)
O1—C2—O3	108.89 (13)	C10—C9—H9	119.1
O1—C2—H2A	109.9	C8—C9—H9	119.1
O3—C2—H2A	109.9	C9—C10—C11	126.05 (14)
O1—C2—H2B	109.9	C9—C10—H10	117.0
O3—C2—H2B	109.9	C11—C10—H10	117.0

H2A—C2—H2B	108.3	C16—C11—C12	117.33 (13)
C3a—O3—C2	106.38 (11)	C16—C11—C10	120.72 (13)
C4—C3a—O3	127.99 (13)	C12—C11—C10	121.95 (13)
C4—C3a—C7a	122.30 (14)	C13—C12—C11	121.39 (14)
O3—C3a—C7a	109.70 (13)	C13—C12—H12	119.3
C3a—C4—C5	118.25 (13)	C11—C12—H12	119.3
C3a—C4—H4	120.9	C12—C13—C14	120.11 (14)
C5—C4—H4	120.9	C12—C13—H13	119.9
N5—C5—C4	117.51 (13)	C14—C13—H13	119.9
N5—C5—C6	122.30 (14)	O14—C14—C15	124.88 (13)
C4—C5—C6	120.19 (14)	O14—C14—C13	115.46 (13)
C5—N5—H5A	120.7	C15—C14—C13	119.65 (13)
C5—N5—H5B	118.4	C14—O14—C141	117.45 (12)
H5A—N5—H5B	118.7	O14—C141—H14A	109.5
C5—C6—C7	118.61 (13)	O14—C141—H14B	109.5
C5—C6—C8	120.27 (13)	H14A—C141—H14B	109.5
C7—C6—C8	121.12 (12)	O14—C141—H14C	109.5
C7a—C7—C6	119.05 (13)	H14A—C141—H14C	109.5
C7a—C7—H7	120.5	H14B—C141—H14C	109.5
C6—C7—H7	120.5	C14—C15—C16	119.70 (14)
C7—C7a—O1	129.08 (13)	C14—C15—H15	120.1
C7—C7a—C3a	121.55 (14)	C16—C15—H15	120.1
O1—C7a—C3a	109.37 (12)	C11—C16—C15	121.78 (14)
O8—C8—C6	122.76 (12)	C11—C16—H16	119.1
O8—C8—C9	118.04 (13)	C15—C16—H16	119.1
C6—C8—C9	119.20 (13)		

C7a—O1—C2—O3	−1.08 (17)	C5—C6—C8—O8	−1.2 (2)
O1—C2—O3—C3a	0.93 (18)	C7—C6—C8—O8	178.52 (14)
C2—O3—C3a—C4	−179.51 (16)	C5—C6—C8—C9	179.63 (13)
C2—O3—C3a—C7a	−0.40 (17)	C7—C6—C8—C9	−0.6 (2)
O3—C3a—C4—C5	177.80 (14)	O8—C8—C9—C10	5.8 (2)
C7a—C3a—C4—C5	−1.2 (2)	C6—C8—C9—C10	−175.03 (14)
C3a—C4—C5—N5	−177.99 (14)	C8—C9—C10—C11	179.77 (14)
C3a—C4—C5—C6	2.5 (2)	C9—C10—C11—C16	−174.69 (15)
N5—C5—C6—C7	178.83 (13)	C9—C10—C11—C12	5.1 (2)
C4—C5—C6—C7	−1.7 (2)	C16—C11—C12—C13	1.7 (2)
N5—C5—C6—C8	−1.4 (2)	C10—C11—C12—C13	−178.04 (14)
C4—C5—C6—C8	178.10 (13)	C11—C12—C13—C14	−0.4 (2)
C5—C6—C7—C7a	−0.5 (2)	C12—C13—C14—O14	177.87 (13)
C8—C6—C7—C7a	179.76 (14)	C12—C13—C14—C15	−1.0 (2)
C6—C7—C7a—O1	−178.04 (14)	C15—C14—O14—C141	2.9 (2)
C6—C7—C7a—C3a	1.8 (2)	C13—C14—O14—C141	−175.91 (13)
C2—O1—C7a—C7	−179.31 (16)	O14—C14—C15—C16	−177.65 (13)
C2—O1—C7a—C3a	0.83 (17)	C13—C14—C15—C16	1.1 (2)
C4—C3a—C7a—C7	−1.0 (2)	C12—C11—C16—C15	−1.6 (2)
O3—C3a—C7a—C7	179.86 (13)	C10—C11—C16—C15	178.15 (14)
C4—C3a—C7a—O1	178.89 (14)	C14—C15—C16—C11	0.2 (2)

O3—C3a—C7a—O1	−0.28 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5*A*···O8	0.96	1.95	2.6301 (15)	126
N5—H5*B*···O8^{i}	0.96	2.49	3.1232 (15)	123
C2—H2*B*···*Cg*2^{ii}	0.99	2.84	3.640 (2)	138
C13—H13···*Cg*1^{iii}	0.95	2.81	3.488 (2)	130

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

(IIb) 1-(6-Amino-1,3-benzodioxol-5-yl)-3-(4-methoxyphenyl)prop-2-en-1-one, triclinic polymorph

Crystal data

$C_{17}H_{15}NO_4$
M_r = 297.30
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 9.5352 (2) Å
b = 10.6193 (3) Å
c = 14.7611 (4) Å
α = 89.1400 (14)°
β = 81.0970 (17)°
γ = 75.7540 (14)°
V = 1430.83 (6) Å^3
Z = 4
F(000) = 624
D_x = 1.380 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 6517 reflections
θ = 3.1–27.5°
μ = 0.10 mm^{-1}
T = 120 K
Block, yellow
0.45 × 0.30 × 0.20 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Radiation source: Bruker-Nonius FR591 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and *ω* scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.951, T_{max} = 0.981
23999 measured reflections
6517 independent reflections
4940 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 27.5°, θ_{min} = 3.1°
h = −12→12
k = −13→13
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.137
S = 1.10
6512 reflections
399 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0778P)^2 + 0.1431P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O11	0.93492 (11)	1.05175 (11)	−0.26740 (7)	0.0293 (3)

C12	0.82075 (18)	1.0549 (2)	−0.19306 (12)	0.0433 (5)
O13	0.88228 (11)	0.97686 (10)	−0.12149 (7)	0.0278 (3)
C13a	1.03083 (15)	0.96323 (13)	−0.14296 (10)	0.0219 (3)
C14	1.13545 (16)	0.91026 (14)	−0.09121 (10)	0.0233 (3)
C15	1.28298 (16)	0.90599 (13)	−0.12808 (10)	0.0212 (3)
N15	1.38864 (14)	0.85000 (13)	−0.07718 (9)	0.0303 (3)
C16	1.31659 (15)	0.95830 (13)	−0.21581 (9)	0.0196 (3)
C17	1.20217 (15)	1.00866 (13)	−0.26744 (10)	0.0211 (3)
C17a	1.06302 (15)	1.01012 (13)	−0.22988 (10)	0.0216 (3)
C18	1.46764 (15)	0.96232 (13)	−0.25280 (10)	0.0214 (3)
O18	1.57336 (11)	0.90408 (10)	−0.21638 (7)	0.0268 (2)
C19	1.49422 (16)	1.03991 (14)	−0.33514 (10)	0.0242 (3)
C110	1.62835 (16)	1.03311 (13)	−0.37915 (10)	0.0222 (3)
C111	1.67155 (15)	1.10589 (13)	−0.45907 (10)	0.0204 (3)
C112	1.57301 (15)	1.17991 (14)	−0.51147 (10)	0.0233 (3)
C113	1.61880 (16)	1.25048 (14)	−0.58487 (10)	0.0239 (3)
C114	1.76706 (16)	1.24714 (13)	−0.60766 (10)	0.0222 (3)
O114	1.82482 (11)	1.31281 (10)	−0.67802 (7)	0.0298 (3)
C141	1.72509 (19)	1.39249 (16)	−0.73043 (12)	0.0346 (4)
C115	1.86764 (15)	1.17362 (14)	−0.55668 (10)	0.0237 (3)
C116	1.82016 (15)	1.10385 (14)	−0.48380 (10)	0.0227 (3)
O21	0.90504 (13)	0.30396 (10)	−0.07496 (7)	0.0326 (3)
C22	1.01329 (19)	0.31569 (15)	−0.15102 (11)	0.0327 (4)
O23	1.02449 (12)	0.44767 (10)	−0.15327 (8)	0.0349 (3)
C23a	0.90742 (16)	0.51538 (14)	−0.09352 (10)	0.0252 (3)
C24	0.86521 (16)	0.64606 (14)	−0.07812 (10)	0.0265 (3)
C25	0.74048 (16)	0.69550 (14)	−0.01122 (10)	0.0232 (3)
N25	0.69942 (15)	0.82629 (12)	0.00494 (9)	0.0301 (3)
C26	0.66336 (16)	0.61005 (13)	0.03725 (10)	0.0223 (3)
C27	0.71473 (16)	0.47289 (14)	0.01813 (10)	0.0242 (3)
C27a	0.83396 (16)	0.43040 (14)	−0.04616 (10)	0.0237 (3)
C28	0.53670 (16)	0.66027 (14)	0.10777 (10)	0.0252 (3)
O28	0.48934 (13)	0.77871 (10)	0.12534 (8)	0.0372 (3)
C29	0.46453 (16)	0.56927 (14)	0.16273 (10)	0.0248 (3)
C210	0.39261 (16)	0.60410 (14)	0.24716 (10)	0.0259 (3)
C211	0.32337 (16)	0.52343 (14)	0.31214 (10)	0.0243 (3)
C212	0.29118 (17)	0.40866 (15)	0.28601 (11)	0.0282 (3)
C213	0.23095 (17)	0.33422 (15)	0.35017 (11)	0.0305 (4)
C214	0.20247 (16)	0.37000 (14)	0.44281 (11)	0.0264 (3)
O214	0.14627 (13)	0.28658 (11)	0.50080 (8)	0.0351 (3)
C241	0.1067 (2)	0.32327 (17)	0.59548 (12)	0.0388 (4)
C215	0.23255 (17)	0.48297 (15)	0.47021 (11)	0.0294 (3)
C216	0.29181 (17)	0.55836 (15)	0.40472 (11)	0.0295 (3)
H12A	0.7431	1.0199	−0.2130	0.052*
H12B	0.7770	1.1455	−0.1703	0.052*
H14	1.1107	0.8772	−0.0323	0.028*
H15A	1.3642	0.8180	−0.0234	0.036*
H15B	1.4813	0.8458	−0.0982	0.036*

H17	1.2229	1.0407	−0.3272	0.025*
H19	1.4131	1.0960	−0.3570	0.029*
H110	1.7057	0.9737	−0.3559	0.027*
H112	1.4719	1.1820	−0.4964	0.028*
H113	1.5497	1.3008	−0.6194	0.029*
H14A	1.6564	1.4607	−0.6906	0.052*
H14B	1.6707	1.3391	−0.7572	0.052*
H14C	1.7797	1.4324	−0.7796	0.052*
H115	1.9687	1.1715	−0.5720	0.028*
H116	1.8897	1.0533	−0.4496	0.027*
H22A	0.9846	0.2919	−0.2089	0.039*
H22B	1.1089	0.2567	−0.1441	0.039*
H24	0.9174	0.7023	−0.1110	0.032*
H25B	0.7498	0.8768	−0.0257	0.036*
H25A	0.6225	0.8604	0.0460	0.036*
H27	0.6660	0.4134	0.0499	0.029*
H29	0.4694	0.4860	0.1379	0.030*
H210	0.3856	0.6903	0.2672	0.031*
H212	0.3113	0.3821	0.2232	0.034*
H213	0.2084	0.2574	0.3311	0.037*
H24A	0.1946	0.3269	0.6210	0.058*
H24B	0.0608	0.2592	0.6284	0.058*
H24C	0.0374	0.4090	0.6022	0.058*
H215	0.2129	0.5088	0.5332	0.035*
H216	0.3115	0.6365	0.4238	0.035*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O11	0.0194 (5)	0.0438 (7)	0.0250 (6)	−0.0092 (5)	−0.0023 (4)	0.0055 (5)
C12	0.0245 (8)	0.0760 (14)	0.0314 (10)	−0.0186 (9)	−0.0013 (7)	0.0129 (9)
O13	0.0211 (5)	0.0374 (6)	0.0255 (6)	−0.0124 (5)	0.0029 (4)	0.0035 (5)
C13a	0.0217 (7)	0.0221 (7)	0.0223 (7)	−0.0105 (6)	0.0038 (6)	−0.0024 (6)
C14	0.0277 (7)	0.0247 (7)	0.0176 (7)	−0.0101 (6)	0.0021 (6)	0.0024 (6)
C15	0.0253 (7)	0.0177 (7)	0.0207 (7)	−0.0070 (6)	−0.0014 (6)	0.0009 (5)
N15	0.0278 (7)	0.0384 (8)	0.0236 (7)	−0.0075 (6)	−0.0033 (6)	0.0130 (6)
C16	0.0221 (7)	0.0181 (7)	0.0183 (7)	−0.0068 (6)	0.0013 (6)	−0.0003 (5)
C17	0.0237 (7)	0.0222 (7)	0.0171 (7)	−0.0078 (6)	0.0008 (6)	0.0024 (5)
C17a	0.0210 (7)	0.0234 (7)	0.0204 (7)	−0.0067 (6)	−0.0019 (6)	0.0012 (6)
C18	0.0233 (7)	0.0192 (7)	0.0213 (7)	−0.0067 (6)	0.0001 (6)	−0.0015 (5)
O18	0.0219 (5)	0.0298 (6)	0.0273 (6)	−0.0046 (4)	−0.0027 (4)	0.0064 (4)
C19	0.0213 (7)	0.0256 (7)	0.0252 (8)	−0.0065 (6)	−0.0016 (6)	0.0052 (6)
C110	0.0233 (7)	0.0225 (7)	0.0206 (7)	−0.0062 (6)	−0.0019 (6)	0.0014 (6)
C111	0.0215 (7)	0.0199 (7)	0.0186 (7)	−0.0058 (6)	0.0018 (6)	−0.0017 (5)
C112	0.0194 (7)	0.0277 (8)	0.0232 (8)	−0.0086 (6)	−0.0006 (6)	0.0009 (6)
C113	0.0229 (7)	0.0266 (8)	0.0225 (8)	−0.0062 (6)	−0.0041 (6)	0.0033 (6)
C114	0.0265 (7)	0.0216 (7)	0.0177 (7)	−0.0076 (6)	0.0018 (6)	0.0014 (5)
O114	0.0280 (6)	0.0346 (6)	0.0260 (6)	−0.0099 (5)	0.0003 (5)	0.0115 (5)

C141	0.0372 (9)	0.0380 (9)	0.0298 (9)	−0.0119 (7)	−0.0061 (7)	0.0146 (7)
C115	0.0190 (7)	0.0258 (7)	0.0249 (8)	−0.0066 (6)	0.0022 (6)	0.0000 (6)
C116	0.0202 (7)	0.0237 (7)	0.0216 (7)	−0.0018 (6)	−0.0009 (6)	0.0016 (6)
O21	0.0424 (7)	0.0222 (6)	0.0282 (6)	−0.0069 (5)	0.0085 (5)	−0.0011 (4)
C22	0.0402 (9)	0.0279 (8)	0.0267 (8)	−0.0085 (7)	0.0055 (7)	−0.0015 (6)
O23	0.0357 (6)	0.0270 (6)	0.0365 (7)	−0.0074 (5)	0.0112 (5)	−0.0026 (5)
C23a	0.0250 (7)	0.0283 (8)	0.0215 (8)	−0.0076 (6)	0.0007 (6)	0.0011 (6)
C24	0.0278 (8)	0.0249 (8)	0.0275 (8)	−0.0100 (6)	−0.0010 (6)	0.0038 (6)
C25	0.0259 (7)	0.0228 (7)	0.0221 (7)	−0.0069 (6)	−0.0058 (6)	0.0033 (6)
N25	0.0318 (7)	0.0213 (7)	0.0349 (8)	−0.0081 (5)	0.0047 (6)	0.0022 (5)
C26	0.0249 (7)	0.0226 (7)	0.0195 (7)	−0.0065 (6)	−0.0033 (6)	0.0033 (6)
C27	0.0303 (8)	0.0239 (7)	0.0198 (7)	−0.0101 (6)	−0.0030 (6)	0.0043 (6)
C27a	0.0299 (8)	0.0206 (7)	0.0208 (7)	−0.0064 (6)	−0.0040 (6)	0.0008 (6)
C28	0.0279 (7)	0.0250 (8)	0.0215 (8)	−0.0048 (6)	−0.0029 (6)	0.0050 (6)
O28	0.0408 (7)	0.0243 (6)	0.0367 (7)	−0.0006 (5)	0.0111 (5)	0.0045 (5)
C29	0.0243 (7)	0.0252 (8)	0.0242 (8)	−0.0052 (6)	−0.0030 (6)	0.0038 (6)
C210	0.0274 (7)	0.0235 (7)	0.0252 (8)	−0.0049 (6)	−0.0014 (6)	0.0038 (6)
C211	0.0234 (7)	0.0233 (7)	0.0236 (8)	−0.0038 (6)	0.0009 (6)	0.0019 (6)
C212	0.0295 (8)	0.0313 (8)	0.0232 (8)	−0.0086 (7)	−0.0007 (6)	−0.0032 (6)
C213	0.0324 (8)	0.0262 (8)	0.0343 (9)	−0.0126 (7)	−0.0006 (7)	−0.0024 (7)
C214	0.0234 (7)	0.0245 (8)	0.0292 (8)	−0.0054 (6)	0.0010 (6)	0.0059 (6)
O214	0.0399 (6)	0.0315 (6)	0.0331 (7)	−0.0147 (5)	0.0056 (5)	0.0061 (5)
C241	0.0389 (9)	0.0411 (10)	0.0315 (9)	−0.0076 (8)	0.0051 (7)	0.0111 (7)
C215	0.0352 (8)	0.0301 (8)	0.0216 (8)	−0.0093 (7)	0.0015 (7)	0.0000 (6)
C216	0.0359 (9)	0.0251 (8)	0.0276 (8)	−0.0108 (7)	0.0002 (7)	−0.0017 (6)

Geometric parameters (Å, º)

O11—C17a	1.3894 (17)	O21—C27a	1.3867 (18)
O11—C12	1.416 (2)	O21—C22	1.4289 (19)
C12—O13	1.440 (2)	C22—O23	1.4310 (19)
C12—H12A	0.99	C22—H22A	0.99
C12—H12B	0.99	C22—H22B	0.99
O13—C13a	1.3745 (17)	O23—C23a	1.3639 (18)
C13a—C14	1.357 (2)	C23a—C24	1.359 (2)
C13a—C17a	1.387 (2)	C23a—C27a	1.391 (2)
C14—C15	1.418 (2)	C24—C25	1.418 (2)
C14—H14	0.95	C24—H24	0.95
C15—N15	1.364 (2)	C25—N25	1.361 (2)
C15—C16	1.423 (2)	C25—C26	1.422 (2)
N15—H15A	0.88	N25—H25B	0.88
N15—H15B	0.88	N25—H25A	0.88
C16—C17	1.420 (2)	C26—C27	1.435 (2)
C16—C18	1.470 (2)	C26—C28	1.462 (2)
C17—C17a	1.353 (2)	C27—C27a	1.350 (2)
C17—H17	0.95	C27—H27	0.95
C18—O18	1.240 (2)	C28—O28	1.244 (2)
C18—C19	1.483 (2)	C28—C29	1.482 (2)

C19—C110	1.327 (2)	C29—C210	1.336 (2)
C19—H19	0.95	C29—H29	0.95
C110—C111	1.4620 (19)	C210—C211	1.465 (2)
C110—H110	0.95	C210—H210	0.95
C111—C112	1.3936 (19)	C211—C216	1.389 (2)
C111—C116	1.4024 (19)	C211—C212	1.401 (2)
C112—C113	1.384 (2)	C212—C213	1.373 (2)
C112—H112	0.95	C212—H212	0.95
C113—C114	1.393 (2)	C213—C214	1.392 (2)
C113—H113	0.95	C213—H213	0.95
C114—O114	1.3633 (17)	C214—O214	1.3688 (18)
C114—C115	1.390 (2)	C214—C215	1.380 (2)
O114—C141	1.4251 (18)	O214—C241	1.424 (2)
C141—H14A	0.98	C241—H24A	0.98
C141—H14B	0.98	C241—H24B	0.98
C141—H14C	0.98	C241—H24C	0.98
C115—C116	1.379 (2)	C215—C216	1.388 (2)
C115—H115	0.95	C215—H215	0.95
C116—H116	0.95	C216—H216	0.95

C17a—O11—C12	104.76 (11)	C27a—O21—C22	105.38 (11)
O11—C12—O13	107.95 (13)	O21—C22—O23	108.06 (12)
O11—C12—H12A	110.1	O21—C22—H22A	110.1
O13—C12—H12A	110.1	O23—C22—H22A	110.1
O11—C12—H12B	110.1	O21—C22—H22B	110.1
O13—C12—H12B	110.1	O23—C22—H22B	110.1
H12A—C12—H12B	108.4	H22A—C22—H22B	108.4
C13a—O13—C12	104.95 (11)	C23a—O23—C22	105.80 (12)
C14—C13a—O13	127.68 (13)	C24—C23a—O23	127.26 (14)
C14—C13a—C17a	122.81 (13)	C24—C23a—C27a	122.60 (14)
O13—C13a—C17a	109.50 (12)	O23—C23a—C27a	110.13 (13)
C13a—C14—C15	117.67 (13)	C23a—C24—C25	117.61 (14)
C13a—C14—H14	121.2	C23a—C24—H24	121.2
C15—C14—H14	121.2	C25—C24—H24	121.2
N15—C15—C14	117.80 (13)	N25—C25—C24	117.53 (13)
N15—C15—C16	122.27 (13)	N25—C25—C26	121.96 (14)
C14—C15—C16	119.93 (13)	C24—C25—C26	120.51 (13)
C15—N15—H15A	120.0	C25—N25—H25B	120.0
C15—N15—H15B	120.0	C25—N25—H25A	120.0
H15A—N15—H15B	120.0	H25B—N25—H25A	120.0
C17—C16—C15	119.39 (12)	C25—C26—C27	118.97 (13)
C17—C16—C18	119.79 (12)	C25—C26—C28	120.71 (13)
C15—C16—C18	120.81 (12)	C27—C26—C28	120.29 (13)
C17a—C17—C16	118.67 (13)	C27a—C27—C26	118.38 (13)
C17a—C17—H17	120.7	C27a—C27—H27	120.8
C16—C17—H17	120.7	C26—C27—H27	120.8
C17—C17a—C13a	121.45 (13)	C27—C27a—O21	128.93 (13)
C17—C17a—O11	129.04 (13)	C27—C27a—C23a	121.94 (14)

C13a—C17a—O11	109.50 (12)	O21—C27a—C23a	109.12 (13)
O18—C18—C16	121.90 (13)	O28—C28—C26	121.63 (13)
O18—C18—C19	119.03 (13)	O28—C28—C29	118.26 (14)
C16—C18—C19	119.07 (12)	C26—C28—C29	120.08 (13)
C110—C19—C18	121.78 (13)	C210—C29—C28	120.12 (14)
C110—C19—H19	119.1	C210—C29—H29	119.9
C18—C19—H19	119.1	C28—C29—H29	119.9
C19—C110—C111	127.91 (13)	C29—C210—C211	126.92 (14)
C19—C110—H110	116.0	C29—C210—H210	116.5
C111—C110—H110	116.0	C211—C210—H210	116.5
C112—C111—C116	117.67 (13)	C216—C211—C212	117.50 (14)
C112—C111—C110	123.79 (13)	C216—C211—C210	119.37 (13)
C116—C111—C110	118.52 (12)	C212—C211—C210	123.09 (14)
C113—C112—C111	121.63 (13)	C213—C212—C211	120.74 (14)
C113—C112—H112	119.2	C213—C212—H212	119.6
C111—C112—H112	119.2	C211—C212—H212	119.6
C112—C113—C114	119.47 (13)	C212—C213—C214	120.78 (14)
C112—C113—H113	120.3	C212—C213—H213	119.6
C114—C113—H113	120.3	C214—C213—H213	119.6
O114—C114—C115	115.27 (12)	O214—C214—C215	124.73 (14)
O114—C114—C113	124.73 (13)	O214—C214—C213	115.69 (13)
C115—C114—C113	120.00 (13)	C215—C214—C213	119.58 (14)
C114—O114—C141	117.17 (11)	C214—O214—C241	117.66 (13)
O114—C141—H14A	109.5	O214—C241—H24A	109.5
O114—C141—H14B	109.5	O214—C241—H24B	109.5
H14A—C141—H14B	109.5	H24A—C241—H24B	109.5
O114—C141—H14C	109.5	O214—C241—H24C	109.5
H14A—C141—H14C	109.5	H24A—C241—H24C	109.5
H14B—C141—H14C	109.5	H24B—C241—H24C	109.5
C116—C115—C114	119.77 (13)	C214—C215—C216	119.21 (14)
C116—C115—H115	120.1	C214—C215—H215	120.4
C114—C115—H115	120.1	C216—C215—H215	120.4
C115—C116—C111	121.44 (13)	C215—C216—C211	122.18 (14)
C115—C116—H116	119.3	C215—C216—H216	118.9
C111—C116—H116	119.3	C211—C216—H216	118.9

C17a—O11—C12—O13	−18.20 (18)	C27a—O21—C22—O23	12.11 (16)
O11—C12—O13—C13a	17.28 (18)	O21—C22—O23—C23a	−11.89 (17)
C12—O13—C13a—C14	171.96 (16)	C22—O23—C23a—C24	−174.23 (15)
C12—O13—C13a—C17a	−9.52 (16)	C22—O23—C23a—C27a	7.09 (17)
O13—C13a—C14—C15	179.64 (13)	O23—C23a—C24—C25	−178.94 (14)
C17a—C13a—C14—C15	1.3 (2)	C27a—C23a—C24—C25	−0.4 (2)
C13a—C14—C15—N15	−178.46 (13)	C23a—C24—C25—N25	179.21 (13)
C13a—C14—C15—C16	1.1 (2)	C23a—C24—C25—C26	−0.1 (2)
N15—C15—C16—C17	176.49 (13)	N25—C25—C26—C27	−178.63 (13)
C14—C15—C16—C17	−3.0 (2)	C24—C25—C26—C27	0.7 (2)
N15—C15—C16—C18	−4.4 (2)	N25—C25—C26—C28	−0.7 (2)
C14—C15—C16—C18	176.13 (12)	C24—C25—C26—C28	178.60 (13)

C15—C16—C17—C17a	2.6 (2)	C25—C26—C27—C27a	−0.7 (2)
C18—C16—C17—C17a	−176.55 (13)	C28—C26—C27—C27a	−178.60 (13)
C16—C17—C17a—C13a	−0.3 (2)	C26—C27—C27a—O21	178.58 (13)
C16—C17—C17a—O11	−178.61 (13)	C26—C27—C27a—C23a	0.2 (2)
C14—C13a—C17a—C17	−1.8 (2)	C22—O21—C27a—C27	173.61 (15)
O13—C13a—C17a—C17	179.65 (12)	C22—O21—C27a—C23a	−7.80 (16)
C14—C13a—C17a—O11	176.87 (13)	C24—C23a—C27a—C27	0.4 (2)
O13—C13a—C17a—O11	−1.74 (16)	O23—C23a—C27a—C27	179.17 (13)
C12—O11—C17a—C17	−169.12 (16)	C24—C23a—C27a—O21	−178.29 (13)
C12—O11—C17a—C13a	12.39 (17)	O23—C23a—C27a—O21	0.46 (17)
C17—C16—C18—O18	−169.98 (13)	C25—C26—C28—O28	2.6 (2)
C15—C16—C18—O18	10.9 (2)	C27—C26—C28—O28	−179.54 (13)
C17—C16—C18—C19	11.0 (2)	C25—C26—C28—C29	−175.23 (13)
C15—C16—C18—C19	−168.15 (13)	C27—C26—C28—C29	2.7 (2)
O18—C18—C19—C110	10.5 (2)	O28—C28—C29—C210	−24.7 (2)
C16—C18—C19—C110	−170.48 (13)	C26—C28—C29—C210	153.13 (14)
C18—C19—C110—C111	−178.12 (13)	C28—C29—C210—C211	−176.05 (13)
C19—C110—C111—C112	−11.3 (2)	C29—C210—C211—C216	160.90 (15)
C19—C110—C111—C116	167.29 (15)	C29—C210—C211—C212	−16.8 (2)
C116—C111—C112—C113	−0.7 (2)	C216—C211—C212—C213	−0.1 (2)
C110—C111—C112—C113	177.92 (13)	C210—C211—C212—C213	177.68 (14)
C111—C112—C113—C114	0.4 (2)	C211—C212—C213—C214	−1.0 (2)
C112—C113—C114—O114	−179.72 (13)	C212—C213—C214—O214	−178.20 (14)
C112—C113—C114—C115	−0.2 (2)	C212—C213—C214—C215	1.3 (2)
C115—C114—O114—C141	−178.20 (13)	C215—C214—O214—C241	4.5 (2)
C113—C114—O114—C141	1.4 (2)	C213—C214—O214—C241	−176.09 (14)
O114—C114—C115—C116	179.80 (13)	O214—C214—C215—C216	178.92 (14)
C113—C114—C115—C116	0.2 (2)	C213—C214—C215—C216	−0.5 (2)
C114—C115—C116—C111	−0.5 (2)	C214—C215—C216—C211	−0.6 (2)
C112—C111—C116—C115	0.7 (2)	C212—C211—C216—C215	0.9 (2)
C110—C111—C116—C115	−177.97 (13)	C210—C211—C216—C215	−176.98 (14)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N15—H15*B*···O18	0.88	1.99	2.6451 (17)	130
N25—H25*A*···O28	0.88	1.95	2.6069 (19)	131
N25—H25*B*···O13	0.88	2.19	3.0586 (17)	170
C12—H12*A*···O18^{i}	0.99	2.27	3.221 (2)	161

Symmetry code: (i) *x*−1, *y*, *z*.

(III) 1-(6-Amino-1,3-benzodioxol-5-yl)-3-[4-(trifluoromethyl)phenyl]prop-2-en-1-one

Crystal data

$C_{17}H_{12}F_3NO_3$	*a* = 7.3420 (2) Å
M_r = 335.28	*b* = 10.9241 (3) Å
Triclinic, $P\overline{1}$	*c* = 18.7176 (5) Å
Hall symbol: -P 1	*α* = 85.0180 (11)°

β = 83.2280 (14)°
γ = 75.8180 (14)°
V = 1442.71 (7) Å^3
Z = 4
F(000) = 688
D_x = 1.544 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 6611 reflections
θ = 3.0–27.6°
μ = 0.13 mm^{-1}
T = 120 K
Block, red
0.60 × 0.60 × 0.50 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Radiation source: Bruker-Nonius FR591 rotating anode
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.920, T_{max} = 0.937

20172 measured reflections
6611 independent reflections
4510 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 27.6°, θ_{min} = 3.0°
h = −9→6
k = −14→13
l = −24→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.157
S = 1.04
6611 reflections
433 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0874P)^2 + 0.3568P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.77 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
F141	0.7571 (4)	0.6220 (2)	0.35107 (10)	0.0990 (9)
F142	0.8294 (3)	0.48202 (16)	0.43056 (11)	0.0721 (6)
F143	0.5784 (2)	0.6202 (2)	0.44684 (13)	0.0963 (9)
O11	0.8022 (2)	−0.19815 (14)	0.06107 (8)	0.0288 (4)
O13	0.6180 (2)	−0.26762 (14)	−0.01200 (8)	0.0271 (3)
O18	0.0728 (2)	0.13519 (14)	0.19270 (8)	0.0272 (3)
N15	0.0344 (2)	−0.00119 (17)	0.08840 (9)	0.0250 (4)
C12	0.8092 (3)	−0.2675 (2)	−0.00123 (12)	0.0266 (5)
C13a	0.5051 (3)	−0.17842 (18)	0.03120 (10)	0.0213 (4)
C14	0.3142 (3)	−0.13762 (19)	0.03553 (11)	0.0220 (4)
C15	0.2260 (3)	−0.04596 (19)	0.08561 (10)	0.0201 (4)
C16	0.3368 (3)	0.00141 (18)	0.12949 (10)	0.0193 (4)
C17	0.5368 (3)	−0.04738 (19)	0.12315 (10)	0.0211 (4)
C17a	0.6138 (3)	−0.13464 (19)	0.07453 (10)	0.0204 (4)
C18	0.2472 (3)	0.09856 (19)	0.18044 (10)	0.0205 (4)
C19	0.3685 (3)	0.15777 (19)	0.21796 (11)	0.0240 (5)
C110	0.2951 (3)	0.2558 (2)	0.25768 (11)	0.0240 (4)
C111	0.3977 (3)	0.32596 (19)	0.29524 (11)	0.0222 (4)

C112	0.5947 (3)	0.2958 (2)	0.29228 (11)	0.0228 (4)
C113	0.6856 (3)	0.3665 (2)	0.32679 (11)	0.0241 (5)
C114	0.5816 (3)	0.46842 (19)	0.36509 (11)	0.0231 (4)
C115	0.3861 (3)	0.4992 (2)	0.36956 (11)	0.0278 (5)
C116	0.2954 (3)	0.4280 (2)	0.33448 (12)	0.0273 (5)
C141	0.6809 (3)	0.5487 (2)	0.39885 (12)	0.0311 (5)
F241	0.7526 (2)	0.43372 (14)	0.03458 (7)	0.0458 (4)
F242	0.70842 (19)	0.29796 (12)	0.11946 (7)	0.0360 (3)
F243	0.88822 (18)	0.42096 (14)	0.13137 (9)	0.0471 (4)
O21	0.1650 (2)	1.18306 (16)	0.45514 (9)	0.0351 (4)
O23	−0.1122 (2)	1.24138 (15)	0.53049 (8)	0.0307 (4)
O28	−0.2621 (2)	0.92780 (16)	0.28116 (8)	0.0341 (4)
N25	−0.4550 (2)	1.02100 (18)	0.40185 (10)	0.0290 (4)
C22	0.0792 (3)	1.2534 (2)	0.51624 (12)	0.0298 (5)
C23a	−0.1391 (3)	1.16850 (19)	0.47839 (11)	0.0222 (4)
C24	−0.2995 (3)	1.13411 (19)	0.46896 (11)	0.0238 (4)
C25	−0.2946 (3)	1.05837 (19)	0.41070 (11)	0.0219 (4)
C26	−0.1245 (3)	1.01753 (19)	0.36568 (10)	0.0208 (4)
C27	0.0388 (3)	1.05749 (19)	0.37799 (11)	0.0227 (4)
C27a	0.0266 (3)	1.1324 (2)	0.43324 (11)	0.0231 (4)
C28	−0.1177 (3)	0.9359 (2)	0.30665 (11)	0.0233 (4)
C29	0.0670 (3)	0.85498 (19)	0.27934 (11)	0.0233 (4)
C210	0.0779 (3)	0.7751 (2)	0.22836 (11)	0.0234 (4)
C211	0.2466 (3)	0.68672 (19)	0.19792 (10)	0.0215 (4)
C212	0.4279 (3)	0.6868 (2)	0.21305 (11)	0.0230 (4)
C213	0.5837 (3)	0.6000 (2)	0.18387 (11)	0.0230 (4)
C214	0.5600 (3)	0.51135 (19)	0.13917 (11)	0.0233 (4)
C215	0.3809 (3)	0.5099 (2)	0.12317 (11)	0.0254 (5)
C216	0.2268 (3)	0.5978 (2)	0.15186 (11)	0.0252 (5)
C241	0.7261 (3)	0.4169 (2)	0.10704 (12)	0.0284 (5)
H12A	0.8844	−0.3553	0.0063	0.032*
H12B	0.8690	−0.2271	−0.0440	0.032*
H14	0.2421	−0.1696	0.0059	0.026*
H15A	−0.0252	0.0449	0.1297	0.030*
H15B	−0.0348	−0.0493	0.0664	0.030*
H17	0.6137	−0.0191	0.1524	0.025*
H19	0.5014	0.1245	0.2133	0.029*
H110	0.1614	0.2838	0.2624	0.029*
H112	0.6664	0.2260	0.2663	0.027*
H113	0.8195	0.3456	0.3244	0.029*
H115	0.3151	0.5682	0.3963	0.033*
H116	0.1614	0.4491	0.3372	0.033*
H22A	0.0817	1.3436	0.5063	0.036*
H22B	0.1492	1.2202	0.5586	0.036*
H24	−0.4115	1.1599	0.5004	0.029*
H25A	−0.4550	0.9877	0.3560	0.035*
H25B	−0.5700	1.0655	0.4273	0.035*
H27	0.1538	1.0323	0.3482	0.027*

H29	0.1787	0.8604	0.2985	0.028*
H210	−0.0371	0.7756	0.2096	0.028*
H212	0.4448	0.7471	0.2438	0.028*
H213	0.7067	0.6012	0.1944	0.028*
H215	0.3644	0.4490	0.0928	0.030*
H216	0.1044	0.5978	0.1400	0.030*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F141	0.173 (2)	0.1098 (17)	0.0615 (12)	−0.1166 (18)	−0.0459 (13)	0.0215 (11)
F142	0.0742 (12)	0.0468 (10)	0.1064 (15)	−0.0045 (9)	−0.0628 (11)	−0.0210 (10)
F143	0.0447 (10)	0.1122 (17)	0.147 (2)	−0.0189 (11)	0.0043 (11)	−0.1106 (16)
O11	0.0201 (7)	0.0332 (9)	0.0337 (8)	−0.0036 (6)	−0.0005 (6)	−0.0163 (7)
O13	0.0238 (8)	0.0274 (8)	0.0316 (8)	−0.0060 (6)	0.0002 (6)	−0.0149 (7)
O18	0.0200 (7)	0.0285 (8)	0.0335 (8)	−0.0042 (6)	−0.0012 (6)	−0.0112 (7)
N15	0.0196 (9)	0.0291 (10)	0.0284 (10)	−0.0064 (7)	−0.0050 (7)	−0.0083 (8)
C12	0.0243 (11)	0.0268 (12)	0.0297 (11)	−0.0061 (9)	0.0003 (9)	−0.0103 (9)
C13a	0.0273 (11)	0.0166 (10)	0.0204 (10)	−0.0063 (8)	0.0006 (8)	−0.0042 (8)
C14	0.0245 (10)	0.0228 (11)	0.0221 (10)	−0.0101 (9)	−0.0044 (8)	−0.0044 (8)
C15	0.0202 (10)	0.0206 (10)	0.0212 (10)	−0.0078 (8)	−0.0033 (8)	−0.0012 (8)
C16	0.0198 (10)	0.0201 (10)	0.0197 (10)	−0.0071 (8)	−0.0019 (8)	−0.0042 (8)
C17	0.0218 (10)	0.0210 (10)	0.0230 (10)	−0.0081 (8)	−0.0031 (8)	−0.0049 (8)
C17a	0.0159 (9)	0.0219 (10)	0.0243 (10)	−0.0062 (8)	−0.0011 (8)	−0.0037 (8)
C18	0.0243 (11)	0.0184 (10)	0.0198 (10)	−0.0064 (8)	−0.0030 (8)	−0.0010 (8)
C19	0.0210 (10)	0.0242 (11)	0.0278 (11)	−0.0043 (9)	−0.0047 (8)	−0.0071 (9)
C110	0.0209 (10)	0.0245 (11)	0.0281 (11)	−0.0068 (9)	−0.0014 (8)	−0.0074 (9)
C111	0.0256 (11)	0.0196 (10)	0.0220 (10)	−0.0061 (9)	−0.0013 (8)	−0.0033 (8)
C112	0.0249 (11)	0.0208 (10)	0.0219 (10)	−0.0022 (8)	−0.0034 (8)	−0.0047 (8)
C113	0.0240 (11)	0.0238 (11)	0.0237 (11)	−0.0020 (9)	−0.0055 (8)	−0.0039 (9)
C114	0.0273 (11)	0.0202 (10)	0.0232 (10)	−0.0057 (9)	−0.0048 (8)	−0.0053 (8)
C115	0.0305 (12)	0.0246 (11)	0.0293 (12)	−0.0057 (9)	−0.0017 (9)	−0.0112 (9)
C116	0.0234 (11)	0.0270 (12)	0.0323 (12)	−0.0054 (9)	−0.0001 (9)	−0.0116 (9)
C141	0.0348 (13)	0.0263 (12)	0.0343 (13)	−0.0070 (10)	−0.0092 (10)	−0.0073 (10)
F241	0.0530 (9)	0.0404 (9)	0.0302 (8)	0.0075 (7)	0.0124 (6)	−0.0019 (6)
F242	0.0409 (8)	0.0208 (7)	0.0419 (8)	0.0000 (6)	0.0022 (6)	−0.0067 (6)
F243	0.0239 (7)	0.0449 (9)	0.0719 (10)	0.0018 (6)	−0.0050 (7)	−0.0294 (8)
O21	0.0249 (8)	0.0418 (10)	0.0436 (10)	−0.0131 (7)	0.0026 (7)	−0.0237 (8)
O23	0.0269 (8)	0.0326 (9)	0.0348 (9)	−0.0083 (7)	0.0012 (7)	−0.0169 (7)
O28	0.0219 (8)	0.0437 (10)	0.0363 (9)	−0.0003 (7)	−0.0075 (7)	−0.0165 (7)
N25	0.0170 (9)	0.0371 (11)	0.0341 (10)	−0.0086 (8)	0.0046 (7)	−0.0131 (9)
C22	0.0274 (11)	0.0280 (12)	0.0355 (13)	−0.0055 (9)	−0.0046 (9)	−0.0119 (10)
C23a	0.0252 (11)	0.0186 (10)	0.0220 (10)	−0.0030 (8)	−0.0013 (8)	−0.0047 (8)
C24	0.0211 (10)	0.0231 (11)	0.0251 (11)	−0.0029 (9)	0.0052 (8)	−0.0063 (9)
C25	0.0194 (10)	0.0216 (11)	0.0240 (10)	−0.0034 (8)	−0.0010 (8)	−0.0034 (8)
C26	0.0204 (10)	0.0198 (10)	0.0209 (10)	−0.0022 (8)	−0.0022 (8)	−0.0019 (8)
C27	0.0186 (10)	0.0242 (11)	0.0238 (10)	−0.0022 (8)	0.0009 (8)	−0.0052 (9)
C27a	0.0183 (10)	0.0243 (11)	0.0277 (11)	−0.0048 (8)	−0.0032 (8)	−0.0066 (9)

C28	0.0204 (10)	0.0247 (11)	0.0234 (10)	−0.0025 (8)	−0.0018 (8)	−0.0029 (9)
C29	0.0205 (10)	0.0255 (11)	0.0237 (10)	−0.0040 (9)	−0.0036 (8)	−0.0035 (9)
C210	0.0216 (10)	0.0242 (11)	0.0237 (11)	−0.0028 (9)	−0.0027 (8)	−0.0035 (9)
C211	0.0249 (10)	0.0187 (10)	0.0196 (10)	−0.0030 (8)	−0.0013 (8)	−0.0012 (8)
C212	0.0264 (11)	0.0221 (11)	0.0217 (10)	−0.0066 (9)	−0.0032 (8)	−0.0045 (8)
C213	0.0221 (10)	0.0228 (11)	0.0241 (10)	−0.0048 (9)	−0.0021 (8)	−0.0033 (8)
C214	0.0260 (11)	0.0184 (10)	0.0229 (10)	−0.0006 (8)	−0.0014 (8)	−0.0022 (8)
C215	0.0277 (11)	0.0215 (11)	0.0270 (11)	−0.0034 (9)	−0.0036 (9)	−0.0078 (9)
C216	0.0237 (11)	0.0241 (11)	0.0284 (11)	−0.0043 (9)	−0.0048 (9)	−0.0058 (9)
C241	0.0301 (12)	0.0247 (12)	0.0287 (12)	−0.0026 (9)	−0.0014 (9)	−0.0058 (9)

Geometric parameters (Å, º)

O11—C17a	1.391 (2)	O21—C27a	1.386 (2)
O11—C12	1.434 (2)	O21—C22	1.429 (3)
C12—O13	1.442 (2)	C22—O23	1.436 (3)
C12—H12A	0.99	C22—H22A	0.99
C12—H12B	0.99	C22—H22B	0.99
O13—C13a	1.369 (2)	O23—C23a	1.372 (2)
C13a—C14	1.358 (3)	C23a—C24	1.355 (3)
C13a—C17a	1.390 (3)	C23a—C27a	1.390 (3)
C14—C15	1.415 (3)	C24—C25	1.417 (3)
C14—H14	0.95	C24—H24	0.95
C15—N15	1.368 (2)	C25—N25	1.369 (3)
C15—C16	1.429 (3)	C25—C26	1.419 (3)
N15—H15A	0.9599	N25—H25A	0.9599
N15—H15B	0.9599	N25—H25B	0.9598
C16—C17	1.428 (3)	C26—C27	1.421 (3)
C16—C18	1.468 (3)	C26—C28	1.468 (3)
C17—C17a	1.350 (3)	C27—C27a	1.355 (3)
C17—H17	0.95	C27—H27	0.95
C18—O18	1.244 (2)	C28—O28	1.237 (2)
C18—C19	1.488 (3)	C28—C29	1.486 (3)
C19—C110	1.323 (3)	C29—C210	1.331 (3)
C19—H19	0.95	C29—H29	0.95
C110—C111	1.466 (3)	C210—C211	1.461 (3)
C110—H110	0.95	C210—H210	0.95
C111—C116	1.396 (3)	C211—C212	1.393 (3)
C111—C112	1.398 (3)	C211—C216	1.397 (3)
C112—C113	1.379 (3)	C212—C213	1.385 (3)
C112—H112	0.95	C212—H212	0.95
C113—C114	1.389 (3)	C213—C214	1.388 (3)
C113—H113	0.95	C213—H213	0.95
C114—C115	1.385 (3)	C214—C215	1.387 (3)
C114—C141	1.490 (3)	C214—C241	1.494 (3)
C141—F143	1.287 (3)	C241—F242	1.335 (2)
C141—F141	1.316 (3)	C241—F243	1.336 (3)
C141—F142	1.326 (3)	C241—F241	1.350 (3)

C115—C116	1.387 (3)	C215—C216	1.380 (3)
C115—H115	0.95	C215—H215	0.95
C116—H116	0.95	C216—H216	0.95
C17a—O11—C12	105.14 (15)	C27a—O21—C22	106.12 (15)
O11—C12—O13	107.71 (16)	O21—C22—O23	108.18 (16)
O11—C12—H12A	110.2	O21—C22—H22A	110.1
O13—C12—H12A	110.2	O23—C22—H22A	110.1
O11—C12—H12B	110.2	O21—C22—H22B	110.1
O13—C12—H12B	110.2	O23—C22—H22B	110.1
H12A—C12—H12B	108.5	H22A—C22—H22B	108.4
C13a—O13—C12	105.69 (14)	C23a—O23—C22	106.25 (16)
C14—C13a—O13	127.59 (18)	C24—C23a—O23	127.64 (19)
C14—C13a—C17a	122.32 (18)	C24—C23a—C27a	122.38 (18)
O13—C13a—C17a	110.05 (17)	O23—C23a—C27a	109.98 (17)
C13a—C14—C15	117.85 (17)	C23a—C24—C25	117.79 (18)
C13a—C14—H14	121.1	C23a—C24—H24	121.1
C15—C14—H14	121.1	C25—C24—H24	121.1
N15—C15—C14	118.10 (17)	N25—C25—C24	118.06 (18)
N15—C15—C16	121.58 (18)	N25—C25—C26	121.56 (18)
C14—C15—C16	120.27 (18)	C24—C25—C26	120.29 (18)
C15—N15—H15A	116.2	C25—N25—H25A	114.7
C15—N15—H15B	117.3	C25—N25—H25B	116.6
H15A—N15—H15B	118.9	H25A—N25—H25B	121.1
C17—C16—C15	118.97 (17)	C25—C26—C27	119.24 (18)
C17—C16—C18	120.30 (16)	C25—C26—C28	120.35 (17)
C15—C16—C18	120.73 (17)	C27—C26—C28	120.41 (17)
C17a—C17—C16	118.38 (17)	C27a—C27—C26	118.50 (18)
C17a—C17—H17	120.8	C27a—C27—H27	120.8
C16—C17—H17	120.8	C26—C27—H27	120.8
C17—C17a—C13a	122.18 (18)	C27—C27a—O21	128.79 (18)
C17—C17a—O11	128.33 (17)	C27—C27a—C23a	121.75 (18)
C13a—C17a—O11	109.44 (17)	O21—C27a—C23a	109.46 (17)
O18—C18—C16	122.49 (17)	O28—C28—C26	122.06 (18)
O18—C18—C19	118.40 (18)	O28—C28—C29	118.85 (18)
C16—C18—C19	119.09 (17)	C26—C28—C29	119.01 (17)
C110—C19—C18	121.33 (19)	C210—C29—C28	120.89 (18)
C110—C19—H19	119.3	C210—C29—H29	119.6
C18—C19—H19	119.3	C28—C29—H29	119.6
C19—C110—C111	127.1 (2)	C29—C210—C211	127.38 (19)
C19—C110—H110	116.4	C29—C210—H210	116.3
C111—C110—H110	116.4	C211—C210—H210	116.3
C116—C111—C112	118.63 (18)	C212—C211—C216	118.20 (19)
C116—C111—C110	118.94 (19)	C212—C211—C210	122.71 (18)
C112—C111—C110	122.43 (19)	C216—C211—C210	119.09 (18)
C113—C112—C111	120.51 (19)	C213—C212—C211	120.79 (19)
C113—C112—H112	119.7	C213—C212—H212	119.6
C111—C112—H112	119.7	C211—C212—H212	119.6

C112—C113—C114	120.08 (19)	C212—C213—C214	119.89 (19)
C112—C113—H113	120.0	C212—C213—H213	120.1
C114—C113—H113	120.0	C214—C213—H213	120.1
C115—C114—C113	120.47 (18)	C215—C214—C213	120.26 (19)
C115—C114—C141	119.70 (19)	C215—C214—C241	118.92 (18)
C113—C114—C141	119.77 (19)	C213—C214—C241	120.82 (19)
F143—C141—F141	107.2 (2)	F242—C241—F243	106.69 (18)
F143—C141—F142	105.9 (2)	F242—C241—F241	104.94 (17)
F141—C141—F142	102.2 (2)	F243—C241—F241	106.09 (17)
F143—C141—C114	115.1 (2)	F242—C241—C214	112.93 (18)
F141—C141—C114	112.47 (19)	F243—C241—C214	113.07 (17)
F142—C141—C114	113.03 (19)	F241—C241—C214	112.50 (18)
C114—C115—C116	119.2 (2)	C216—C215—C214	119.36 (19)
C114—C115—H115	120.4	C216—C215—H215	120.3
C116—C115—H115	120.4	C214—C215—H215	120.3
C115—C116—C111	121.1 (2)	C215—C216—C211	121.49 (19)
C115—C116—H116	119.5	C215—C216—H216	119.3
C111—C116—H116	119.5	C211—C216—H216	119.3

C17a—O11—C12—O13	−14.0 (2)	C27a—O21—C22—O23	0.9 (2)
O11—C12—O13—C13a	13.3 (2)	O21—C22—O23—C23a	−1.3 (2)
C12—O13—C13a—C14	174.7 (2)	C22—O23—C23a—C24	−178.4 (2)
C12—O13—C13a—C17a	−7.4 (2)	C22—O23—C23a—C27a	1.2 (2)
O13—C13a—C14—C15	178.55 (19)	O23—C23a—C24—C25	179.64 (19)
C17a—C13a—C14—C15	0.9 (3)	C27a—C23a—C24—C25	0.1 (3)
C13a—C14—C15—N15	177.71 (18)	C23a—C24—C25—N25	178.58 (19)
C13a—C14—C15—C16	0.3 (3)	C23a—C24—C25—C26	1.9 (3)
N15—C15—C16—C17	−178.80 (18)	N25—C25—C26—C27	−178.95 (19)
C14—C15—C16—C17	−1.5 (3)	C24—C25—C26—C27	−2.4 (3)
N15—C15—C16—C18	1.3 (3)	N25—C25—C26—C28	1.6 (3)
C14—C15—C16—C18	178.61 (18)	C24—C25—C26—C28	178.13 (19)
C15—C16—C17—C17a	1.5 (3)	C25—C26—C27—C27a	0.8 (3)
C18—C16—C17—C17a	−178.59 (18)	C28—C26—C27—C27a	−179.72 (19)
C16—C17—C17a—C13a	−0.4 (3)	C26—C27—C27a—O21	−179.6 (2)
C16—C17—C17a—O11	−177.34 (19)	C26—C27—C27a—C23a	1.2 (3)
C14—C13a—C17a—C17	−0.9 (3)	C22—O21—C27a—C27	−179.4 (2)
O13—C13a—C17a—C17	−178.90 (18)	C22—O21—C27a—C23a	−0.2 (2)
C14—C13a—C17a—O11	176.60 (18)	C24—C23a—C27a—C27	−1.8 (3)
O13—C13a—C17a—O11	−1.4 (2)	O23—C23a—C27a—C27	178.65 (19)
C12—O11—C17a—C17	−173.1 (2)	C24—C23a—C27a—O21	178.93 (19)
C12—O11—C17a—C13a	9.6 (2)	O23—C23a—C27a—O21	−0.7 (2)
C17—C16—C18—O18	−173.09 (19)	C25—C26—C28—O28	20.2 (3)
C15—C16—C18—O18	6.8 (3)	C27—C26—C28—O28	−159.2 (2)
C17—C16—C18—C19	8.2 (3)	C25—C26—C28—C29	−156.36 (19)
C15—C16—C18—C19	−171.93 (18)	C27—C26—C28—C29	24.2 (3)
O18—C18—C19—C110	−6.9 (3)	O28—C28—C29—C210	0.0 (3)
C16—C18—C19—C110	171.9 (2)	C26—C28—C29—C210	176.7 (2)
C18—C19—C110—C111	−177.59 (19)	C28—C29—C210—C211	−177.65 (19)

C19—C110—C111—C116	179.6 (2)	C29—C210—C211—C212	−8.6 (3)
C19—C110—C111—C112	0.7 (3)	C29—C210—C211—C216	171.2 (2)
C116—C111—C112—C113	−0.7 (3)	C216—C211—C212—C213	−0.8 (3)
C110—C111—C112—C113	178.18 (19)	C210—C211—C212—C213	179.01 (19)
C111—C112—C113—C114	0.2 (3)	C211—C212—C213—C214	−0.2 (3)
C112—C113—C114—C115	0.6 (3)	C212—C213—C214—C215	0.5 (3)
C112—C113—C114—C141	−176.65 (19)	C212—C213—C214—C241	179.96 (19)
C115—C114—C141—F143	18.8 (3)	C215—C214—C241—F242	−53.3 (3)
C113—C114—C141—F143	−164.0 (2)	C213—C214—C241—F242	127.2 (2)
C115—C114—C141—F141	−104.3 (3)	C215—C214—C241—F243	−174.60 (18)
C113—C114—C141—F141	72.9 (3)	C213—C214—C241—F243	5.9 (3)
C115—C114—C141—F142	140.6 (2)	C215—C214—C241—F241	65.2 (3)
C113—C114—C141—F142	−42.1 (3)	C213—C214—C241—F241	−114.2 (2)
C113—C114—C115—C116	−0.9 (3)	C213—C214—C215—C216	0.3 (3)
C141—C114—C115—C116	176.4 (2)	C241—C214—C215—C216	−179.2 (2)
C114—C115—C116—C111	0.3 (3)	C214—C215—C216—C211	−1.3 (3)
C112—C111—C116—C115	0.5 (3)	C212—C211—C216—C215	1.6 (3)
C110—C111—C116—C115	−178.5 (2)	C210—C211—C216—C215	−178.22 (19)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N15—H15*A*···O18	0.96	1.91	2.637 (2)	131
N15—H15*B*···O11^{i}	0.96	2.26	3.161 (2)	156
N25—H25*A*···O28	0.96	1.91	2.662 (2)	134
N25—H25*B*···O21^{i}	0.96	2.09	3.024 (2)	165

Symmetry code: (i) *x*−1, *y*, *z*.