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Functionalized 3-(5-aryloxy-3-methyl-1-phenyl-1H-pyrazol-4-yl)-1-(4-substituted-phenyl)prop-2-en-1-ones: synthetic pathway, and the structures of six examples

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Five examples each of 3-(5-aryloxy-3-methyl-1-phenyl-1H-pyrazol-4-yl)-1-[4-(prop-2-yn-1-yloxy)phenyl]prop-2-en-1-ones and the corresponding 1-(4-azido-phenyl)-3-(5-aryloxy-3-methyl-1-phenyl-1H-pyrazol-4-yl)prop-2-en-1-ones have been synthesized in a highly efficient manner, starting from a common source precursor, and structures have been determined for three examples of each type. In each of 3-[5-(2-chlorophenoxy)-3-methyl-1-phenyl-1H-pyrazol-4-yl]-1-[4-(prop-2-yn-1-yloxy)phenyl]prop-2-en-1-one, $C_{28}H_{21}ClN_2O_3$, (Ib), the isomeric 3-[5-(2-chlorophenoxy)-3-methyl-1-phenyl-1H-pyrazol-4-yl]-1-[4-(prop-2-yn-1-yloxy)phenyl]prop-2-en-1-one, (Ic), and 3-[3-methyl-5-(naphthalen-2-yloxy)-1-phenyl-1H-pyrazol-4-yl]-1-[4-(prop-2-ynyloxy)phenyl]prop-2-en-1-one, $C_{32}H_{24}N_2O_3$, (Ie), the molecules are linked into chains of rings, formed by two independent C—H···O hydrogen bonds in (Ib) and by a combination of C—H···O and C—H···π(arene) hydrogen bonds in each of (Ic) and (Ie). There are no direction-specific intermolecular interactions in the structure of 1-(4-azidophenyl)-3-[3-methyl-5-(2-methylphenoxy)-1-phenyl-1H-pyrazol-4-yl]prop-2-en-1-one, $C_{26}H_{21}N_5O_2$, (IIa). In 1-(4-azidophenyl)-3-[5-(2,4-dichlorophenoxy)-3-methyl-1-phenyl-1H-pyrazol-4-yl]prop-2-en-1-one, $C_{25}H_{17}Cl_2N_5O_2$, (IId), the dichlorophenyl group is disordered over two sets of atomic sites having occupancies 0.55 (4) and 0.45 (4), and the molecules are linked by a single C—H···O hydrogen bond to form cyclic, centrosymmetric $R_2^2(20)$ dimers. Similar dimers are formed in 1-(4-azidophenyl)-3-[3-methyl-5-(naphthalen-2-yloxy)-1-phenyl-1H-pyrazol-4-yl]prop-2-en-1-one, $C_{29}H_{21}N_5O_2$, (IIe), but here the dimers are linked into a chain of rings by two independent C—H..π(arene) hydrogen bonds. Comparisons are made between the molecular conformations within both series of compounds.

1. Chemical context

Chalcones, 1.3-disubstituted-prop-2-en-1-ones of type R^1COCH=CHR^2, exhibit a wide range of biological activities, particularly when they incorporate functionalized substituents; these include anticancer (Murthy et al., 2013), antimalarial (Mishra et al., 2008; Yadav et al., 2012), antitripanosomal (Carvalho et al., 2012) and antiviral (Sharma et al., 2011) activities. With these properties in mind, we have developed a versatile and efficient route to functionalized chalcones, which are themselves the basis for further

elaboration to provide a wide range of multiply substituted chalcones. Here we report the synthesis and characterization of five 3-(5-aryloxy-3-methyl-1-phenyl-1H-pyrazol-4-yl)-1-(4-(prop-2-yn-1- yloxy)phenyl)prop-2-en-1-ones (I) and a corresponding series of five 1-(4-azidophenyl)-3-(3-methyl-1-phenyl-5-(aryloxy)-1H-pyrazol-4-yl)-prop-2-en-1-ones (II), together with the structures of a representative selection of three examples of each type, namely (Ib), (Ic) and (Ie) and (IIa), (IId) and (IIe) (Figs. 1–6). The compounds of types (I) and (II) were prepared using a common synthetic scheme starting from the commercially available 3-methyl-1-phenyl-1H-pyrazole, which was readily converted, under Vilsmaeier–Haack conditions, to the key precursor 5-chloro-3-methyl-1-phenyl-1H-pyrazole-4-carbaldehyde (A) (Fig. 7), which was then converted to a series of 5-aryloxy derivatives (B), by reaction with substituted phenols under basic conditions as previously described (Kiran Kumar et al., 2019). The 5-aryloxy compounds (B) were then condensed with 1-[4-(prop-2-yn-1-yloxy)phenyl]ethan-1-one to give the products (Ia)–(Ie) (Fig. 7) or with 1-(4-azidophenyl)ethan-1-one to give the corresponding series of products (IIa)–(IIe). Thus the synthesis of these two matched series of products (I) and (II) from common precursors, is highly efficient. The presence of the alkyne unit in the type (I) products and of the azido unit in the type (II) products means that a small library is now available for use in Huisgen-type cycloaddition reactions to form bis(-chalcone)-substituted 1,2,3-triazoles. Such highly functionalized triazoles are an attractive synthetic target as 1,2,3-triazoles, which exhibit a very wide range of biological activity of potential medicinal values (Kharb et al., 2011; Dheer et al., 2017).

Figure 1
The molecular structure of compound (Ib) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

Figure 2
The molecular structure of compound (Ic) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

(Ib) R = 2-ClC$_6$H$_4$
(Ic) R = 4-ClC$_6$H$_4$
(Ie) R = 2-naphthyl

(IIa) R = 2-MeC$_6$H$_4$
(IId) R = 2,4-Cl$_2$C$_6$H$_3$
(IIe) R = 2-naphthyl

Figure 3
The molecular structure of compound (Ie) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

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

2. Structural commentary

Compounds (Ib) and (Ic) are geometrical isomers (Figs. 1 and 2), although they are not isomorphous (Table 3). Although the constitutions of compounds (Ie) and (IIe) differ only in the identity of the small substituent at atom C14 (Figs. 3 and 6), these compounds crystallize in different space groups (Table 3).

Figure 5
The molecular structure of compound (IId) showing the atom-labelling scheme, and the disorder in the 2,4-dichlorophenyl group. The major disorder component is drawn using full lines and the minor disorder component is drawn using broken lines. Displacement ellipsoids are drawn at the 30% probability level.
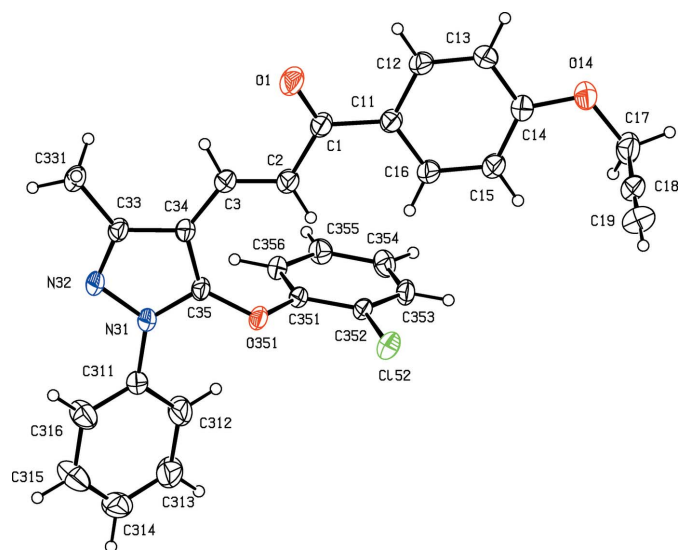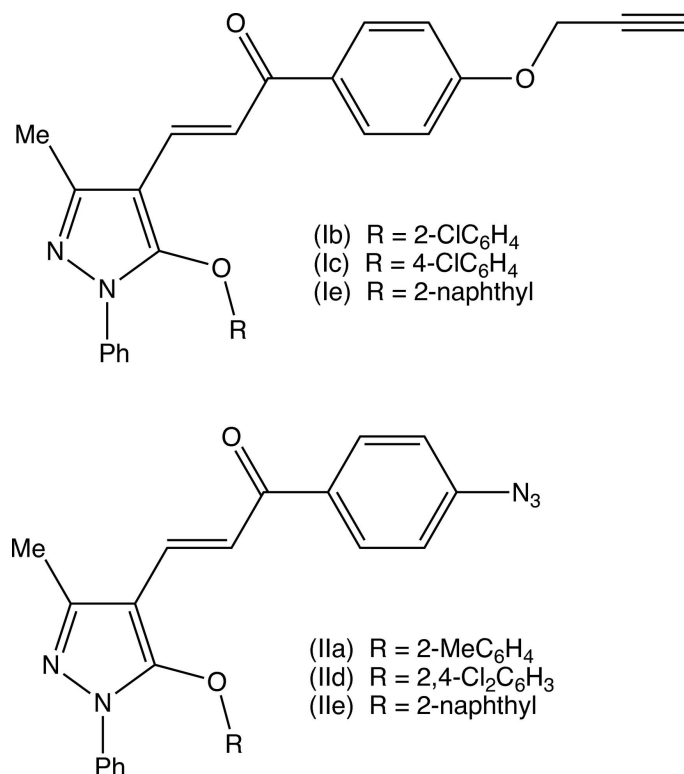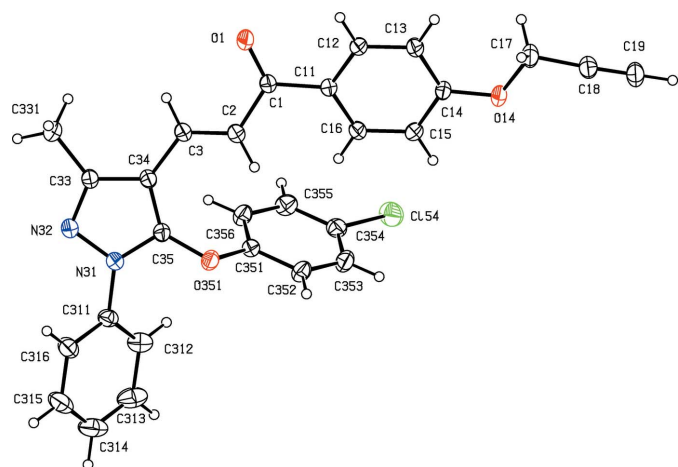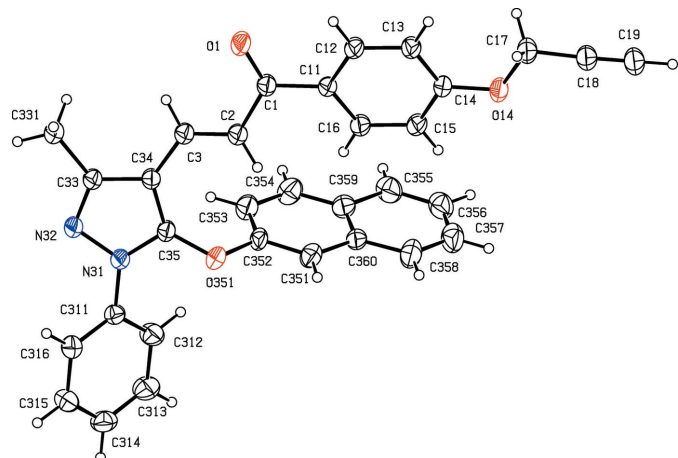

Figure 6
The molecular structure of compound (IIe) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.
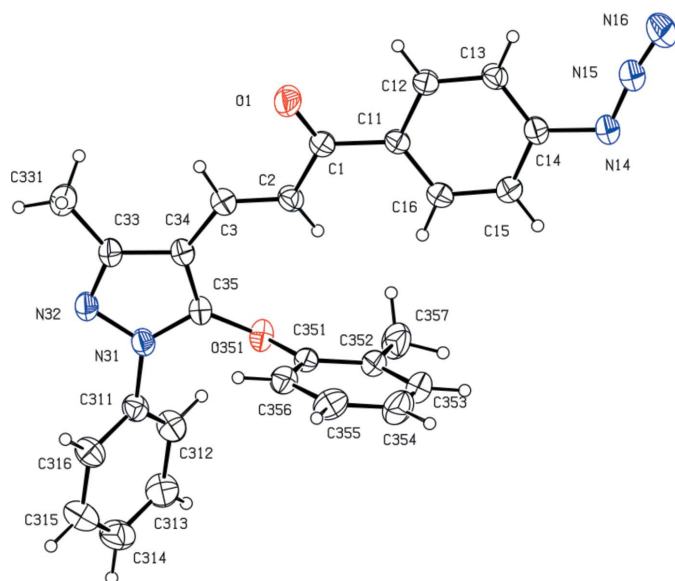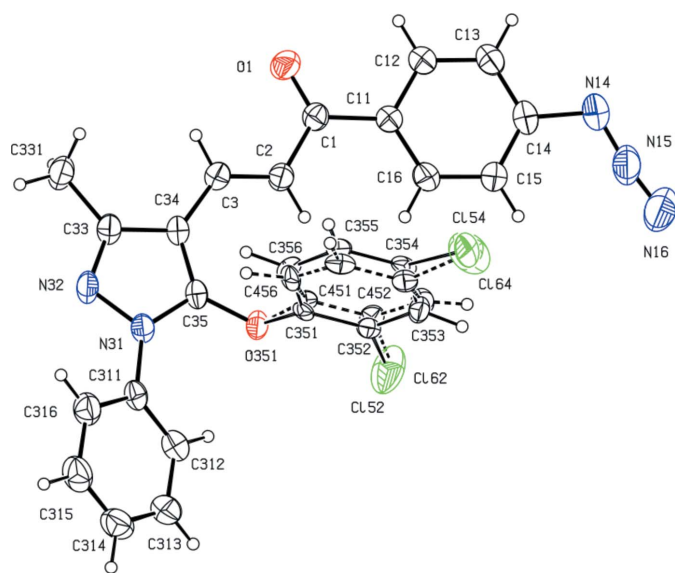
In each of the compounds reported here, the central core of the molecules, comprising the pyrazole ring and the adjacent prop-2-en-1-one unit is very nearly planar. However, the three substituents at atoms C1, N31 and C35 (Figs. 1–6) are all twisted out of the plane of the molecular core, as indicated by the relevant torsional angles (Table 1). None of the molecules, therefore, exhibits any internal symmetry, so that all are conformationally chiral: however, the space groups confirm that they have all crystallized as conformational racemates. In each case, the reference molecule was selected to be one having a positive sign for the torsional angle N32—N31—C311—C312 (Table 1).

In compound (IId), the 2,4-dichlorophenoxy substituent was found to be disordered over two sets of atomic sites, having occupancies 0.55 (4) and 0.45 (4) (Fig. 5). The disorder involves slight differences in the torsional angles around the bond C35-O351 (Fig. 5), thus C34—C35—O351—Cx51 = 78.3 (16)° when x = 3, and 65.0 (12)° when x = 4: on the other hand, the torsional angles around the bonds O351—Cx51 (x = 3 or 4) are the same within experimental uncertainty, thus C35—O351—Cx51—Cx52 = −164 (2)° for x = 3 and −163.6 (18)° when x = 4.

The orientation of the OCH$_2$CCH substituent relative to the adjacent aryl ring is different in compound (Ib), as compared with (Ic) and (Ie) (Table 1, Figs. 1–3) and similarly the orientation of the azido substituent is different in (IId), as compared with (IIa) and (IIe). In the case of the type (I) compounds, it is tempting to associate the observed differences with the different patterns of hydrogen bonding (Table 2, and Section 3, below), where atom O14 acts as an acceptor only in (Ib) while atom C19 acts as a donor in (Ic) and (Ie) but not in (Ib). However, in none of the type (II) compounds do any of the N atoms of the azido unit act as a hydrogen-bond acceptor. Hence, in these compounds, at least, the role of this

Table 1
Selected torsional angles (°) for compounds (Ib), (Ic), (Ie), (IIa), (IId) and (IIe).

Parameter	(Ib)	(Ic)	(Ie)	(IIa)	(IId)	(IIe)
N32—N31—C311—C312	151.1 (3)	137.0 (2)	139.9 (3)	135.1 (2)	149.6 (4)	140.9 (3)
C2—C1—C11—C12	168.8 (2)	−163.4 (2)	−162.8 (3)	166.8 (2)	−172.7 (4)	−171.4 (3)
C13—C14—O14—C17	169.8 (2)	3.5 (3)	−0.4 (4)			
C14—O14—C17—C18	−68.7 (3)	−177.1 (2)	−174.7 (3)			
C13—C14—N14—N15				−2.7 (3)	−172.6 (5)	−5.0 (6)
C34—C35—O351—C351	−76.7 (3)	−69.3 (3)		70.1 (3)	78.3 (16)	
C34—C35—O451—C451					65.0 (12)	
C35—O351—C351—C352	157.6 (2)	169.5 (2)		−159.6 (2)	−164 (2)	
C35—O451—C451—C45					−163.6 (18)	
C34—C35—O351—C352			−70.6 (4)			−71.1 (5)
C35—O351—C352—C351			161.8 (2)			150.9 (3)

substituent may be mainly that of a space filler, with the conformation adopted being that which most effectively fills any available space between the molecules.

3. Supramolecular features

The supramolecular assembly in the structures reported here is dominated by C—H···O hydrogen bonds (Table 2), along with C—H···π(arene) hydrogen bonds in compounds (Ic), (Ie) and (IIe). However, in none of the compounds containing chlorophenoxy substituents, [(Ib), (Ic) and (IId)] are there any short C—Cl···π(arene) contacts (cf. Imai *et al.*, 2008).

There are two C—H···O hydrogen bonds in the structure of compound (Ib) (Table 2), and together these link the molecules into a chain of centrosymmetric rings running parallel to the [010] direction, with rings of $R_2^2(20)$ type (Etter, 1990; Etter *et al.*, 1990; Bernstein *et al.*, 1995) centred at (1, n + 0.5, 0.5) alternating with rings of $R_2^2(30)$ type centred at (1, n, 0.5), where n represents an integer in each case (Fig. 7). In addition to an intramolecular C—H···π(arene) hydrogen bond (Table 2), the structure of compound (Ic), isomeric with (Ib), contains two hydrogen bonds, one each of C—H···O and

C—H···π(arene) types. The C—H···O hydrogen bond links molecules related by translation into a $C(11)$ running parallel to the [110] direction, and inversion-related pairs of such chains are linked by the C—H···π(arene) hydrogen bond to form a chain of rings running parallel to [110] (Fig. 8). Although there are no intramolecular hydrogen bonds in the

Table 2
Hydrogen bonds and short intra- and inter-molecular contacts (Å, °) for compounds (Ib), (Ic), (Ie), (IIa), (IId) and (IIe).

$Cg1$, $Cg2$, $Cg3$ and $Cg4$ represent the centroids of the rings C311–C316), (C351–C356), (C351–C354/C359/C360) and (C355–C360), respectively

Compound	D—H···A	D—A	H···A	D···A	D—H···A
(Ib)	C355—H355···O14[i]	0.93	2.59	3.468 (4)	158
	C356—H356···O1[ii]	0.93	2.51	3.360 (4)	152
(Ic)	C19—H19···O1[iii]	0.93	2.25	3.161 (3)	165
	C16—H16···Cg2	0.93	2.98	3.882 (2)	165
	C356—H356···Cg1[iv]	0.93	2.88	3.685 (2)	146
(Ie)	C19—H19···O1[v]	0.93	2.32	3.233 (5)	165
	C353—H353···Cg1[vi]	0.93	2.86	3.708 (3)	152
(IIa)	C357—H35B···O1[vii]	0.96	2.51	3.396 (4)	154
(IId)	C356—H356···O1[viii]	0.93	2.32	3.115 (18)	143
	C456—H456···O1[viii]	0.93	2.47	3.21 (2)	137
(IIe)	C353—H353···O1[ix]	0.93	2.47	3.288 (4)	147
	C12—H12···Cg3[x]	0.93	2.93	3.761 (4)	150
	C13—H13···Cg4[x]	0.93	2.73	3.547 (4)	148

Symmetry codes: (i) 2 − x, −y, 1 − z; (ii) 2 − x, 1 − y, 1 − z; (iii) 1 + x, 1 + y, z; (iv) 1 − x, −y, 2 − z; (v) −1 + x, 1 + y, z; (vi) 1 − x, −y, 1 − z; (vii) 1 − x, 1 − y, 2 − z; (viii) 1 − x, 2 − y, 1 − z; (ix) 1 − x, 1 − y, −z; (x) −1 + x, y, z.

Figure 7
Part of the crystal structure of compound (Ib) showing the formation of a chain of centrosymmetric rings parallel to [010]. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, the H atoms not involved in the motifs shown have been omitted.

H···O hydrogen bond in the structure of compound (IId), with fairly similar dimensions for each of the two disorder components. Hence it is necessary to consider only the major disorder component, where the inversion-related pairs of molecules are linked into cyclic, centrosymmetric $R_2^2(20)$ dimers (Fig. 10). In the structure of compound (IIe), inversion-related pairs of molecules are linked by paired C—H···O hydrogen bonds to form cyclic, centrosymmetric $R_2^2(20)$ dimers, which in turn are linked into a chain of rings running parallel to the [100] direction (Fig. 11) by the combined action of two C—H···π(arene) hydrogen bonds, which utilize both rings of the 2-naphthyl substituent as the acceptors.

Thus, the supramolecular assembly in the isomeric pair of compounds (Ib) and (Ic) is different in terms of the hydrogen bonds involved (Table 2), although chains of rings, different in

Figure 8
Part of the crystal structure of compound (Ic) showing the formation of a chain of rings parallel to [110]. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, the H atoms not involved in the motifs shown have been omitted.

structure of compound (Ie), the intermolecular hydrogen bonds (Table 2) are similar to those in compound (Ic), although the C—H···π(arene) interaction involves a different donor atom; again a chain of rings is formed, but this time it runs parallel to the [1$\bar{1}$0] direction (Fig. 9).

The only direction-specific intermolecular contact in compound (IIa) involves a methyl group. Because such groups CH$_3$—E generally undergo rapid rotation about the C—E bonds, even in the solid state (Riddell & Rogerson, 1996, 1997), particularly when, as here, the methyl group is bonded to a unit having local C_{2v} (mm2) symmetry, when the rotational barrier is particularly low (Tannenbaum et al., 1956; Naylor & Wilson, 1957). Accordingly, such a contact is not regarded as structurally significant. There is a single C—

Figure 9
Part of the crystal structure of compound (Ie) showing the formation of a chain of rings parallel to [1$\bar{1}$0]. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, the H atoms not involved in the motifs shown have been omitted.

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Figure 10
Part of the crystal structure of compound (IId) showing the formation of a centrosymmetric dimer. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, the unit-cell outline, the minor disorder component and the H atoms not involved in the motifs shown have been omitted. The atoms marked with an asterisk (*) are at the symmetry position $(1 - x, 2 - y, 1 - z)$.

each case, are found in all three of the type (I) compounds. Amongst the type (II) compounds, (IIa) and (IId) exhibit either no direction-specific intermolecular interactions, as in (IIa), or finite, zero-dimensional aggregation, as in (IId). In (IIe), a chain of rings is again found, but different from those in any of the type (I) series, although the $R_2^2(20)$ motif can be identified in each of (Ib), (IId) and (IIe).

4. Database survey

The structures have recently been reported (Vinutha *et al.*, 2014; Glidewell *et al.*, 2019; Kiran Kumar *et al.*, 2019) of a number of carbaldehyde precursors of type (B) (Fig. 12), including examples in which R = 2-chlorophenyl, 4-chlorophenyl and 2-naphthyl, *i.e.* the direct precursors for compounds (Ib), (Ic), (Ie), (IIb), (IIc) and (IIe). Structures have also been reported (Cuartas *et al.*, 2017) for both an amino analogue of (B), namely 5-[benzyl(methyl)amino]-3-methyl-1-phenyl-1*H*-pyrazole-4-carbaldehyde and of the chalcone derived from this by condensation with 4-bromo-benzaldehyde; for the 5-(*N*-methylpiperazino) analogue (Sunitha *et al.*, 2016) and for the 5-piperidino analogue (Kiran Kumar, 2019). Finally, we note the structures of two isostructural 3-(5-aryloxy-3-methyl-1-phenyl-1*H*-pyrazole-1-
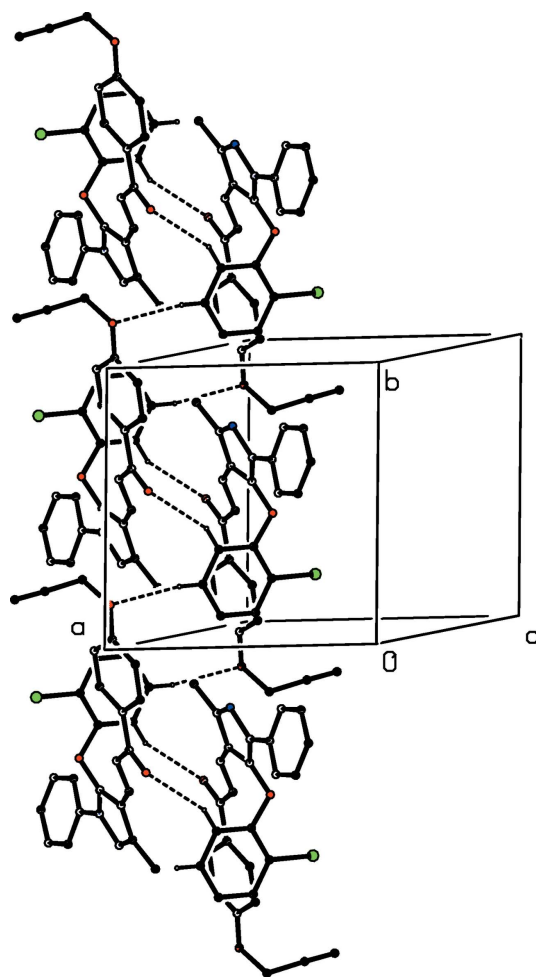

Figure 11
Part of the crystal structure of compound (IIe) showing the formation of a chain of rings parallel to [100]. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, the H not involved in the motifs shown have been omitted.

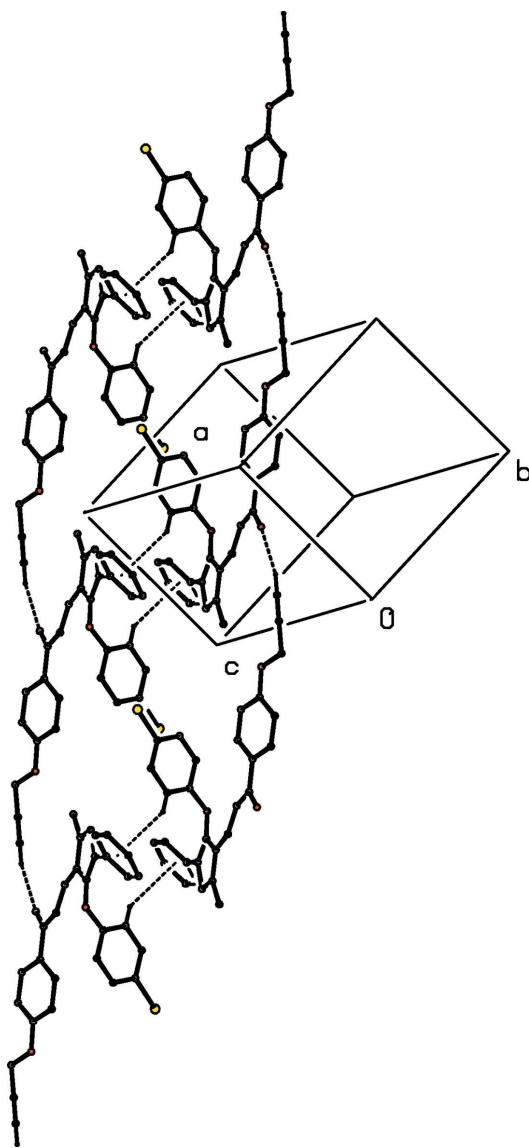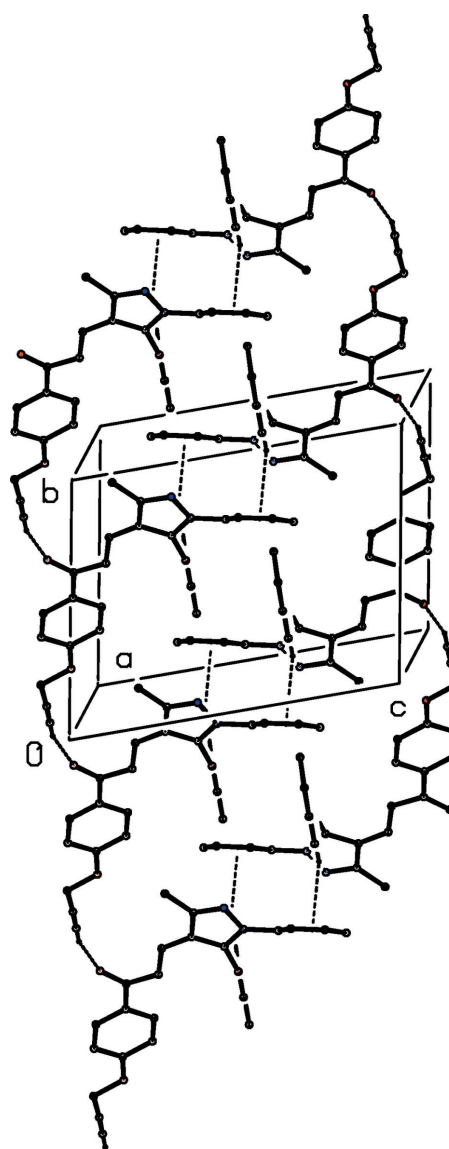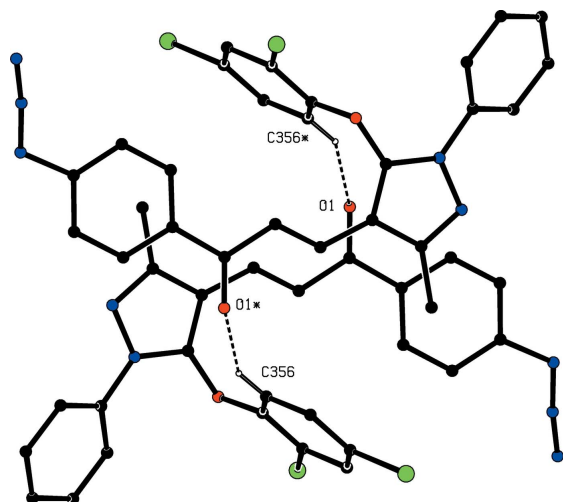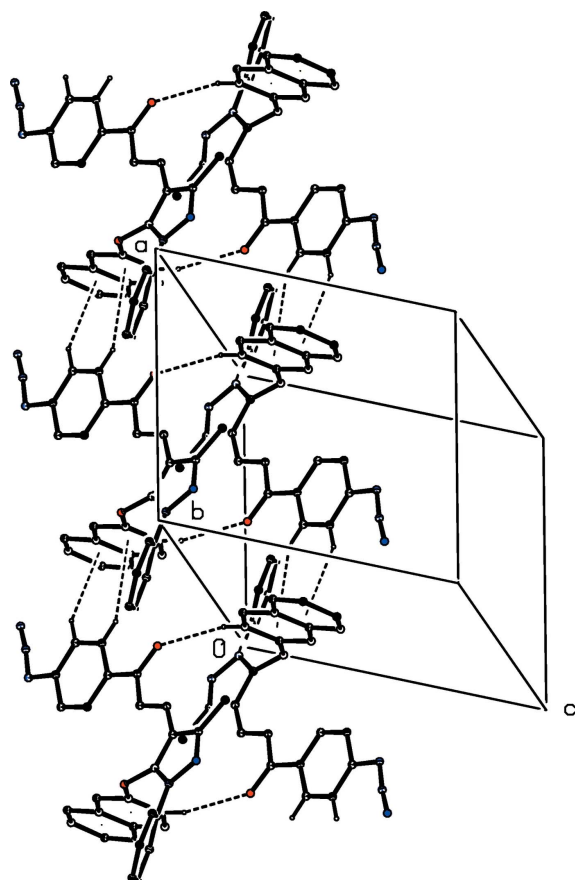**Figure 12**
The synthetic pathway used for the preparation of compounds (Ia)–(Ie) and (IIa)–(IIe).

Table 3
Experimental details.

	(Ib)	(Ic)	(Ie)
Crystal data			
Chemical formula	$C_{28}H_{21}ClN_2O_3$	$C_{28}H_{21}ClN_2O_3$	$C_{32}H_{24}N_2O_3$
M_r	468.92	468.92	484.53
Crystal system, space group	Triclinic, $P\bar{1}$	Triclinic, $P\bar{1}$	Triclinic, $P\bar{1}$
Temperature (K)	297	297	297
a, b, c (Å)	9.909 (7), 10.193 (6), 12.024 (8)	8.9959 (14), 9.7380 (15), 13.637 (2)	8.8615 (6), 10.4973 (7), 13.6588 (10)
α, β, γ (°)	90.94 (2), 106.27 (2), 92.75 (2)	95.901 (4), 94.122 (4), 95.959 (4)	79.006 (3), 89.412 (3), 80.971 (3)
V (Å³)	1163.9 (13)	1177.8 (3)	1231.54 (15)
Z	2	2	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm⁻¹)	0.20	0.20	0.08
Crystal size (mm)	$0.18 \times 0.15 \times 0.10$	$0.20 \times 0.15 \times 0.15$	$0.20 \times 0.16 \times 0.16$
Data collection			
Diffractometer	Bruker APEXII	Bruker APEXII	Bruker APEXII
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)	Multi-scan (*SADABS*; Bruker, 2016)	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.833, 0.980	0.901, 0.971	0.898, 0.987
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	43789, 5884, 4402	49011, 4837, 3930	30613, 4373, 3474
R_{int}	0.061	0.061	0.055
$(\sin\theta/\lambda)_{max}$ (Å⁻¹)	0.672	0.629	0.598
Refinement			
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.058, 0.155, 1.10	0.048, 0.125, 1.15	0.070, 0.187, 1.07
No. of reflections	5884	4837	4373
No. of parameters	309	309	336
No. of restraints	0	0	0
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å⁻³)	0.43, −0.41	0.35, −0.45	0.85, −0.29

	(IIa)	(IId)	(IIe)
Crystal data			
Chemical formula	$C_{26}H_{21}N_5O_2$	$C_{25}H_{17}Cl_2N_5O_2$	$C_{29}H_{21}N_5O_2$
M_r	435.48	490.33	471.51
Crystal system, space group	Triclinic, $P\bar{1}$	Monoclinic, $C2/c$	Monoclinic, $P2_1/n$
Temperature (K)	297	297	297
a, b, c (Å)	9.8432 (6), 11.7441 (7), 12.3005 (7)	28.1916 (17), 8.0537 (5), 22.0446 (12)	9.8460 (8), 22.4303 (18), 11.0490 (9)
α, β, γ (°)	114.120 (2), 111.139 (2), 96.537 (2)	90, 109.070 (1), 90	90, 104.157 (2), 90
V (Å³)	1152.06 (12)	4730.5 (5)	2366.0 (3)
Z	2	8	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm⁻¹)	0.08	0.31	0.09
Crystal size (mm)	$0.20 \times 0.20 \times 0.18$	$0.18 \times 0.15 \times 0.15$	$0.22 \times 0.21 \times 0.16$
Data collection			
Diffractometer	Bruker APEXII	Bruker APEXII	Bruker APEXII
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)	Multi-scan (*SADABS*; Bruker, 2016)	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.868, 0.985	0.881, 0.955	0.930, 0.986
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	17379, 4050, 2957	31508, 4174, 3181	43217, 4196, 2463
R_{int}	0.048	0.049	0.092
$(\sin\theta/\lambda)_{max}$ (Å⁻¹)	0.596	0.595	0.597
Refinement			
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.050, 0.148, 1.10	0.086, 0.155, 1.36	0.063, 0.154, 1.06
No. of reflections	4050	4174	4196
No. of parameters	301	382	327
No. of restraints	0	291	0
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å⁻³)	0.19, −0.22	0.21, −0.23	0.23, −0.27

Computer programs: *APEX3*, *SAINT* and *XPREP* (Bruker, 2016), *SHELXT2014/5* (Sheldrick, 2015a), *SHELXL2014* (Sheldrick, 2015b) and *PLATON* (Spek, 2020).

yl)-1-thiophen-2-yl)prop-2-en-1-ones, both of which exhibit disorder in the orientation of the thiophene unit (Shaibah *et al.*, 2020).

5. Synthesis and crystallization

For the preparation of the prop-2-yn-1-yl compounds (I), a solution of potassium hydroxide (0.31g, 5.7 mmol) in ethanol

(30 ml) was added to a mixture of the appropriate intermediate of type (B, Fig.7), 5.7 mmol) and 4-(prop-2-yn-1-yl-oxy)acetophenone (1.0 g, 5.7 mmol) in ethanol (30 ml). The mixtures were then stirred at ambient temperature for 4 h, after which time, TLC indicated that the reactions were complete. The solid products were then collected by filtration, washed with water, dried in air and recrystallized from an ethanol–dimethylformamide (initial composition 3:1, v/v). Compound (Ia). Yield 82%, m.p. 551 K. IR (cm^{-1}) 3228 (alkyne C-H), 2312 (alkyne C-C), 1672 (C=O), 1578 (C=N). MS (m/z) 449 (M+1)$^+$. Compound (Ib). Yield 67%, m.p. 438 K. IR (cm^{-1}) 3230 (alkyne C—H), 2352 (alkyne C—C), 1661 (C=O), 1581 (C=N). MS (m/z) 469 (M+1)$^+$. Compound (Ic). Yield 77%, m.p. 406–407 K. IR (cm^{-1}) 3234 (alkyne C-H), 2356 (alkyne C—C), 1668 (C=O), 1576 (C=N). MS (m/z) 469 (M+1)$^+$. Compound (Id). Yield 65%, m.p. 485–486 K. IR (cm^{-1}) 3237 (alkyne C—H), 2342 (alkyne C—C), 1676 (C=O), 1559 (C=N). MS (m/z) 469 (M+1)$^+$. Compound (Ie). Yield 69%, m.p. 447–449 K. IR (cm^{-1}) 3227 (alkyne C—H), 2360 (alkyne C—C), 1654 (C=O), 1588 (C=N). MS (m/z) 485 (M+1)$^+$. For the preparation of the azido compounds (II), a solution of potassium hydroxide (0.34g, 6.2 mmol) in ethanol (30 ml) was added to a solution of 4-azidoacetophenone (1.0 g, 6.2 mmol) in ethanol (30 ml). The appropriate intermediate (B) (6.2 mmol) was then added and the mixtures were then stirred for 30 min, after which time TLC indicated that the reactions were complete. The solid products were then collected by filtration, washed with water, dried in air and recrystallized from an ethanol–dimethylformamide (initial composition 3:1, v/v). Compound (IIa). Yield 96%, m.p. 385–387 K. IR (cm^{-1}) 2359 (azide), 1650 (C=O), 1592 (C=N). MS (m/z) 436 (M+1)$^+$. Compound (IIb). Yield 74%, m.p. 394–396 K. IR (cm^{-1}) 2355 (azide), 1674 (C=O), 1561 (C=N). MS (m/z) 456 (M+1)$^+$. Compound (IIc). Yield 79%, m.p. 425–427 K. IR (cm^{-1}) 2351 (azide), 1671 (C=O), 15612 (C=N). MS (m/z) 456 (M+1)$^+$. Compound (IId). Yield 70%, m.p. 505 K. IR (cm^{-1}) 2349 (azide), 1656 (C=O), 1592 (C=N). MS (m/z) 490 (M+1)$^+$. Compound (IIe). Yield 74%, m.p. 489–490 K. IR (cm^{-1}) 2354 (azide), 1676 (C=O), 1565 (C=N). MS (m/z) 472 (M+1)$^+$. Crystals of compounds (Ib), (Ic), (Ie), (IIa), (IId) and (IIe) which were suitable for single-crystal X-ray diffraction were selected directly from the prepared samples: despite repeated efforts, no crystal suitable for single-crystal X-ray diffraction have yet been obtained for compounds (Ia), (Id), (IIb) or (IIc).

6. Refinement

Crystal data, data collection and refinement details are summarized in Table 3. For a number of the structures, [(Ie), (IIa), (IId) and (IIe)], the diffraction data at values of $\theta > 25°$ were uniformly of very indifferent quality, particular in terms of the symmetry-equivalent reflections. This is probably a consequence of the indifferent crystal quality, exemplifying the general difficulty within the series (I) and (II) of growing crystals suitable for single-crystal X-ray diffraction (cf. Section 5, above). These higher-angle reflections were therefore

rejected during the data-reduction process: we note also that the intensity statistics indicated that very few of these reflections were likely to be labelled as observed for compounds (Ie), (IIa), (IId) and (IIe). A number of low-angle reflections for (Ib) and (Ie) were also discarded at this stage because of attenuation by the beam stop. Some further low-angle reflections that had been attenuated by the beam stop were omitted from the data sets before the final refinements, thus: for (Ib) (101), (110), (002), (202) and ($\bar{2}$02); for (Ic) ($\bar{1}$10) and (002); for (Ie) (002), (111) and (012); for (IIa) ($\bar{1}$11); and for (IId) ($\bar{1}$12). In addition, the bad outlier reflections ($\bar{2}$04) for (Id) and (130) for (IIe) were also omitted. All H atoms were located in difference maps and then treated as riding atoms in geometrically idealized positions with C—H distances 0.93 Å (aromatic), 0.96 Å (CH$_3$) or 0.97 Å (CH$_2$), and with U_{iso}(H) = kU_{eq}(C), where k = 1.5 for the methyl groups which were permitted to rotate but not to tilt, and 1.2 for all other H atoms. The final difference map for compound (Ie) contained two significant peaks, 0.85 e Å^{-3} at (0.227, 0.557, 0.598), and 0.81 e Å^{-3} at (0.380, 0.488, 0.596), respectively 1.27 and 1.14 Å from atom C351: however, attempts to develop a plausible disorder model based upon these two peaks were not fruitful. For the minor disorder compound in compound (IId), the bonded distances and the [1,3] non-bonded distances were restrained to be the same as the corresponding distances in the major disorder component, subject to s.u. values of 0.01 and 0.02 Å, respectively. In addition, similarity restraints were applied to the anisotropic displacement parameters of the partial-occupancy atoms in the disorder components. Subject to these conditions, the occupancies for the two disorder components refined to values of 0.55 (4) and 0.45 (4). Examination of the final refined structures using PLATON (Spek, 2020) showed that the structure of compound (IIa) contained a void space, of volume 64 Å^3, centred at (0.5, 0, 0), but further examination of this structure using the SQUEEZE procedure (Spek, 2015) showed that the void contained negligible electron density, consistent with the final difference map.

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supporting information

Functionalized 3-(5-aryloxy-3-methyl-1-phenyl-1*H*-pyrazol-4-yl)-1-(4-substituted-phenyl)prop-2-en-1-ones: synthetic pathway, and the structures of six examples

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Computing details

For all structures, data collection: *APEX3* (Bruker, 2016). Cell refinement: *APEX3*/*SAINT* V8.37A (Bruker, 2016) for (Ib), (Ic), (Ie), (IIa); *APEX3*/*SAINT* (Bruker, 2016) for (IId), (IIe). For all structures, data reduction: *SAINT*/*XPREP* (Bruker, 2016); program(s) used to solve structure: *SHELXT2014/5* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *PLATON* (Spek, 2020); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015b) and *PLATON* (Spek, 2020).

3-[5-(2-Chlorophenoxy)-3-methyl-1-phenyl-1*H*-pyrazol-4-yl]-1-[4-(prop-2-yn-1-yloxy)phenyl]prop-2-en-1-one (Ib)

Crystal data

$C_{28}H_{21}ClN_2O_3$	$Z = 2$
$M_r = 468.92$	$F(000) = 488$
Triclinic, $P\bar{1}$	$D_x = 1.338$ Mg m^{-3}
$a = 9.909$ (7) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 10.193$ (6) Å	Cell parameters from 7088 reflections
$c = 12.024$ (8) Å	$\theta = 3.0$–31.0°
$\alpha = 90.94$ (2)°	$\mu = 0.20$ mm^{-1}
$\beta = 106.27$ (2)°	$T = 297$ K
$\gamma = 92.75$ (2)°	Block, colourless
$V = 1163.9$ (13) Å^3	$0.18 \times 0.15 \times 0.10$ mm

Data collection

Bruker APEXII diffractometer	43789 measured reflections
Radiation source: fine focussealed tube	5884 independent reflections
Graphite monochromator	4402 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.061$
Absorption correction: multi-scan (SADABS; Bruker, 2016)	$\theta_{max} = 28.6°$, $\theta_{min} = 3.6°$
$T_{min} = 0.833$, $T_{max} = 0.980$	$h = -13 \rightarrow 13$
	$k = -13 \rightarrow 13$
	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.058$	$w = 1/[\sigma^2(F_o^2) + (0.0448P)^2 + 0.9732P]$
$wR(F^2) = 0.155$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.10$	$(\Delta/\sigma)_{max} < 0.001$
5884 reflections	$\Delta\rho_{max} = 0.43$ e Å^{-3}
309 parameters	$\Delta\rho_{min} = -0.41$ e Å^{-3}
0 restraints	Extinction correction: SHELXL,
Primary atom site location: dual	$Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
	Extinction coefficient: 0.154 (8)

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	0.9482 (2)	0.3833 (2)	0.72250 (19)	0.0464 (5)
O1	1.0416 (2)	0.45886 (19)	0.78220 (17)	0.0709 (6)
C2	0.8469 (2)	0.4294 (2)	0.6173 (2)	0.0478 (5)
H2	0.7655	0.3779	0.5825	0.057*
C3	0.8698 (2)	0.5434 (2)	0.57132 (18)	0.0405 (4)
H3	0.9508	0.5925	0.6116	0.049*
C11	0.9385 (2)	0.2433 (2)	0.75289 (18)	0.0417 (5)
C12	1.0483 (2)	0.1947 (2)	0.8398 (2)	0.0486 (5)
H12	1.1247	0.2507	0.8778	0.058*
C13	1.0451 (2)	0.0655 (2)	0.8700 (2)	0.0488 (5)
H13	1.1197	0.0347	0.9274	0.059*
C14	0.9314 (2)	−0.0192 (2)	0.8155 (2)	0.0446 (5)
C15	0.8204 (3)	0.0268 (2)	0.7302 (2)	0.0511 (6)
H15	0.7431	−0.0292	0.6939	0.061*
C16	0.8253 (2)	0.1567 (2)	0.6991 (2)	0.0496 (5)
H16	0.7511	0.1868	0.6410	0.060*
O14	0.94132 (19)	−0.14583 (16)	0.85166 (17)	0.0576 (5)
C17	0.8187 (3)	−0.2343 (3)	0.8139 (3)	0.0654 (7)
H17A	0.8452	−0.3230	0.8345	0.078*
H17B	0.7829	−0.2328	0.7301	0.078*
C18	0.7077 (3)	−0.2002 (3)	0.8653 (3)	0.0608 (7)
C19	0.6186 (4)	−0.1767 (4)	0.9061 (3)	0.0824 (10)
H19	0.5469	−0.1578	0.9390	0.099*
N31	0.62180 (18)	0.64397 (15)	0.30547 (16)	0.0394 (4)
N32	0.71026 (19)	0.75523 (16)	0.33267 (16)	0.0425 (4)
C33	0.8087 (2)	0.72855 (19)	0.42829 (19)	0.0394 (4)
C34	0.7862 (2)	0.60108 (18)	0.46748 (18)	0.0373 (4)
C35	0.6653 (2)	0.55241 (18)	0.38607 (18)	0.0368 (4)

C311	0.4990 (2)	0.6447 (2)	0.20822 (19)	0.0424 (5)
C312	0.4418 (4)	0.5309 (3)	0.1454 (3)	0.0775 (10)
H312	0.4841	0.4517	0.1647	0.093*
C313	0.3214 (4)	0.5351 (3)	0.0534 (3)	0.0871 (11)
H313	0.2826	0.4581	0.0117	0.104*
C314	0.2589 (3)	0.6505 (3)	0.0231 (2)	0.0684 (8)
H314	0.1766	0.6523	−0.0375	0.082*
C315	0.3188 (4)	0.7634 (3)	0.0829 (3)	0.0777 (9)
H315	0.2781	0.8428	0.0615	0.093*
C316	0.4393 (3)	0.7612 (3)	0.1752 (2)	0.0639 (7)
H316	0.4797	0.8390	0.2146	0.077*
C331	0.9235 (3)	0.8287 (2)	0.4843 (2)	0.0514 (6)
H33A	0.9224	0.9001	0.4330	0.077*
H33B	1.0128	0.7893	0.5005	0.077*
H33C	0.9093	0.8613	0.5553	0.077*
O351	0.58686 (15)	0.43808 (13)	0.38387 (13)	0.0395 (3)
C351	0.6396 (2)	0.32381 (17)	0.35285 (17)	0.0335 (4)
C352	0.5909 (2)	0.20629 (18)	0.38930 (17)	0.0358 (4)
Cl52	0.47497 (6)	0.21019 (6)	0.47407 (5)	0.05086 (19)
C353	0.6332 (2)	0.08826 (19)	0.35823 (19)	0.0431 (5)
H353	0.6008	0.0102	0.3832	0.052*
C354	0.7248 (3)	0.0858 (2)	0.2892 (2)	0.0478 (5)
H354	0.7528	0.0060	0.2671	0.057*
C355	0.7740 (2)	0.2023 (2)	0.2537 (2)	0.0455 (5)
H355	0.8358	0.2006	0.2081	0.055*
C356	0.7321 (2)	0.32177 (19)	0.28541 (18)	0.0396 (4)
H356	0.7659	0.3999	0.2616	0.048*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0496 (12)	0.0438 (12)	0.0403 (11)	−0.0114 (9)	0.0060 (9)	0.0047 (9)
O1	0.0757 (13)	0.0561 (11)	0.0594 (11)	−0.0278 (9)	−0.0116 (9)	0.0101 (9)
C2	0.0484 (12)	0.0413 (11)	0.0463 (12)	−0.0082 (9)	0.0027 (10)	0.0037 (9)
C3	0.0404 (10)	0.0360 (10)	0.0430 (11)	−0.0028 (8)	0.0092 (8)	−0.0004 (8)
C11	0.0438 (11)	0.0435 (11)	0.0346 (10)	−0.0065 (9)	0.0072 (8)	0.0038 (8)
C12	0.0434 (11)	0.0512 (13)	0.0443 (11)	−0.0071 (9)	0.0027 (9)	0.0045 (10)
C13	0.0434 (12)	0.0514 (13)	0.0478 (12)	0.0026 (10)	0.0060 (9)	0.0089 (10)
C14	0.0501 (12)	0.0399 (11)	0.0460 (11)	0.0008 (9)	0.0174 (10)	0.0028 (9)
C15	0.0495 (13)	0.0445 (12)	0.0525 (13)	−0.0089 (10)	0.0050 (10)	0.0025 (10)
C16	0.0462 (12)	0.0477 (12)	0.0466 (12)	−0.0074 (10)	0.0005 (10)	0.0091 (10)
O14	0.0601 (10)	0.0410 (9)	0.0728 (12)	0.0041 (7)	0.0196 (9)	0.0106 (8)
C17	0.0797 (19)	0.0404 (13)	0.0728 (18)	−0.0059 (12)	0.0178 (15)	0.0014 (12)
C18	0.0536 (15)	0.0500 (14)	0.0678 (17)	−0.0125 (11)	0.0010 (13)	0.0083 (12)
C19	0.0546 (17)	0.088 (2)	0.096 (2)	−0.0109 (16)	0.0100 (17)	0.0045 (19)
N31	0.0418 (9)	0.0256 (8)	0.0468 (9)	−0.0026 (6)	0.0067 (7)	0.0027 (7)
N32	0.0465 (10)	0.0249 (8)	0.0515 (10)	−0.0059 (7)	0.0079 (8)	0.0017 (7)
C33	0.0426 (11)	0.0283 (9)	0.0468 (11)	−0.0032 (8)	0.0127 (9)	−0.0023 (8)

C34	0.0393 (10)	0.0273 (9)	0.0443 (11)	−0.0002 (7)	0.0104 (8)	0.0000 (8)
C35	0.0401 (10)	0.0243 (8)	0.0460 (11)	−0.0019 (7)	0.0127 (8)	0.0007 (7)
C311	0.0429 (11)	0.0378 (10)	0.0443 (11)	−0.0002 (8)	0.0085 (9)	0.0063 (8)
C312	0.086 (2)	0.0420 (14)	0.0768 (19)	−0.0110 (13)	−0.0211 (16)	0.0075 (13)
C313	0.094 (2)	0.0627 (18)	0.074 (2)	−0.0247 (16)	−0.0216 (18)	0.0134 (15)
C314	0.0563 (15)	0.089 (2)	0.0507 (14)	−0.0010 (14)	0.0002 (12)	0.0174 (14)
C315	0.087 (2)	0.076 (2)	0.0599 (17)	0.0339 (17)	−0.0013 (15)	0.0082 (15)
C316	0.0765 (18)	0.0487 (14)	0.0565 (15)	0.0156 (13)	0.0005 (13)	−0.0013 (11)
C331	0.0540 (13)	0.0357 (11)	0.0585 (14)	−0.0118 (9)	0.0084 (11)	−0.0002 (10)
O351	0.0391 (7)	0.0245 (6)	0.0563 (9)	−0.0031 (5)	0.0164 (6)	0.0009 (6)
C351	0.0338 (9)	0.0260 (8)	0.0374 (9)	−0.0023 (7)	0.0055 (7)	0.0005 (7)
C352	0.0353 (9)	0.0307 (9)	0.0382 (10)	−0.0063 (7)	0.0063 (8)	0.0020 (7)
Cl52	0.0522 (3)	0.0465 (3)	0.0582 (4)	−0.0102 (2)	0.0248 (3)	0.0016 (2)
C353	0.0521 (12)	0.0265 (9)	0.0479 (11)	−0.0043 (8)	0.0106 (9)	0.0040 (8)
C354	0.0604 (14)	0.0322 (10)	0.0508 (12)	0.0068 (9)	0.0149 (11)	0.0003 (9)
C355	0.0481 (12)	0.0432 (11)	0.0486 (12)	0.0061 (9)	0.0187 (10)	0.0035 (9)
C356	0.0409 (10)	0.0322 (10)	0.0460 (11)	−0.0023 (8)	0.0129 (9)	0.0062 (8)

Geometric parameters (Å, º)

C1—O1	1.228 (3)	C33—C331	1.492 (3)
C1—C2	1.477 (3)	C34—C35	1.382 (3)
C1—C11	1.484 (3)	C35—O351	1.365 (2)
C2—C3	1.331 (3)	C311—C316	1.367 (3)
C2—H2	0.9300	C311—C312	1.380 (3)
C3—C34	1.443 (3)	C312—C313	1.384 (4)
C3—H3	0.9300	C312—H312	0.9300
C11—C16	1.391 (3)	C313—C314	1.363 (5)
C11—C12	1.398 (3)	C313—H313	0.9300
C12—C13	1.373 (3)	C314—C315	1.364 (5)
C12—H12	0.9300	C314—H314	0.9300
C13—C14	1.385 (3)	C315—C316	1.385 (4)
C13—H13	0.9300	C315—H315	0.9300
C14—O14	1.368 (3)	C316—H316	0.9300
C14—C15	1.384 (3)	C331—H33A	0.9600
C15—C16	1.384 (3)	C331—H33B	0.9600
C15—H15	0.9300	C331—H33C	0.9600
C16—H16	0.9300	O351—C351	1.387 (2)
O14—C17	1.438 (3)	C351—C356	1.384 (3)
C17—C18	1.458 (4)	C351—C352	1.393 (3)
C17—H17A	0.9700	C352—C353	1.373 (3)
C17—H17B	0.9700	C352—Cl52	1.737 (2)
C18—C19	1.157 (5)	C353—C354	1.391 (3)
C19—H19	0.9300	C353—H353	0.9300
N31—C35	1.353 (3)	C354—C355	1.382 (3)
N31—N32	1.376 (2)	C354—H354	0.9300
N31—C311	1.433 (3)	C355—C356	1.386 (3)
N32—C33	1.325 (3)	C355—H355	0.9300

C33—C34	1.418 (3)	C356—H356	0.9300
O1—C1—C2	120.4 (2)	N31—C35—O351	120.93 (18)
O1—C1—C11	120.4 (2)	N31—C35—C34	109.00 (17)
C2—C1—C11	119.14 (19)	O351—C35—C34	129.85 (19)
C3—C2—C1	121.3 (2)	C316—C311—C312	119.3 (2)
C3—C2—H2	119.3	C316—C311—N31	119.2 (2)
C1—C2—H2	119.3	C312—C311—N31	121.5 (2)
C2—C3—C34	128.8 (2)	C311—C312—C313	119.8 (3)
C2—C3—H3	115.6	C311—C312—H312	120.1
C34—C3—H3	115.6	C313—C312—H312	120.1
C16—C11—C12	117.7 (2)	C314—C313—C312	120.9 (3)
C16—C11—C1	123.4 (2)	C314—C313—H313	119.6
C12—C11—C1	118.86 (19)	C312—C313—H313	119.6
C13—C12—C11	121.1 (2)	C313—C314—C315	119.1 (3)
C13—C12—H12	119.5	C313—C314—H314	120.5
C11—C12—H12	119.5	C315—C314—H314	120.5
C12—C13—C14	120.4 (2)	C314—C315—C316	120.9 (3)
C12—C13—H13	119.8	C314—C315—H315	119.6
C14—C13—H13	119.8	C316—C315—H315	119.6
O14—C14—C15	125.2 (2)	C311—C316—C315	120.0 (3)
O14—C14—C13	115.0 (2)	C311—C316—H316	120.0
C15—C14—C13	119.8 (2)	C315—C316—H316	120.0
C16—C15—C14	119.5 (2)	C33—C331—H33A	109.5
C16—C15—H15	120.2	C33—C331—H33B	109.5
C14—C15—H15	120.2	H33A—C331—H33B	109.5
C15—C16—C11	121.6 (2)	C33—C331—H33C	109.5
C15—C16—H16	119.2	H33A—C331—H33C	109.5
C11—C16—H16	119.2	H33B—C331—H33C	109.5
C14—O14—C17	118.3 (2)	C35—O351—C351	117.09 (16)
O14—C17—C18	112.3 (2)	C356—C351—O351	123.40 (17)
O14—C17—H17A	109.1	C356—C351—C352	119.82 (18)
C18—C17—H17A	109.1	O351—C351—C352	116.73 (18)
O14—C17—H17B	109.1	C353—C352—C351	120.49 (19)
C18—C17—H17B	109.1	C353—C352—Cl52	120.13 (15)
H17A—C17—H17B	107.9	C351—C352—Cl52	119.38 (16)
C19—C18—C17	178.1 (3)	C352—C353—C354	119.80 (19)
C18—C19—H19	180.0	C352—C353—H353	120.1
C35—N31—N32	110.06 (17)	C354—C353—H353	120.1
C35—N31—C311	130.75 (17)	C355—C354—C353	119.8 (2)
N32—N31—C311	118.98 (16)	C355—C354—H354	120.1
C33—N32—N31	105.58 (16)	C353—C354—H354	120.1
N32—C33—C34	112.02 (18)	C354—C355—C356	120.6 (2)
N32—C33—C331	120.60 (19)	C354—C355—H355	119.7
C34—C33—C331	127.3 (2)	C356—C355—H355	119.7
C35—C34—C33	103.31 (18)	C351—C356—C355	119.51 (18)
C35—C34—C3	130.32 (19)	C351—C356—H356	120.2
C33—C34—C3	126.33 (19)	C355—C356—H356	120.2

O1—C1—C2—C3	13.8 (4)	C311—N31—C35—C34	−175.8 (2)
C11—C1—C2—C3	−164.2 (2)	C33—C34—C35—N31	0.5 (2)
C1—C2—C3—C34	177.4 (2)	C3—C34—C35—N31	178.0 (2)
O1—C1—C11—C16	171.0 (2)	C33—C34—C35—O351	−174.0 (2)
C2—C1—C11—C16	−11.1 (4)	C3—C34—C35—O351	3.5 (4)
O1—C1—C11—C12	−9.2 (4)	C35—N31—C311—C316	147.1 (3)
C2—C1—C11—C12	168.8 (2)	N32—N31—C311—C316	−27.1 (3)
C16—C11—C12—C13	0.7 (4)	C35—N31—C311—C312	−34.7 (4)
C1—C11—C12—C13	−179.2 (2)	N32—N31—C311—C312	151.1 (3)
C11—C12—C13—C14	−0.8 (4)	C316—C311—C312—C313	−3.1 (5)
C12—C13—C14—O14	179.0 (2)	N31—C311—C312—C313	178.7 (3)
C12—C13—C14—C15	−0.1 (4)	C311—C312—C313—C314	0.7 (6)
O14—C14—C15—C16	−178.1 (2)	C312—C313—C314—C315	1.7 (6)
C13—C14—C15—C16	0.9 (4)	C313—C314—C315—C316	−1.6 (5)
C14—C15—C16—C11	−1.0 (4)	C312—C311—C316—C315	3.1 (5)
C12—C11—C16—C15	0.1 (4)	N31—C311—C316—C315	−178.6 (3)
C1—C11—C16—C15	−180.0 (2)	C314—C315—C316—C311	−0.8 (5)
C15—C14—O14—C17	−11.2 (4)	N31—C35—O351—C351	109.4 (2)
C13—C14—O14—C17	169.8 (2)	C34—C35—O351—C351	−76.7 (3)
C14—O14—C17—C18	−68.7 (3)	C35—O351—C351—C356	−24.8 (3)
C35—N31—N32—C33	1.5 (2)	C35—O351—C351—C352	157.64 (18)
C311—N31—N32—C33	176.81 (18)	C356—C351—C352—C353	−0.4 (3)
N31—N32—C33—C34	−1.2 (2)	O351—C351—C352—C353	177.27 (18)
N31—N32—C33—C331	−179.43 (19)	C356—C351—C352—Cl52	179.92 (15)
N32—C33—C34—C35	0.5 (2)	O351—C351—C352—Cl52	−2.4 (2)
C331—C33—C34—C35	178.5 (2)	C351—C352—C353—C354	−0.4 (3)
N32—C33—C34—C3	−177.2 (2)	Cl52—C352—C353—C354	179.26 (17)
C331—C33—C34—C3	0.9 (4)	C352—C353—C354—C355	0.9 (3)
C2—C3—C34—C35	−3.3 (4)	C353—C354—C355—C356	−0.5 (4)
C2—C3—C34—C33	173.7 (2)	O351—C351—C356—C355	−176.76 (19)
N32—N31—C35—O351	173.80 (17)	C352—C351—C356—C355	0.8 (3)
C311—N31—C35—O351	−0.8 (3)	C354—C355—C356—C351	−0.3 (3)
N32—N31—C35—C34	−1.3 (2)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C355—H355···O14[i]	0.93	2.59	3.468 (4)	158
C356—H356···O1[ii]	0.93	2.51	3.360 (4)	152

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

3-[5-(4-Chlorophenoxy)-3-methyl-1-phenyl-1*H*-pyrazol-4-yl]-1-[4-(prop-2-yn-1-yloxy)phenyl]prop-2-en-1-one (Ic)

Crystal data

$C_{28}H_{21}ClN_2O_3$	$Z = 2$
$M_r = 468.92$	$F(000) = 488$
Triclinic, $P\overline{1}$	$D_x = 1.322$ Mg m^{-3}
$a = 8.9959$ (14) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 9.7380$ (15) Å	Cell parameters from 4839 reflections
$c = 13.637$ (2) Å	$\theta = 2.9–26.5°$
$\alpha = 95.901$ (4)°	$\mu = 0.20$ mm^{-1}
$\beta = 94.122$ (4)°	$T = 297$ K
$\gamma = 95.959$ (4)°	Block, brown
$V = 1177.8$ (3) Å^3	$0.20 \times 0.15 \times 0.15$ mm

Data collection

Bruker APEXII diffractometer	49011 measured reflections
Radiation source: fine focussealed tube	4837 independent reflections
Graphite monochromator	3930 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.061$
Absorption correction: multi-scan (SADABS; Bruker, 2016)	$\theta_{max} = 26.5°$, $\theta_{min} = 3.3°$
$T_{min} = 0.901$, $T_{max} = 0.971$	$h = -11\rightarrow11$
	$k = -12\rightarrow12$
	$l = -17\rightarrow17$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.048$	$w = 1/[\sigma^2(F_o^2) + (0.0384P)^2 + 0.6832P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.125$	
$S = 1.15$	$(\Delta/\sigma)_{max} = 0.001$
4837 reflections	$\Delta\rho_{max} = 0.35$ e Å^{-3}
309 parameters	$\Delta\rho_{min} = -0.45$ e Å^{-3}
0 restraints	Extinction correction: SHELXL, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: dual	Extinction coefficient: 0.092 (6)

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	0.5453 (2)	0.2711 (2)	0.53901 (14)	0.0354 (4)
O1	0.49212 (18)	0.19336 (17)	0.46533 (11)	0.0535 (4)
C2	0.5001 (2)	0.2494 (2)	0.63858 (14)	0.0375 (4)
H2	0.5266	0.3202	0.6899	0.045*
C3	0.4233 (2)	0.1329 (2)	0.65779 (14)	0.0360 (4)
H3	0.3999	0.0635	0.6051	0.043*

C11	0.65551 (19)	0.39198 (18)	0.52879 (13)	0.0321 (4)
C12	0.6706 (2)	0.4355 (2)	0.43611 (13)	0.0366 (4)
H12	0.6103	0.3890	0.3821	0.044*
C13	0.7725 (2)	0.54596 (19)	0.42185 (14)	0.0373 (4)
H13	0.7803	0.5739	0.3591	0.045*
C14	0.8635 (2)	0.61513 (19)	0.50232 (14)	0.0351 (4)
C15	0.8509 (2)	0.5724 (2)	0.59521 (14)	0.0430 (5)
H15	0.9124	0.6180	0.6490	0.052*
C16	0.7477 (2)	0.4627 (2)	0.60857 (14)	0.0408 (5)
H16	0.7394	0.4355	0.6715	0.049*
O14	0.96866 (16)	0.72487 (15)	0.49739 (10)	0.0466 (4)
C17	0.9800 (2)	0.7763 (2)	0.40407 (16)	0.0456 (5)
H17A	0.8869	0.8110	0.3832	0.055*
H17B	0.9987	0.7023	0.3548	0.055*
C18	1.1030 (2)	0.8878 (2)	0.41364 (16)	0.0445 (5)
C19	1.2031 (3)	0.9760 (2)	0.41795 (18)	0.0541 (6)
H19	1.2825	1.0460	0.4214	0.065*
N31	0.32759 (17)	0.10603 (16)	0.91019 (11)	0.0359 (4)
N32	0.23589 (18)	−0.00607 (17)	0.86251 (12)	0.0387 (4)
C33	0.2648 (2)	−0.00897 (19)	0.76832 (14)	0.0350 (4)
C34	0.37180 (19)	0.10233 (19)	0.75252 (13)	0.0324 (4)
C35	0.4074 (2)	0.17216 (19)	0.84574 (13)	0.0333 (4)
C311	0.3251 (2)	0.14030 (19)	1.01431 (14)	0.0376 (4)
C312	0.4578 (3)	0.1772 (2)	1.07284 (16)	0.0498 (5)
H312	0.5492	0.1809	1.0449	0.060*
C313	0.4527 (3)	0.2086 (3)	1.17386 (18)	0.0639 (7)
H313	0.5415	0.2351	1.2136	0.077*
C314	0.3191 (4)	0.2013 (3)	1.21603 (18)	0.0660 (7)
H314	0.3172	0.2223	1.2840	0.079*
C315	0.1875 (3)	0.1625 (3)	1.15721 (19)	0.0633 (7)
H315	0.0966	0.1564	1.1858	0.076*
C316	0.1892 (3)	0.1326 (2)	1.05553 (16)	0.0478 (5)
H316	0.1001	0.1077	1.0158	0.057*
C331	0.1866 (3)	−0.1197 (2)	0.69277 (16)	0.0485 (5)
H33A	0.1505	−0.1984	0.7248	0.073*
H33B	0.2555	−0.1473	0.6458	0.073*
H33C	0.1037	−0.0845	0.6591	0.073*
O351	0.48920 (15)	0.29585 (13)	0.87828 (10)	0.0397 (3)
C351	0.6443 (2)	0.31066 (19)	0.87288 (13)	0.0335 (4)
C352	0.7128 (2)	0.4453 (2)	0.89176 (16)	0.0431 (5)
H352	0.6565	0.5182	0.9075	0.052*
C353	0.8669 (2)	0.4705 (2)	0.88698 (16)	0.0460 (5)
H353	0.9148	0.5607	0.8994	0.055*
C354	0.9481 (2)	0.3613 (2)	0.86373 (14)	0.0415 (5)
Cl54	1.14061 (6)	0.39375 (8)	0.85575 (5)	0.0630 (2)
C355	0.8803 (2)	0.2275 (2)	0.84574 (16)	0.0458 (5)
H355	0.9369	0.1547	0.8305	0.055*
C356	0.7260 (2)	0.2015 (2)	0.85043 (16)	0.0422 (5)

| H356 | 0.6785 | 0.1112 | 0.8385 | 0.051* |

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0323 (9)	0.0385 (10)	0.0340 (9)	−0.0033 (7)	0.0052 (7)	0.0035 (8)
O1	0.0583 (9)	0.0560 (9)	0.0383 (8)	−0.0266 (7)	0.0049 (7)	0.0008 (7)
C2	0.0375 (10)	0.0407 (10)	0.0334 (9)	−0.0029 (8)	0.0059 (7)	0.0046 (8)
C3	0.0340 (9)	0.0396 (10)	0.0338 (9)	−0.0022 (8)	0.0067 (7)	0.0042 (8)
C11	0.0306 (9)	0.0326 (9)	0.0323 (9)	−0.0010 (7)	0.0053 (7)	0.0020 (7)
C12	0.0371 (10)	0.0393 (10)	0.0307 (9)	−0.0050 (8)	0.0015 (7)	0.0014 (7)
C13	0.0399 (10)	0.0376 (10)	0.0339 (9)	−0.0031 (8)	0.0041 (8)	0.0074 (8)
C14	0.0329 (9)	0.0306 (9)	0.0401 (10)	−0.0043 (7)	0.0071 (7)	0.0002 (7)
C15	0.0430 (11)	0.0469 (11)	0.0332 (10)	−0.0121 (9)	0.0010 (8)	−0.0040 (8)
C16	0.0433 (11)	0.0464 (11)	0.0300 (9)	−0.0092 (9)	0.0050 (8)	0.0045 (8)
O14	0.0492 (8)	0.0414 (8)	0.0439 (8)	−0.0179 (6)	0.0045 (6)	0.0028 (6)
C17	0.0423 (11)	0.0432 (11)	0.0492 (12)	−0.0097 (9)	0.0002 (9)	0.0125 (9)
C18	0.0425 (11)	0.0422 (11)	0.0486 (12)	−0.0027 (9)	0.0061 (9)	0.0095 (9)
C19	0.0508 (13)	0.0523 (13)	0.0564 (13)	−0.0144 (10)	0.0057 (10)	0.0119 (10)
N31	0.0378 (8)	0.0370 (8)	0.0320 (8)	−0.0017 (7)	0.0059 (6)	0.0035 (6)
N32	0.0384 (8)	0.0393 (9)	0.0370 (8)	−0.0047 (7)	0.0066 (7)	0.0040 (7)
C33	0.0336 (9)	0.0352 (9)	0.0357 (9)	−0.0014 (7)	0.0045 (7)	0.0059 (7)
C34	0.0305 (8)	0.0340 (9)	0.0330 (9)	0.0010 (7)	0.0051 (7)	0.0062 (7)
C35	0.0306 (9)	0.0330 (9)	0.0359 (9)	0.0003 (7)	0.0046 (7)	0.0033 (7)
C311	0.0488 (11)	0.0342 (9)	0.0317 (9)	0.0091 (8)	0.0076 (8)	0.0055 (7)
C312	0.0551 (13)	0.0560 (13)	0.0395 (11)	0.0168 (10)	0.0017 (9)	0.0015 (9)
C313	0.0885 (19)	0.0621 (15)	0.0411 (12)	0.0254 (14)	−0.0089 (12)	−0.0017 (11)
C314	0.111 (2)	0.0559 (15)	0.0355 (12)	0.0270 (15)	0.0143 (14)	0.0046 (10)
C315	0.0897 (19)	0.0548 (14)	0.0540 (14)	0.0181 (13)	0.0390 (14)	0.0136 (11)
C316	0.0553 (13)	0.0459 (12)	0.0455 (12)	0.0062 (10)	0.0177 (10)	0.0104 (9)
C331	0.0481 (12)	0.0482 (12)	0.0439 (11)	−0.0120 (9)	0.0045 (9)	−0.0027 (9)
O351	0.0352 (7)	0.0334 (7)	0.0484 (8)	−0.0014 (5)	0.0065 (6)	−0.0024 (6)
C351	0.0350 (9)	0.0334 (9)	0.0314 (9)	0.0010 (7)	0.0026 (7)	0.0027 (7)
C352	0.0432 (11)	0.0320 (10)	0.0518 (12)	0.0024 (8)	0.0019 (9)	−0.0028 (8)
C353	0.0460 (11)	0.0387 (11)	0.0490 (12)	−0.0073 (9)	−0.0020 (9)	0.0009 (9)
C354	0.0363 (10)	0.0530 (12)	0.0333 (10)	0.0000 (9)	0.0001 (8)	0.0024 (8)
Cl54	0.0371 (3)	0.0877 (5)	0.0605 (4)	−0.0021 (3)	0.0037 (2)	0.0001 (3)
C355	0.0422 (11)	0.0451 (11)	0.0500 (12)	0.0100 (9)	0.0022 (9)	0.0000 (9)
C356	0.0428 (11)	0.0324 (10)	0.0494 (11)	0.0010 (8)	0.0022 (9)	0.0002 (8)

Geometric parameters (Å, º)

C1—O1	1.229 (2)	C33—C331	1.493 (3)
C1—C2	1.474 (3)	C34—C35	1.378 (3)
C1—C11	1.483 (2)	C35—O351	1.360 (2)
C2—C3	1.327 (3)	C311—C316	1.380 (3)
C2—H2	0.9300	C311—C312	1.380 (3)
C3—C34	1.452 (2)	C312—C313	1.385 (3)

C3—H3	0.9300	C312—H312	0.9300
C11—C12	1.385 (3)	C313—C314	1.367 (4)
C11—C16	1.394 (3)	C313—H313	0.9300
C12—C13	1.378 (3)	C314—C315	1.377 (4)
C12—H12	0.9300	C314—H314	0.9300
C13—C14	1.390 (3)	C315—C316	1.389 (3)
C13—H13	0.9300	C315—H315	0.9300
C14—O14	1.362 (2)	C316—H316	0.9300
C14—C15	1.382 (3)	C331—H33A	0.9600
C15—C16	1.376 (3)	C331—H33B	0.9600
C15—H15	0.9300	C331—H33C	0.9600
C16—H16	0.9300	O351—C351	1.396 (2)
O14—C17	1.421 (2)	C351—C356	1.375 (3)
C17—C18	1.456 (3)	C351—C352	1.380 (3)
C17—H17A	0.9700	C352—C353	1.391 (3)
C17—H17B	0.9700	C352—H352	0.9300
C18—C19	1.172 (3)	C353—C354	1.374 (3)
C19—H19	0.9300	C353—H353	0.9300
N31—C35	1.347 (2)	C354—C355	1.370 (3)
N31—N32	1.373 (2)	C354—Cl54	1.741 (2)
N31—C311	1.427 (2)	C355—C356	1.393 (3)
N32—C33	1.327 (2)	C355—H355	0.9300
C33—C34	1.418 (2)	C356—H356	0.9300
O1—C1—C2	121.66 (17)	N31—C35—O351	118.65 (16)
O1—C1—C11	120.01 (16)	N31—C35—C34	108.52 (15)
C2—C1—C11	118.32 (16)	O351—C35—C34	132.30 (16)
C3—C2—C1	122.56 (18)	C316—C311—C312	120.78 (19)
C3—C2—H2	118.7	C316—C311—N31	119.17 (19)
C1—C2—H2	118.7	C312—C311—N31	120.03 (18)
C2—C3—C34	126.73 (18)	C311—C312—C313	119.0 (2)
C2—C3—H3	116.6	C311—C312—H312	120.5
C34—C3—H3	116.6	C313—C312—H312	120.5
C12—C11—C16	118.11 (16)	C314—C313—C312	121.0 (3)
C12—C11—C1	119.13 (16)	C314—C313—H313	119.5
C16—C11—C1	122.75 (16)	C312—C313—H313	119.5
C13—C12—C11	121.74 (17)	C313—C314—C315	119.5 (2)
C13—C12—H12	119.1	C313—C314—H314	120.2
C11—C12—H12	119.1	C315—C314—H314	120.2
C12—C13—C14	119.29 (17)	C314—C315—C316	120.6 (2)
C12—C13—H13	120.4	C314—C315—H315	119.7
C14—C13—H13	120.4	C316—C315—H315	119.7
O14—C14—C15	115.58 (16)	C311—C316—C315	119.0 (2)
O14—C14—C13	124.66 (17)	C311—C316—H316	120.5
C15—C14—C13	119.77 (16)	C315—C316—H316	120.5
C16—C15—C14	120.34 (17)	C33—C331—H33A	109.5
C16—C15—H15	119.8	C33—C331—H33B	109.5
C14—C15—H15	119.8	H33A—C331—H33B	109.5

C15—C16—C11	120.75 (17)	C33—C331—H33C	109.5
C15—C16—H16	119.6	H33A—C331—H33C	109.5
C11—C16—H16	119.6	H33B—C331—H33C	109.5
C14—O14—C17	117.27 (15)	C35—O351—C351	119.84 (14)
O14—C17—C18	108.51 (17)	C356—C351—C352	121.22 (18)
O14—C17—H17A	110.0	C356—C351—O351	123.80 (16)
C18—C17—H17A	110.0	C352—C351—O351	114.98 (16)
O14—C17—H17B	110.0	C351—C352—C353	119.14 (19)
C18—C17—H17B	110.0	C351—C352—H352	120.4
H17A—C17—H17B	108.4	C353—C352—H352	120.4
C19—C18—C17	177.6 (2)	C354—C353—C352	119.52 (19)
C18—C19—H19	180.0	C354—C353—H353	120.2
C35—N31—N32	110.96 (15)	C352—C353—H353	120.2
C35—N31—C311	129.06 (16)	C355—C354—C353	121.34 (19)
N32—N31—C311	119.96 (15)	C355—C354—Cl54	119.29 (17)
C33—N32—N31	104.91 (14)	C353—C354—Cl54	119.37 (16)
N32—C33—C34	112.02 (16)	C354—C355—C356	119.44 (19)
N32—C33—C331	120.58 (17)	C354—C355—H355	120.3
C34—C33—C331	127.39 (17)	C356—C355—H355	120.3
C35—C34—C33	103.56 (15)	C351—C356—C355	119.34 (18)
C35—C34—C3	130.35 (17)	C351—C356—H356	120.3
C33—C34—C3	126.06 (17)	C355—C356—H356	120.3
O1—C1—C2—C3	13.1 (3)	N32—N31—C35—C34	−1.2 (2)
C11—C1—C2—C3	−168.12 (19)	C311—N31—C35—C34	−179.14 (18)
C1—C2—C3—C34	−179.02 (18)	C33—C34—C35—N31	0.2 (2)
O1—C1—C11—C12	15.4 (3)	C3—C34—C35—N31	178.15 (19)
C2—C1—C11—C12	−163.41 (18)	C33—C34—C35—O351	−171.11 (19)
O1—C1—C11—C16	−163.3 (2)	C3—C34—C35—O351	6.8 (3)
C2—C1—C11—C16	17.9 (3)	C35—N31—C311—C316	136.4 (2)
C16—C11—C12—C13	−0.4 (3)	N32—N31—C311—C316	−41.4 (3)
C1—C11—C12—C13	−179.10 (18)	C35—N31—C311—C312	−45.3 (3)
C11—C12—C13—C14	0.4 (3)	N32—N31—C311—C312	137.0 (2)
C12—C13—C14—O14	179.64 (18)	C316—C311—C312—C313	−1.0 (3)
C12—C13—C14—C15	0.1 (3)	N31—C311—C312—C313	−179.3 (2)
O14—C14—C15—C16	179.76 (19)	C311—C312—C313—C314	1.1 (4)
C13—C14—C15—C16	−0.6 (3)	C312—C313—C314—C315	−0.2 (4)
C14—C15—C16—C11	0.7 (3)	C313—C314—C315—C316	−0.8 (4)
C12—C11—C16—C15	−0.2 (3)	C312—C311—C316—C315	0.0 (3)
C1—C11—C16—C15	178.48 (19)	N31—C311—C316—C315	178.35 (19)
C15—C14—O14—C17	−176.97 (19)	C314—C315—C316—C311	0.9 (3)
C13—C14—O14—C17	3.5 (3)	N31—C35—O351—C351	120.05 (18)
C14—O14—C17—C18	−177.14 (18)	C34—C35—O351—C351	−69.3 (3)
C35—N31—N32—C33	1.7 (2)	C35—O351—C351—C356	−10.4 (3)
C311—N31—N32—C33	179.86 (16)	C35—O351—C351—C352	169.43 (17)
N31—N32—C33—C34	−1.6 (2)	C356—C351—C352—C353	0.6 (3)
N31—N32—C33—C331	179.10 (18)	O351—C351—C352—C353	−179.26 (18)
N32—C33—C34—C35	0.9 (2)	C351—C352—C353—C354	−0.1 (3)

C331—C33—C34—C35	−179.9 (2)	C352—C353—C354—C355	−0.4 (3)
N32—C33—C34—C3	−177.16 (18)	C352—C353—C354—Cl54	178.92 (16)
C331—C33—C34—C3	2.1 (3)	C353—C354—C355—C356	0.4 (3)
C2—C3—C34—C35	−11.4 (3)	Cl54—C354—C355—C356	−178.93 (16)
C2—C3—C34—C33	166.1 (2)	C352—C351—C356—C355	−0.6 (3)
N32—N31—C35—O351	171.48 (15)	O351—C351—C356—C355	179.23 (18)
C311—N31—C35—O351	−6.4 (3)	C354—C355—C356—C351	0.1 (3)

Hydrogen-bond geometry (Å, º)

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C19—H19$\cdots$O1^i	0.93	2.25	3.161 (3)	165
C16—H16$\cdots Cg$2	0.93	2.98	3.882 (2)	165
C356—H356$\cdots Cg$1^{ii}	0.93	2.88	3.685 (2)	146

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

3-[3-Methyl-5-(naphthalen-2-yloxy)-1-phenyl-1*H***-pyrazol-4-yl]-1-[4-(prop-2-ynyloxy)phenyl]prop-2-en-1-one**

(Ie)

Crystal data

$C_{32}H_{24}N_2O_3$	$Z = 2$
$M_r = 484.53$	$F(000) = 508$
Triclinic, $P\bar{1}$	$D_x = 1.307$ Mg m^{-3}
$a = 8.8615$ (6) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 10.4973$ (7) Å	Cell parameters from 4376 reflections
$c = 13.6588$ (10) Å	$\theta = 3.0–25.2°$
$\alpha = 79.006$ (3)°	$\mu = 0.08$ mm^{-1}
$\beta = 89.412$ (3)°	$T = 297$ K
$\gamma = 80.971$ (3)°	Block, brown
$V = 1231.54$ (15) Å^3	0.20 × 0.16 × 0.16 mm

Data collection

Bruker APEXII diffractometer	30613 measured reflections
Radiation source: fine focussealed tube	4373 independent reflections
Graphite monochromator	3474 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.055$
Absorption correction: multi-scan (SADABS; Bruker, 2016)	$\theta_{max} = 25.2°$, $\theta_{min} = 3.4°$
	$h = −10\rightarrow10$
	$k = −12\rightarrow12$
$T_{min} = 0.898$, $T_{max} = 0.987$	$l = −16\rightarrow16$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.070$	$w = 1/[\sigma^2(F_o^2) + (0.0694P)^2 + 1.3194P]$
$wR(F^2) = 0.187$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} < 0.001$
4373 reflections	$\Delta\rho_{max} = 0.85$ e Å^{-3}
336 parameters	$\Delta\rho_{min} = −0.29$ e Å^{-3}
0 restraints	
Primary atom site location: dual	

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

Extinction coefficient: 0.114 (12)

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	0.4597 (3)	0.2692 (3)	0.9522 (2)	0.0466 (6)
O1	0.5161 (3)	0.1921 (2)	1.02722 (15)	0.0682 (7)
C2	0.5008 (3)	0.2472 (3)	0.8514 (2)	0.0479 (7)
H2	0.4710	0.3145	0.7970	0.057*
C3	0.5789 (3)	0.1344 (3)	0.8354 (2)	0.0456 (6)
H3	0.6058	0.0694	0.8916	0.055*
C11	0.3503 (3)	0.3873 (3)	0.96264 (18)	0.0421 (6)
C12	0.3407 (3)	0.4290 (3)	1.0532 (2)	0.0491 (7)
H12	0.4032	0.3815	1.1063	0.059*
C13	0.2408 (3)	0.5392 (3)	1.0669 (2)	0.0501 (7)
H13	0.2376	0.5664	1.1279	0.060*
C14	0.1454 (3)	0.6086 (3)	0.9883 (2)	0.0463 (6)
C15	0.1510 (3)	0.5671 (3)	0.8978 (2)	0.0539 (7)
H15	0.0854	0.6126	0.8457	0.065*
C16	0.2528 (3)	0.4595 (3)	0.8848 (2)	0.0516 (7)
H16	0.2572	0.4340	0.8232	0.062*
O14	0.0419 (2)	0.7179 (2)	0.99303 (15)	0.0600 (6)
C17	0.0309 (4)	0.7627 (3)	1.0852 (2)	0.0584 (8)
H17A	0.1258	0.7910	1.0999	0.070*
H17B	0.0129	0.6918	1.1387	0.070*
C18	−0.0948 (4)	0.8721 (3)	1.0781 (2)	0.0584 (8)
C19	−0.1963 (4)	0.9573 (4)	1.0761 (3)	0.0695 (9)
H19	−0.2776	1.0256	1.0745	0.083*
N31	0.6678 (2)	0.0973 (2)	0.58075 (16)	0.0441 (5)
N32	0.7581 (3)	−0.0141 (2)	0.63236 (16)	0.0481 (6)
C33	0.7313 (3)	−0.0118 (3)	0.72742 (19)	0.0439 (6)
C34	0.6271 (3)	0.1007 (3)	0.74080 (19)	0.0415 (6)
C35	0.5907 (3)	0.1663 (2)	0.64457 (19)	0.0423 (6)
C311	0.6675 (3)	0.1256 (3)	0.47431 (19)	0.0435 (6)
C312	0.5324 (3)	0.1747 (3)	0.4211 (2)	0.0591 (8)
H312	0.4408	0.1889	0.4542	0.071*
C313	0.5349 (4)	0.2025 (4)	0.3179 (2)	0.0681 (9)
H313	0.4444	0.2372	0.2819	0.082*
C314	0.6679 (4)	0.1798 (3)	0.2679 (2)	0.0631 (9)
H314	0.6683	0.1990	0.1986	0.076*
C315	0.8004 (4)	0.1285 (3)	0.3216 (2)	0.0640 (9)
H315	0.8910	0.1114	0.2881	0.077*

C316	0.8019 (3)	0.1014 (3)	0.4253 (2)	0.0535 (7)
H316	0.8927	0.0674	0.4610	0.064*
C331	0.8082 (4)	−0.1185 (3)	0.8078 (2)	0.0595 (8)
H33A	0.8606	−0.1889	0.7786	0.089*
H33B	0.7331	−0.1508	0.8529	0.089*
H33C	0.8805	−0.0843	0.8435	0.089*
O351	0.5097 (2)	0.28760 (18)	0.60811 (15)	0.0528 (5)
C351	0.2781 (4)	0.4338 (3)	0.6115 (2)	0.0550 (7)
H351	0.3356	0.5017	0.5971	0.066*
C352	0.3475 (3)	0.3071 (3)	0.62206 (19)	0.0507 (7)
C353	0.2669 (4)	0.2018 (3)	0.6420 (2)	0.0595 (8)
H353	0.3175	0.1160	0.6483	0.071*
C354	0.1103 (4)	0.2270 (3)	0.6523 (3)	0.0669 (9)
H354	0.0553	0.1573	0.6654	0.080*
C355	−0.1287 (4)	0.3815 (4)	0.6550 (3)	0.0695 (10)
H355	−0.1864	0.3137	0.6686	0.083*
C356	−0.1961 (4)	0.5079 (4)	0.6457 (3)	0.0737 (10)
H356	−0.3013	0.5257	0.6533	0.088*
C357	−0.1145 (5)	0.6132 (4)	0.6250 (3)	0.0791 (11)
H357	−0.1648	0.6991	0.6188	0.095*
C358	0.0361 (4)	0.5891 (4)	0.6142 (3)	0.0722 (10)
H358	0.0898	0.6596	0.6005	0.087*
C359	0.0346 (4)	0.3537 (3)	0.6434 (2)	0.0589 (8)
C360	0.1159 (3)	0.4629 (3)	0.62260 (19)	0.0447 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0466 (15)	0.0465 (15)	0.0434 (15)	0.0048 (12)	−0.0025 (12)	−0.0100 (12)
O1	0.0799 (15)	0.0664 (14)	0.0440 (12)	0.0306 (12)	−0.0081 (10)	−0.0086 (10)
C2	0.0506 (16)	0.0482 (15)	0.0404 (14)	0.0053 (12)	−0.0009 (12)	−0.0081 (12)
C3	0.0443 (14)	0.0458 (15)	0.0430 (14)	0.0025 (12)	−0.0013 (11)	−0.0073 (11)
C11	0.0435 (14)	0.0418 (14)	0.0369 (13)	0.0038 (11)	−0.0001 (11)	−0.0057 (11)
C12	0.0500 (16)	0.0516 (16)	0.0394 (14)	0.0096 (13)	−0.0072 (12)	−0.0066 (12)
C13	0.0572 (17)	0.0491 (16)	0.0403 (14)	0.0077 (13)	−0.0031 (12)	−0.0125 (12)
C14	0.0474 (15)	0.0408 (14)	0.0455 (15)	0.0065 (11)	0.0018 (12)	−0.0064 (11)
C15	0.0555 (17)	0.0577 (17)	0.0395 (15)	0.0138 (14)	−0.0085 (12)	−0.0047 (12)
C16	0.0578 (17)	0.0541 (17)	0.0371 (14)	0.0104 (13)	−0.0051 (12)	−0.0098 (12)
O14	0.0669 (13)	0.0537 (12)	0.0499 (12)	0.0219 (10)	−0.0050 (10)	−0.0115 (9)
C17	0.0588 (18)	0.0567 (18)	0.0560 (18)	0.0116 (14)	−0.0046 (14)	−0.0186 (14)
C18	0.0595 (18)	0.0551 (18)	0.0574 (18)	0.0067 (15)	0.0010 (14)	−0.0154 (14)
C19	0.069 (2)	0.069 (2)	0.065 (2)	0.0181 (18)	0.0005 (16)	−0.0209 (17)
N31	0.0445 (12)	0.0460 (12)	0.0375 (12)	0.0039 (10)	−0.0017 (9)	−0.0063 (9)
N32	0.0472 (13)	0.0491 (13)	0.0411 (12)	0.0104 (10)	−0.0011 (10)	−0.0058 (10)
C33	0.0415 (14)	0.0466 (15)	0.0399 (14)	0.0034 (11)	−0.0016 (11)	−0.0070 (11)
C34	0.0405 (13)	0.0416 (14)	0.0402 (14)	0.0016 (11)	−0.0006 (11)	−0.0093 (11)
C35	0.0400 (13)	0.0393 (13)	0.0442 (14)	0.0031 (11)	−0.0019 (11)	−0.0073 (11)
C311	0.0474 (15)	0.0425 (14)	0.0391 (14)	−0.0045 (11)	−0.0006 (11)	−0.0063 (11)

C312	0.0474 (16)	0.080 (2)	0.0448 (16)	−0.0008 (15)	−0.0030 (13)	−0.0073 (15)
C313	0.068 (2)	0.082 (2)	0.0475 (18)	−0.0024 (18)	−0.0128 (15)	−0.0033 (16)
C314	0.088 (2)	0.0583 (19)	0.0422 (16)	−0.0131 (17)	0.0016 (16)	−0.0058 (14)
C315	0.071 (2)	0.064 (2)	0.0561 (19)	−0.0086 (16)	0.0194 (16)	−0.0115 (15)
C316	0.0483 (16)	0.0547 (17)	0.0538 (17)	0.0009 (13)	0.0028 (13)	−0.0084 (13)
C331	0.0629 (19)	0.0568 (18)	0.0476 (16)	0.0169 (15)	0.0012 (14)	−0.0034 (13)
O351	0.0560 (12)	0.0400 (10)	0.0550 (12)	0.0056 (9)	−0.0027 (9)	−0.0010 (8)
C351	0.0598 (18)	0.0475 (16)	0.0546 (17)	0.0006 (13)	−0.0104 (14)	−0.0086 (13)
C352	0.0560 (17)	0.0508 (16)	0.0369 (14)	0.0199 (13)	−0.0108 (12)	−0.0100 (12)
C353	0.0554 (18)	0.0525 (17)	0.067 (2)	−0.0013 (14)	−0.0014 (15)	−0.0078 (14)
C354	0.064 (2)	0.0564 (19)	0.078 (2)	−0.0105 (16)	−0.0048 (17)	−0.0048 (16)
C355	0.0440 (17)	0.097 (3)	0.064 (2)	−0.0023 (17)	−0.0037 (14)	−0.0123 (18)
C356	0.0541 (19)	0.099 (3)	0.061 (2)	0.009 (2)	−0.0034 (16)	−0.0163 (19)
C357	0.077 (3)	0.087 (3)	0.065 (2)	0.025 (2)	−0.0158 (18)	−0.0249 (19)
C358	0.084 (3)	0.062 (2)	0.065 (2)	0.0198 (18)	−0.0179 (18)	−0.0212 (16)
C359	0.0627 (19)	0.067 (2)	0.0421 (16)	0.0060 (15)	−0.0094 (13)	−0.0100 (14)
C360	0.0479 (15)	0.0489 (15)	0.0351 (13)	0.0018 (12)	−0.0072 (11)	−0.0094 (11)

Geometric parameters (Å, º)

C1—O1	1.233 (3)	C311—C312	1.382 (4)
C1—C2	1.472 (4)	C312—C313	1.384 (4)
C1—C11	1.478 (4)	C312—H312	0.9300
C2—C3	1.330 (4)	C313—C314	1.367 (5)
C2—H2	0.9300	C313—H313	0.9300
C3—C34	1.446 (4)	C314—C315	1.369 (5)
C3—H3	0.9300	C314—H314	0.9300
C11—C12	1.385 (4)	C315—C316	1.390 (4)
C11—C16	1.399 (4)	C315—H315	0.9300
C12—C13	1.384 (4)	C316—H316	0.9300
C12—H12	0.9300	C331—H33A	0.9600
C13—C14	1.387 (4)	C331—H33B	0.9600
C13—H13	0.9300	C331—H33C	0.9600
C14—O14	1.363 (3)	O351—C352	1.435 (3)
C14—C15	1.384 (4)	C351—C352	1.356 (4)
C15—C16	1.370 (4)	C351—C360	1.434 (4)
C15—H15	0.9300	C351—H351	0.9300
C16—H16	0.9300	C352—C353	1.391 (4)
O14—C17	1.423 (3)	C353—C354	1.382 (5)
C17—C18	1.459 (4)	C353—H353	0.9300
C17—H17A	0.9700	C354—C359	1.376 (5)
C17—H17B	0.9700	C354—H354	0.9300
C18—C19	1.161 (4)	C355—C356	1.350 (5)
C19—H19	0.9300	C355—C359	1.444 (4)
N31—C35	1.351 (3)	C355—H355	0.9300
N31—N32	1.382 (3)	C356—C357	1.397 (6)
N31—C311	1.427 (3)	C356—H356	0.9300
N32—C33	1.322 (3)	C357—C358	1.331 (5)

C33—C34	1.420 (4)	C357—H357	0.9300
C33—C331	1.494 (4)	C358—C360	1.385 (4)
C34—C35	1.378 (4)	C358—H358	0.9300
C35—O351	1.365 (3)	C359—C360	1.430 (4)
C311—C316	1.372 (4)		
O1—C1—C2	121.3 (2)	C311—C312—C313	119.2 (3)
O1—C1—C11	120.0 (2)	C311—C312—H312	120.4
C2—C1—C11	118.8 (2)	C313—C312—H312	120.4
C3—C2—C1	122.2 (3)	C314—C313—C312	121.2 (3)
C3—C2—H2	118.9	C314—C313—H313	119.4
C1—C2—H2	118.9	C312—C313—H313	119.4
C2—C3—C34	127.7 (3)	C313—C314—C315	119.0 (3)
C2—C3—H3	116.2	C313—C314—H314	120.5
C34—C3—H3	116.2	C315—C314—H314	120.5
C12—C11—C16	117.6 (2)	C314—C315—C316	121.2 (3)
C12—C11—C1	119.5 (2)	C314—C315—H315	119.4
C16—C11—C1	122.8 (2)	C316—C315—H315	119.4
C13—C12—C11	121.9 (2)	C311—C316—C315	119.1 (3)
C13—C12—H12	119.0	C311—C316—H316	120.4
C11—C12—H12	119.0	C315—C316—H316	120.4
C12—C13—C14	119.1 (3)	C33—C331—H33A	109.5
C12—C13—H13	120.5	C33—C331—H33B	109.5
C14—C13—H13	120.5	H33A—C331—H33B	109.5
O14—C14—C15	115.7 (2)	C33—C331—H33C	109.5
O14—C14—C13	124.4 (2)	H33A—C331—H33C	109.5
C15—C14—C13	119.9 (2)	H33B—C331—H33C	109.5
C16—C15—C14	120.2 (2)	C35—O351—C352	118.0 (2)
C16—C15—H15	119.9	C352—C351—C360	119.9 (3)
C14—C15—H15	119.9	C352—C351—H351	120.0
C15—C16—C11	121.2 (3)	C360—C351—H351	120.0
C15—C16—H16	119.4	C351—C352—C353	122.5 (3)
C11—C16—H16	119.4	C351—C352—O351	116.0 (3)
C14—O14—C17	117.5 (2)	C353—C352—O351	121.5 (2)
O14—C17—C18	109.2 (2)	C354—C353—C352	118.9 (3)
O14—C17—H17A	109.8	C354—C353—H353	120.6
C18—C17—H17A	109.8	C352—C353—H353	120.6
O14—C17—H17B	109.8	C359—C354—C353	121.0 (3)
C18—C17—H17B	109.8	C359—C354—H354	119.5
H17A—C17—H17B	108.3	C353—C354—H354	119.5
C19—C18—C17	177.5 (4)	C356—C355—C359	118.8 (4)
C18—C19—H19	180.0	C356—C355—H355	120.6
C35—N31—N32	110.6 (2)	C359—C355—H355	120.6
C35—N31—C311	129.4 (2)	C355—C356—C357	122.7 (3)
N32—N31—C311	119.9 (2)	C355—C356—H356	118.6
C33—N32—N31	104.8 (2)	C357—C356—H356	118.6
N32—C33—C34	112.5 (2)	C358—C357—C356	119.3 (4)
N32—C33—C331	120.9 (2)	C358—C357—H357	120.4

C34—C33—C331	126.6 (2)	C356—C357—H357	120.4
C35—C34—C33	103.3 (2)	C357—C358—C360	122.4 (4)
C35—C34—C3	130.6 (2)	C357—C358—H358	118.8
C33—C34—C3	126.0 (2)	C360—C358—H358	118.8
N31—C35—O351	119.2 (2)	C354—C359—C360	120.8 (3)
N31—C35—C34	108.7 (2)	C354—C359—C355	121.6 (3)
O351—C35—C34	131.6 (2)	C360—C359—C355	117.6 (3)
C316—C311—C312	120.3 (3)	C358—C360—C359	119.3 (3)
C316—C311—N31	119.5 (2)	C358—C360—C351	123.7 (3)
C312—C311—N31	120.2 (2)	C359—C360—C351	117.0 (3)
O1—C1—C2—C3	11.5 (5)	C3—C34—C35—O351	6.6 (5)
C11—C1—C2—C3	−169.4 (3)	C35—N31—C311—C316	140.2 (3)
C1—C2—C3—C34	−179.5 (3)	N32—N31—C311—C316	−39.0 (4)
O1—C1—C11—C12	16.3 (4)	C35—N31—C311—C312	−40.9 (4)
C2—C1—C11—C12	−162.8 (3)	N32—N31—C311—C312	139.9 (3)
O1—C1—C11—C16	−162.8 (3)	C316—C311—C312—C313	−1.7 (5)
C2—C1—C11—C16	18.0 (4)	N31—C311—C312—C313	179.4 (3)
C16—C11—C12—C13	−1.1 (4)	C311—C312—C313—C314	1.3 (5)
C1—C11—C12—C13	179.7 (3)	C312—C313—C314—C315	0.1 (5)
C11—C12—C13—C14	1.3 (5)	C313—C314—C315—C316	−1.1 (5)
C12—C13—C14—O14	179.6 (3)	C312—C311—C316—C315	0.7 (4)
C12—C13—C14—C15	0.1 (5)	N31—C311—C316—C315	179.6 (3)
O14—C14—C15—C16	179.0 (3)	C314—C315—C316—C311	0.7 (5)
C13—C14—C15—C16	−1.5 (5)	N31—C35—O351—C352	118.5 (3)
C14—C15—C16—C11	1.6 (5)	C34—C35—O351—C352	−70.6 (4)
C12—C11—C16—C15	−0.3 (5)	C360—C351—C352—C353	1.1 (4)
C1—C11—C16—C15	178.8 (3)	C360—C351—C352—O351	179.0 (2)
C15—C14—O14—C17	179.1 (3)	C35—O351—C352—C351	161.8 (2)
C13—C14—O14—C17	−0.4 (4)	C35—O351—C352—C353	−20.2 (4)
C14—O14—C17—C18	−174.7 (3)	C351—C352—C353—C354	−0.5 (5)
C35—N31—N32—C33	1.2 (3)	O351—C352—C353—C354	−178.4 (3)
C311—N31—N32—C33	−179.5 (2)	C352—C353—C354—C359	−0.2 (5)
N31—N32—C33—C34	−1.3 (3)	C359—C355—C356—C357	0.2 (5)
N31—N32—C33—C331	179.5 (3)	C355—C356—C357—C358	−0.2 (6)
N32—C33—C34—C35	0.8 (3)	C356—C357—C358—C360	0.1 (5)
C331—C33—C34—C35	−180.0 (3)	C353—C354—C359—C360	0.5 (5)
N32—C33—C34—C3	−177.6 (3)	C353—C354—C359—C355	−179.4 (3)
C331—C33—C34—C3	1.6 (5)	C356—C355—C359—C354	179.8 (3)
C2—C3—C34—C35	−9.1 (5)	C356—C355—C359—C360	0.0 (5)
C2—C3—C34—C33	168.9 (3)	C357—C358—C360—C359	0.1 (5)
N32—N31—C35—O351	172.1 (2)	C357—C358—C360—C351	−179.9 (3)
C311—N31—C35—O351	−7.1 (4)	C354—C359—C360—C358	−180.0 (3)
N32—N31—C35—C34	−0.7 (3)	C355—C359—C360—C358	−0.2 (4)
C311—N31—C35—C34	−180.0 (3)	C354—C359—C360—C351	0.0 (4)
C33—C34—C35—N31	−0.1 (3)	C355—C359—C360—C351	179.9 (3)
C3—C34—C35—N31	178.3 (3)	C352—C351—C360—C358	179.2 (3)
C33—C34—C35—O351	−171.7 (3)	C352—C351—C360—C359	−0.8 (4)

Hydrogen-bond geometry (Å, º)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C19—H19⋯O1^i	0.93	2.32	3.233 (5)	165
C353—H353⋯Cg1^{ii}	0.93	2.86	3.708 (3)	152

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

1-(4-Azidophenyl)-3-[3-methyl-5-(2-methylphenoxy)-1-phenyl-1*H*-pyrazol-4-yl]prop-2-en-1-one (IIa)

Crystal data

$C_{26}H_{21}N_5O_2$	$Z = 2$
$M_r = 435.48$	$F(000) = 456$
Triclinic, $P\bar{1}$	$D_x = 1.255$ Mg m^{-3}
$a = 9.8432$ (6) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 11.7441$ (7) Å	Cell parameters from 4051 reflections
$c = 12.3005$ (7) Å	$\theta = 3.2–25.1°$
$\alpha = 114.120$ (2)°	$\mu = 0.08$ mm^{-1}
$\beta = 111.139$ (2)°	$T = 297$ K
$\gamma = 96.537$ (2)°	Block, brown
$V = 1152.06$ (12) Å^3	$0.20 \times 0.20 \times 0.18$ mm

Data collection

Bruker APEXII diffractometer	17379 measured reflections
Radiation source: fine focussealed tube	4050 independent reflections
Graphite monochromator	2957 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.048$
Absorption correction: multi-scan (SADABS; Bruker, 2016)	$\theta_{max} = 25.1°$, $\theta_{min} = 3.5°$
$T_{min} = 0.868$, $T_{max} = 0.985$	$h = -11\rightarrow11$
	$k = -13\rightarrow13$
	$l = -14\rightarrow14$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.050$	$w = 1/[\sigma^2(F_o^2) + (0.0547P)^2 + 0.4937P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.148$	
$S = 1.10$	$(\Delta/\sigma)_{max} < 0.001$
4050 reflections	$\Delta\rho_{max} = 0.19$ e Å^{-3}
301 parameters	$\Delta\rho_{min} = -0.21$ e Å^{-3}
0 restraints	Extinction correction: SHELXL, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: dual	Extinction coefficient: 0.179 (14)

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6025 (2)	0.4013 (2)	0.8884 (2)	0.0473 (5)
O1	0.66955 (18)	0.33036 (17)	0.9214 (2)	0.0733 (5)
C2	0.4353 (2)	0.3679 (2)	0.8333 (2)	0.0465 (5)
H2	0.3862	0.4229	0.8090	0.056*
C3	0.3533 (2)	0.2591 (2)	0.8180 (2)	0.0459 (5)
H3	0.4098	0.2111	0.8489	0.055*
C11	0.6922 (2)	0.52181 (19)	0.90264 (19)	0.0415 (5)
C12	0.8501 (2)	0.5646 (2)	0.9800 (2)	0.0462 (5)
H12	0.8940	0.5193	1.0229	0.055*
C13	0.9434 (2)	0.6725 (2)	0.9947 (2)	0.0471 (5)
H13	1.0487	0.6991	1.0463	0.057*
C14	0.8781 (2)	0.7402 (2)	0.9317 (2)	0.0480 (5)
C15	0.7212 (2)	0.7008 (2)	0.8556 (2)	0.0557 (6)
H15	0.6776	0.7473	0.8142	0.067*
C16	0.6292 (2)	0.5923 (2)	0.8413 (2)	0.0519 (5)
H16	0.5239	0.5662	0.7900	0.062*
N14	0.9637 (2)	0.85203 (19)	0.9401 (2)	0.0656 (6)
N15	1.1040 (3)	0.8907 (2)	1.0131 (2)	0.0693 (6)
N16	1.2312 (3)	0.9373 (3)	1.0751 (3)	0.1029 (10)
N31	−0.06215 (19)	0.16164 (16)	0.66089 (17)	0.0471 (4)
N32	−0.0398 (2)	0.05926 (17)	0.68563 (18)	0.0511 (5)
C33	0.1109 (2)	0.0864 (2)	0.7460 (2)	0.0469 (5)
C34	0.1897 (2)	0.20522 (19)	0.7604 (2)	0.0433 (5)
C35	0.0727 (2)	0.24912 (19)	0.7052 (2)	0.0442 (5)
C311	−0.2141 (2)	0.1559 (2)	0.5825 (2)	0.0471 (5)
C312	−0.2664 (3)	0.2627 (2)	0.6210 (3)	0.0611 (6)
H312	−0.2034	0.3406	0.6988	0.073*
C313	−0.4142 (3)	0.2524 (3)	0.5418 (3)	0.0711 (7)
H313	−0.4506	0.3239	0.5667	0.085*
C314	−0.5075 (3)	0.1371 (3)	0.4269 (3)	0.0735 (7)
H314	−0.6063	0.1312	0.3741	0.088*
C315	−0.4550 (3)	0.0308 (3)	0.3900 (3)	0.0719 (7)
H315	−0.5184	−0.0472	0.3124	0.086*
C316	−0.3082 (3)	0.0397 (2)	0.4679 (2)	0.0578 (6)
H316	−0.2728	−0.0324	0.4433	0.069*
C331	0.1796 (3)	−0.0017 (2)	0.7929 (3)	0.0645 (6)
H33A	0.1019	−0.0622	0.7896	0.097*
H33B	0.2587	0.0498	0.8829	0.097*
H33C	0.2225	−0.0495	0.7365	0.097*
O351	0.07382 (17)	0.36198 (13)	0.69897 (15)	0.0522 (4)
C351	0.1292 (2)	0.38472 (19)	0.6168 (2)	0.0434 (5)
C352	0.1738 (2)	0.5152 (2)	0.6479 (2)	0.0522 (5)
C353	0.2282 (3)	0.5403 (3)	0.5678 (3)	0.0690 (7)
H353	0.2599	0.6264	0.5855	0.083*
C354	0.2367 (3)	0.4418 (3)	0.4633 (3)	0.0780 (8)

H354	0.2741	0.4618	0.4116	0.094*
C355	0.1900 (3)	0.3135 (3)	0.4344 (3)	0.0699 (7)
H355	0.1952	0.2469	0.3630	0.084*
C356	0.1353 (3)	0.2839 (2)	0.5119 (2)	0.0547 (6)
H356	0.1032	0.1976	0.4934	0.066*
C357	0.1657 (4)	0.6222 (2)	0.7625 (3)	0.0777 (8)
H35A	0.1876	0.7031	0.7608	0.116*
H35B	0.2393	0.6314	0.8446	0.116*
H35C	0.0649	0.6010	0.7559	0.116*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0414 (11)	0.0494 (12)	0.0507 (12)	0.0123 (9)	0.0182 (9)	0.0266 (10)
O1	0.0471 (9)	0.0702 (11)	0.1105 (14)	0.0170 (8)	0.0231 (9)	0.0615 (11)
C2	0.0378 (11)	0.0456 (11)	0.0520 (12)	0.0110 (9)	0.0178 (9)	0.0227 (9)
C3	0.0441 (11)	0.0436 (11)	0.0474 (11)	0.0122 (9)	0.0214 (9)	0.0194 (9)
C11	0.0362 (10)	0.0439 (11)	0.0414 (10)	0.0110 (8)	0.0166 (8)	0.0192 (9)
C12	0.0382 (11)	0.0490 (12)	0.0531 (12)	0.0147 (9)	0.0193 (9)	0.0270 (10)
C13	0.0364 (10)	0.0470 (11)	0.0531 (12)	0.0111 (9)	0.0171 (9)	0.0231 (10)
C14	0.0467 (12)	0.0430 (11)	0.0500 (12)	0.0074 (9)	0.0213 (9)	0.0207 (9)
C15	0.0478 (12)	0.0569 (13)	0.0614 (13)	0.0119 (10)	0.0157 (10)	0.0366 (11)
C16	0.0375 (11)	0.0573 (13)	0.0544 (12)	0.0091 (9)	0.0127 (9)	0.0298 (11)
N14	0.0517 (12)	0.0558 (12)	0.0775 (14)	0.0002 (9)	0.0136 (10)	0.0397 (11)
N15	0.0585 (14)	0.0588 (13)	0.0799 (14)	0.0032 (10)	0.0187 (12)	0.0388 (11)
N16	0.0565 (15)	0.0956 (19)	0.130 (2)	−0.0054 (13)	0.0099 (15)	0.0668 (18)
N31	0.0417 (9)	0.0424 (9)	0.0551 (10)	0.0076 (7)	0.0203 (8)	0.0245 (8)
N32	0.0485 (11)	0.0443 (10)	0.0612 (11)	0.0080 (8)	0.0232 (9)	0.0291 (9)
C33	0.0492 (12)	0.0416 (11)	0.0514 (12)	0.0091 (9)	0.0252 (10)	0.0228 (9)
C34	0.0422 (11)	0.0403 (10)	0.0463 (11)	0.0090 (8)	0.0219 (9)	0.0191 (9)
C35	0.0463 (11)	0.0375 (10)	0.0512 (11)	0.0100 (9)	0.0256 (9)	0.0212 (9)
C311	0.0418 (11)	0.0519 (12)	0.0499 (11)	0.0116 (9)	0.0236 (9)	0.0246 (10)
C312	0.0542 (14)	0.0539 (14)	0.0674 (15)	0.0149 (11)	0.0269 (12)	0.0235 (12)
C313	0.0613 (16)	0.0702 (17)	0.0894 (19)	0.0309 (13)	0.0369 (15)	0.0399 (15)
C314	0.0505 (14)	0.090 (2)	0.0758 (17)	0.0230 (14)	0.0233 (13)	0.0408 (16)
C315	0.0518 (14)	0.0762 (18)	0.0609 (15)	0.0108 (13)	0.0165 (12)	0.0197 (13)
C316	0.0495 (13)	0.0558 (13)	0.0584 (13)	0.0142 (10)	0.0248 (11)	0.0193 (11)
C331	0.0663 (15)	0.0588 (14)	0.0765 (16)	0.0175 (12)	0.0304 (13)	0.0418 (13)
O351	0.0583 (9)	0.0427 (8)	0.0694 (10)	0.0185 (7)	0.0374 (8)	0.0310 (7)
C351	0.0370 (10)	0.0452 (11)	0.0485 (11)	0.0107 (8)	0.0155 (9)	0.0269 (9)
C352	0.0491 (12)	0.0476 (12)	0.0575 (13)	0.0125 (10)	0.0163 (10)	0.0307 (10)
C353	0.0742 (17)	0.0632 (16)	0.0749 (17)	0.0127 (13)	0.0264 (14)	0.0464 (14)
C354	0.089 (2)	0.091 (2)	0.0798 (18)	0.0248 (16)	0.0440 (16)	0.0589 (17)
C355	0.0818 (18)	0.0828 (18)	0.0608 (15)	0.0339 (15)	0.0380 (14)	0.0411 (14)
C356	0.0580 (13)	0.0497 (12)	0.0547 (13)	0.0166 (10)	0.0219 (11)	0.0267 (11)
C357	0.104 (2)	0.0467 (14)	0.0809 (18)	0.0248 (14)	0.0384 (16)	0.0318 (13)

Geometric parameters (Å, º)

C1—O1	1.226 (3)	C311—C316	1.380 (3)
C1—C2	1.468 (3)	C312—C313	1.386 (3)
C1—C11	1.488 (3)	C312—H312	0.9300
C2—C3	1.334 (3)	C313—C314	1.375 (4)
C2—H2	0.9300	C313—H313	0.9300
C3—C34	1.440 (3)	C314—C315	1.373 (4)
C3—H3	0.9300	C314—H314	0.9300
C11—C16	1.389 (3)	C315—C316	1.379 (3)
C11—C12	1.392 (3)	C315—H315	0.9300
C12—C13	1.382 (3)	C316—H316	0.9300
C12—H12	0.9300	C331—H33A	0.9600
C13—C14	1.380 (3)	C331—H33B	0.9600
C13—H13	0.9300	C331—H33C	0.9600
C14—C15	1.384 (3)	O351—C351	1.405 (2)
C14—N14	1.422 (3)	C351—C356	1.377 (3)
C15—C16	1.383 (3)	C351—C352	1.391 (3)
C15—H15	0.9300	C352—C353	1.389 (3)
C16—H16	0.9300	C352—C357	1.493 (3)
N14—N15	1.245 (3)	C353—C354	1.372 (4)
N15—N16	1.124 (3)	C353—H353	0.9300
N31—C35	1.351 (3)	C354—C355	1.377 (4)
N31—N32	1.378 (2)	C354—H354	0.9300
N31—C311	1.433 (3)	C355—C356	1.385 (3)
N32—C33	1.326 (3)	C355—H355	0.9300
C33—C34	1.426 (3)	C356—H356	0.9300
C33—C331	1.493 (3)	C357—H35A	0.9600
C34—C35	1.382 (3)	C357—H35B	0.9600
C35—O351	1.358 (2)	C357—H35C	0.9600
C311—C312	1.379 (3)		
O1—C1—C2	120.91 (19)	C311—C312—H312	120.5
O1—C1—C11	119.34 (19)	C313—C312—H312	120.5
C2—C1—C11	119.74 (18)	C314—C313—C312	120.5 (2)
C3—C2—C1	120.4 (2)	C314—C313—H313	119.8
C3—C2—H2	119.8	C312—C313—H313	119.8
C1—C2—H2	119.8	C315—C314—C313	120.1 (2)
C2—C3—C34	129.2 (2)	C315—C314—H314	119.9
C2—C3—H3	115.4	C313—C314—H314	119.9
C34—C3—H3	115.4	C314—C315—C316	120.0 (2)
C16—C11—C12	118.03 (19)	C314—C315—H315	120.0
C16—C11—C1	124.29 (18)	C316—C315—H315	120.0
C12—C11—C1	117.66 (18)	C315—C316—C311	119.9 (2)
C13—C12—C11	121.75 (19)	C315—C316—H316	120.1
C13—C12—H12	119.1	C311—C316—H316	120.1
C11—C12—H12	119.1	C33—C331—H33A	109.5
C14—C13—C12	119.10 (19)	C33—C331—H33B	109.5

C14—C13—H13	120.5	H33A—C331—H33B	109.5
C12—C13—H13	120.5	C33—C331—H33C	109.5
C13—C14—C15	120.35 (19)	H33A—C331—H33C	109.5
C13—C14—N14	123.5 (2)	H33B—C331—H33C	109.5
C15—C14—N14	116.1 (2)	C35—O351—C351	119.16 (15)
C16—C15—C14	120.0 (2)	C356—C351—C352	123.0 (2)
C16—C15—H15	120.0	C356—C351—O351	121.83 (18)
C14—C15—H15	120.0	C352—C351—O351	115.11 (18)
C15—C16—C11	120.80 (19)	C353—C352—C351	116.3 (2)
C15—C16—H16	119.6	C353—C352—C357	122.0 (2)
C11—C16—H16	119.6	C351—C352—C357	121.7 (2)
N15—N14—C14	116.1 (2)	C354—C353—C352	121.9 (2)
N16—N15—N14	171.8 (3)	C354—C353—H353	119.1
C35—N31—N32	111.11 (17)	C352—C353—H353	119.1
C35—N31—C311	129.06 (18)	C353—C354—C355	120.3 (2)
N32—N31—C311	119.38 (16)	C353—C354—H354	119.8
C33—N32—N31	104.82 (16)	C355—C354—H354	119.8
N32—C33—C34	112.21 (18)	C354—C355—C356	119.8 (3)
N32—C33—C331	120.41 (18)	C354—C355—H355	120.1
C34—C33—C331	127.4 (2)	C356—C355—H355	120.1
C35—C34—C33	103.40 (17)	C351—C356—C355	118.7 (2)
C35—C34—C3	131.01 (19)	C351—C356—H356	120.7
C33—C34—C3	125.58 (19)	C355—C356—H356	120.7
N31—C35—O351	119.52 (18)	C352—C357—H35A	109.5
N31—C35—C34	108.45 (18)	C352—C357—H35B	109.5
O351—C35—C34	131.82 (18)	H35A—C357—H35B	109.5
C312—C311—C316	120.5 (2)	C352—C357—H35C	109.5
C312—C311—N31	121.14 (19)	H35A—C357—H35C	109.5
C316—C311—N31	118.35 (19)	H35B—C357—H35C	109.5
C311—C312—C313	119.0 (2)		

O1—C1—C2—C3	−0.1 (3)	C311—N31—C35—C34	−171.57 (19)
C11—C1—C2—C3	178.86 (19)	C33—C34—C35—N31	−0.9 (2)
C1—C2—C3—C34	−176.53 (19)	C3—C34—C35—N31	178.1 (2)
O1—C1—C11—C16	164.2 (2)	C33—C34—C35—O351	173.7 (2)
C2—C1—C11—C16	−14.8 (3)	C3—C34—C35—O351	−7.3 (4)
O1—C1—C11—C12	−14.2 (3)	C35—N31—C311—C312	−53.4 (3)
C2—C1—C11—C12	166.82 (18)	N32—N31—C311—C312	135.1 (2)
C16—C11—C12—C13	−1.0 (3)	C35—N31—C311—C316	127.5 (2)
C1—C11—C12—C13	177.49 (19)	N32—N31—C311—C316	−44.0 (3)
C11—C12—C13—C14	0.4 (3)	C316—C311—C312—C313	−0.9 (4)
C12—C13—C14—C15	0.4 (3)	N31—C311—C312—C313	−180.0 (2)
C12—C13—C14—N14	−179.8 (2)	C311—C312—C313—C314	0.2 (4)
C13—C14—C15—C16	−0.7 (3)	C312—C313—C314—C315	0.4 (4)
N14—C14—C15—C16	179.5 (2)	C313—C314—C315—C316	−0.3 (4)
C14—C15—C16—C11	0.1 (4)	C314—C315—C316—C311	−0.4 (4)
C12—C11—C16—C15	0.7 (3)	C312—C311—C316—C315	1.0 (3)
C1—C11—C16—C15	−177.7 (2)	N31—C311—C316—C315	−179.9 (2)

C13—C14—N14—N15	−2.7 (3)	N31—C35—O351—C351	−115.8 (2)
C15—C14—N14—N15	177.1 (2)	C34—C35—O351—C351	70.1 (3)
C35—N31—N32—C33	0.1 (2)	C35—O351—C351—C356	21.6 (3)
C311—N31—N32—C33	173.08 (17)	C35—O351—C351—C352	−159.61 (18)
N31—N32—C33—C34	−0.7 (2)	C356—C351—C352—C353	−1.0 (3)
N31—N32—C33—C331	177.90 (19)	O351—C351—C352—C353	−179.69 (19)
N32—C33—C34—C35	1.0 (2)	C356—C351—C352—C357	180.0 (2)
C331—C33—C34—C35	−177.5 (2)	O351—C351—C352—C357	1.3 (3)
N32—C33—C34—C3	−178.03 (18)	C351—C352—C353—C354	0.4 (4)
C331—C33—C34—C3	3.5 (3)	C357—C352—C353—C354	179.4 (3)
C2—C3—C34—C35	0.2 (4)	C352—C353—C354—C355	0.3 (4)
C2—C3—C34—C33	179.0 (2)	C353—C354—C355—C356	−0.4 (4)
N32—N31—C35—O351	−174.87 (17)	C352—C351—C356—C355	0.8 (3)
C311—N31—C35—O351	13.1 (3)	O351—C351—C356—C355	179.5 (2)
N32—N31—C35—C34	0.5 (2)	C354—C355—C356—C351	−0.1 (4)

Hydrogen-bond geometry (Å, º)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C357—H35B⋯O1[i]	0.96	2.51	3.396 (4)	154

Symmetry code: (i) −x+1, −y+1, −z+2.

1-(4-Azidophenyl)-3-[5-(2,4-dichlorophenoxy)-3-methyl-1-phenyl-1H-pyrazol-4-yl]prop-2-en-1-one (IId)

Crystal data

$C_{25}H_{17}Cl_2N_5O_2$	$F(000) = 2016$
$M_r = 490.33$	$D_x = 1.377$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 28.1916$ (17) Å	Cell parameters from 4176 reflections
$b = 8.0537$ (5) Å	$\theta = 2.9–25.0°$
$c = 22.0446$ (12) Å	$\mu = 0.31$ mm^{-1}
$\beta = 109.070$ (1)°	$T = 297$ K
$V = 4730.5$ (5) Å^3	Block, colourless
$Z = 8$	$0.18 \times 0.15 \times 0.15$ mm

Data collection

Bruker APEXII diffractometer	31508 measured reflections
Radiation source: fine focussealed tube	4174 independent reflections
Graphite monochromator	3181 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.049$
Absorption correction: multi-scan (SADABS; Bruker, 2016)	$\theta_{max} = 25.0°$, $\theta_{min} = 2.9°$
$T_{min} = 0.881$, $T_{max} = 0.955$	$h = -33 \rightarrow 33$
	$k = -9 \rightarrow 9$
	$l = -26 \rightarrow 21$

Refinement

Refinement on F^2	4174 reflections
Least-squares matrix: full	382 parameters
$R[F^2 > 2\sigma(F^2)] = 0.086$	291 restraints
$wR(F^2) = 0.155$	Primary atom site location: dual
$S = 1.36$	

Hydrogen site location: inferred from
   neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0265P)^2 + 10.5248P]$
   where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Extinction correction: SHELXL,
   Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Extinction coefficient: 0.0018 (3)

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.42395 (14)	0.7441 (5)	0.5060 (2)	0.0497 (10)	
O1	0.42708 (11)	0.8068 (4)	0.55744 (16)	0.0731 (10)	
C2	0.46893 (14)	0.7129 (5)	0.48791 (19)	0.0509 (10)	
H2	0.4658	0.6524	0.4509	0.061*	
C3	0.51394 (14)	0.7686 (5)	0.52294 (19)	0.0505 (10)	
H3	0.5154	0.8257	0.5602	0.061*	
C11	0.37362 (14)	0.6992 (5)	0.45998 (19)	0.0472 (10)	
C12	0.33110 (15)	0.7460 (6)	0.4740 (2)	0.0637 (12)	
H12	0.3346	0.8036	0.5118	0.076*	
C13	0.28398 (17)	0.7089 (7)	0.4332 (2)	0.0741 (14)	
H13	0.2558	0.7415	0.4433	0.089*	
C14	0.27855 (15)	0.6239 (6)	0.3778 (2)	0.0624 (12)	
C15	0.31991 (16)	0.5748 (6)	0.3626 (2)	0.0665 (13)	
H15	0.3162	0.5164	0.3250	0.080*	
C16	0.36695 (15)	0.6129 (6)	0.4038 (2)	0.0616 (12)	
H16	0.3950	0.5796	0.3935	0.074*	
N14	0.22805 (15)	0.5915 (6)	0.3381 (2)	0.0911 (15)	
N15	0.22323 (16)	0.4999 (7)	0.2916 (3)	0.0912 (15)	
N16	0.2133 (2)	0.4157 (9)	0.2483 (3)	0.128 (2)	
N31	0.61980 (11)	0.6957 (4)	0.46689 (15)	0.0498 (9)	
N32	0.64431 (12)	0.7692 (5)	0.52519 (15)	0.0577 (10)	
C33	0.60890 (15)	0.8028 (5)	0.55055 (19)	0.0539 (11)	
C34	0.56086 (13)	0.7514 (5)	0.51031 (18)	0.0455 (9)	
C35	0.56997 (13)	0.6852 (5)	0.45798 (18)	0.0449 (9)	
C311	0.64806 (14)	0.6459 (5)	0.42690 (18)	0.0489 (10)	
C312	0.63370 (15)	0.5118 (6)	0.3859 (2)	0.0576 (11)	
H312	0.6047	0.4527	0.3832	0.069*	
C313	0.66276 (18)	0.4666 (6)	0.3491 (2)	0.0702 (13)	
H313	0.6530	0.3781	0.3207	0.084*	
C314	0.70616 (19)	0.5516 (7)	0.3541 (2)	0.0782 (15)	
H314	0.7257	0.5199	0.3293	0.094*	
C315	0.72076 (17)	0.6827 (7)	0.3955 (3)	0.0784 (15)	
H315	0.7505	0.7383	0.3993	0.094*	

C316	0.69143 (16)	0.7326 (6)	0.4316 (2)	0.0672 (13)
H316	0.7008	0.8237	0.4588	0.081*
C331	0.62281 (17)	0.8821 (7)	0.6150 (2)	0.0720 (14)
H33A	0.6193	0.8029	0.6457	0.108*
H33B	0.6011	0.9752	0.6133	0.108*
H33C	0.6570	0.9194	0.6274	0.108*
O351	0.53813 (9)	0.6158 (3)	0.40327 (12)	0.0483 (7)	0.55 (4)
C351	0.5093 (7)	0.7253 (10)	0.3559 (10)	0.030 (5)	0.55 (4)
C352	0.4689 (8)	0.6575 (11)	0.3090 (11)	0.038 (5)	0.55 (4)
Cl52	0.4590 (5)	0.4465 (11)	0.3077 (7)	0.118 (4)	0.55 (4)
C353	0.4349 (9)	0.7567 (14)	0.2647 (12)	0.048 (6)	0.55 (4)
H353	0.4104	0.7108	0.2295	0.058*	0.55 (4)
C354	0.4384 (7)	0.9280 (13)	0.2743 (10)	0.039 (5)	0.55 (4)
Cl54	0.3988 (2)	1.0506 (7)	0.2131 (2)	0.067 (3)	0.55 (4)
C355	0.4787 (8)	0.9973 (14)	0.3206 (11)	0.043 (5)	0.55 (4)
H355	0.4832	1.1119	0.3225	0.051*	0.55 (4)
C356	0.5125 (8)	0.8960 (12)	0.3640 (12)	0.047 (6)	0.55 (4)
H356	0.5374	0.9420	0.3987	0.056*	0.55 (4)
O451	0.53813 (9)	0.6158 (3)	0.40327 (12)	0.0483 (7)	0.45 (4)
C451	0.5023 (8)	0.7239 (13)	0.3633 (11)	0.031 (5)	0.45 (4)
C452	0.4619 (10)	0.6533 (14)	0.3172 (14)	0.040 (7)	0.45 (4)
Cl62	0.4539 (3)	0.4410 (9)	0.3133 (5)	0.058 (3)	0.45 (4)
C453	0.4287 (11)	0.7504 (17)	0.2708 (15)	0.047 (6)	0.45 (4)
H453	0.3980	0.7085	0.2450	0.056*	0.45 (4)
C454	0.4429 (9)	0.913 (2)	0.2639 (13)	0.050 (7)	0.45 (4)
Cl64	0.3977 (4)	1.0402 (17)	0.2112 (5)	0.128 (6)	0.45 (4)
C455	0.4832 (9)	0.9854 (19)	0.3095 (14)	0.045 (6)	0.45 (4)
H455	0.4889	1.0989	0.3086	0.054*	0.45 (4)
C456	0.5150 (8)	0.8875 (16)	0.3564 (13)	0.036 (5)	0.45 (4)
H456	0.5450	0.9310	0.3835	0.043*	0.45 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.048 (2)	0.045 (2)	0.056 (3)	−0.0079 (19)	0.016 (2)	−0.003 (2)
O1	0.0606 (19)	0.091 (3)	0.072 (2)	−0.0197 (17)	0.0267 (16)	−0.0303 (19)
C2	0.048 (2)	0.056 (3)	0.047 (2)	−0.0035 (19)	0.0118 (19)	−0.003 (2)
C3	0.051 (2)	0.053 (2)	0.045 (2)	−0.004 (2)	0.0127 (19)	−0.0012 (19)
C11	0.048 (2)	0.043 (2)	0.051 (2)	0.0000 (18)	0.0167 (19)	0.0025 (19)
C12	0.051 (3)	0.079 (3)	0.061 (3)	0.006 (2)	0.018 (2)	−0.008 (2)
C13	0.049 (3)	0.100 (4)	0.071 (3)	0.011 (3)	0.017 (2)	−0.004 (3)
C14	0.045 (2)	0.069 (3)	0.065 (3)	−0.001 (2)	0.006 (2)	0.007 (2)
C15	0.051 (3)	0.079 (3)	0.063 (3)	−0.001 (2)	0.011 (2)	−0.016 (3)
C16	0.044 (2)	0.072 (3)	0.070 (3)	−0.002 (2)	0.019 (2)	−0.010 (3)
N14	0.053 (3)	0.113 (4)	0.091 (3)	0.001 (2)	0.003 (2)	−0.017 (3)
N15	0.061 (3)	0.122 (4)	0.077 (3)	−0.009 (3)	0.004 (3)	−0.003 (3)
N16	0.105 (4)	0.175 (6)	0.087 (4)	−0.023 (4)	0.009 (3)	−0.030 (4)
N31	0.0388 (18)	0.061 (2)	0.0452 (19)	−0.0027 (16)	0.0082 (15)	0.0027 (16)

N32	0.0398 (18)	0.081 (3)	0.044 (2)	−0.0093 (18)	0.0023 (15)	0.0002 (18)
C33	0.045 (2)	0.066 (3)	0.046 (2)	−0.008 (2)	0.0093 (19)	0.002 (2)
C34	0.038 (2)	0.049 (2)	0.044 (2)	−0.0037 (18)	0.0057 (17)	0.0053 (19)
C35	0.037 (2)	0.047 (2)	0.044 (2)	−0.0005 (17)	0.0041 (17)	0.0081 (19)
C311	0.038 (2)	0.057 (3)	0.046 (2)	0.0033 (19)	0.0063 (18)	0.012 (2)
C312	0.047 (2)	0.059 (3)	0.065 (3)	0.004 (2)	0.016 (2)	0.010 (2)
C313	0.063 (3)	0.074 (3)	0.072 (3)	0.015 (3)	0.020 (3)	−0.002 (3)
C314	0.065 (3)	0.103 (4)	0.073 (3)	0.016 (3)	0.032 (3)	0.006 (3)
C315	0.052 (3)	0.105 (4)	0.081 (4)	−0.010 (3)	0.025 (3)	0.006 (3)
C316	0.051 (3)	0.083 (3)	0.064 (3)	−0.007 (2)	0.015 (2)	−0.001 (3)
C331	0.057 (3)	0.103 (4)	0.050 (3)	−0.017 (3)	0.010 (2)	−0.012 (3)
O351	0.0413 (14)	0.0446 (15)	0.0480 (16)	−0.0012 (12)	−0.0005 (12)	−0.0026 (12)
C351	0.019 (5)	0.035 (6)	0.043 (8)	−0.004 (4)	0.018 (5)	−0.006 (4)
C352	0.035 (6)	0.041 (6)	0.043 (8)	0.001 (4)	0.017 (7)	−0.008 (5)
Cl52	0.132 (7)	0.064 (4)	0.115 (6)	−0.019 (4)	−0.018 (4)	−0.012 (4)
C353	0.035 (7)	0.067 (9)	0.039 (8)	−0.011 (6)	0.008 (6)	−0.013 (6)
C354	0.042 (6)	0.046 (6)	0.030 (7)	0.028 (5)	0.013 (6)	0.010 (5)
Cl54	0.058 (3)	0.068 (4)	0.051 (3)	0.033 (2)	−0.014 (3)	0.020 (2)
C355	0.046 (6)	0.045 (6)	0.039 (7)	0.009 (5)	0.017 (7)	−0.007 (5)
C356	0.052 (10)	0.049 (8)	0.035 (7)	0.000 (6)	0.010 (7)	−0.016 (6)
O451	0.0413 (14)	0.0446 (15)	0.0480 (16)	−0.0012 (12)	−0.0005 (12)	−0.0026 (12)
C451	0.026 (8)	0.057 (9)	0.018 (5)	0.000 (5)	0.017 (6)	0.000 (5)
C452	0.040 (9)	0.050 (8)	0.037 (8)	−0.011 (6)	0.021 (7)	−0.006 (6)
Cl62	0.052 (4)	0.036 (3)	0.073 (4)	−0.012 (2)	0.001 (2)	−0.013 (2)
C453	0.035 (8)	0.067 (10)	0.039 (8)	0.002 (7)	0.011 (8)	0.000 (7)
C454	0.042 (8)	0.077 (11)	0.036 (9)	0.005 (8)	0.020 (6)	−0.008 (7)
Cl64	0.103 (8)	0.150 (10)	0.130 (9)	0.036 (6)	0.037 (7)	0.023 (6)
C455	0.057 (9)	0.041 (8)	0.047 (10)	0.009 (6)	0.032 (7)	0.003 (7)
C456	0.020 (7)	0.047 (10)	0.043 (10)	−0.009 (7)	0.012 (6)	0.002 (8)

Geometric parameters (Å, º)

C1—O1	1.218 (5)	C313—H313	0.9300
C1—C2	1.470 (5)	C314—C315	1.367 (7)
C1—C11	1.494 (5)	C314—H314	0.9300
C2—C3	1.329 (5)	C315—C316	1.382 (6)
C2—H2	0.9300	C315—H315	0.9300
C3—C34	1.445 (5)	C316—H316	0.9300
C3—H3	0.9300	C331—H33A	0.9600
C11—C16	1.378 (6)	C331—H33B	0.9600
C11—C12	1.385 (5)	C331—H33C	0.9600
C12—C13	1.372 (6)	O351—C351	1.405 (7)
C12—H12	0.9300	C351—C352	1.377 (7)
C13—C14	1.365 (6)	C351—C356	1.385 (11)
C13—H13	0.9300	C352—C353	1.377 (8)
C14—C15	1.372 (6)	C352—Cl52	1.721 (7)
C14—N14	1.431 (6)	C353—C354	1.395 (13)
C15—C16	1.375 (6)	C353—H353	0.9300

C15—H15	0.9300	C354—C355	1.374 (8)
C16—H16	0.9300	C354—Cl54	1.748 (6)
N14—N15	1.233 (7)	C355—C356	1.375 (8)
N15—N16	1.128 (7)	C355—H355	0.9300
N31—C35	1.356 (5)	C356—H356	0.9300
N31—N32	1.378 (4)	C451—C452	1.378 (8)
N31—C311	1.425 (5)	C451—C456	1.387 (12)
N32—C33	1.322 (5)	C452—C453	1.381 (8)
C33—C34	1.417 (5)	C452—Cl62	1.723 (7)
C33—C331	1.488 (6)	C453—C454	1.394 (15)
C34—C35	1.369 (5)	C453—H453	0.9300
C35—O351	1.366 (4)	C454—C455	1.376 (9)
C311—C312	1.381 (6)	C454—Cl64	1.746 (8)
C311—C316	1.382 (6)	C455—C456	1.373 (8)
C312—C313	1.378 (6)	C455—H455	0.9300
C312—H312	0.9300	C456—H456	0.9300
C313—C314	1.374 (7)		
O1—C1—C2	121.1 (4)	C315—C314—C313	120.3 (5)
O1—C1—C11	119.8 (4)	C315—C314—H314	119.8
C2—C1—C11	119.1 (4)	C313—C314—H314	119.8
C3—C2—C1	121.9 (4)	C314—C315—C316	120.1 (5)
C3—C2—H2	119.0	C314—C315—H315	119.9
C1—C2—H2	119.0	C316—C315—H315	119.9
C2—C3—C34	128.0 (4)	C315—C316—C311	119.4 (5)
C2—C3—H3	116.0	C315—C316—H316	120.3
C34—C3—H3	116.0	C311—C316—H316	120.3
C16—C11—C12	117.7 (4)	C33—C331—H33A	109.5
C16—C11—C1	123.5 (4)	C33—C331—H33B	109.5
C12—C11—C1	118.8 (4)	H33A—C331—H33B	109.5
C13—C12—C11	121.2 (4)	C33—C331—H33C	109.5
C13—C12—H12	119.4	H33A—C331—H33C	109.5
C11—C12—H12	119.4	H33B—C331—H33C	109.5
C14—C13—C12	119.8 (4)	C35—O351—C351	117.0 (5)
C14—C13—H13	120.1	C352—C351—C356	119.5 (7)
C12—C13—H13	120.1	C352—C351—O351	116.5 (6)
C13—C14—C15	120.5 (4)	C356—C351—O351	122.0 (9)
C13—C14—N14	115.9 (4)	C351—C352—C353	121.1 (6)
C15—C14—N14	123.6 (5)	C351—C352—Cl52	119.6 (6)
C14—C15—C16	119.2 (4)	C353—C352—Cl52	119.3 (6)
C14—C15—H15	120.4	C352—C353—C354	117.9 (8)
C16—C15—H15	120.4	C352—C353—H353	121.1
C15—C16—C11	121.6 (4)	C354—C353—H353	121.1
C15—C16—H16	119.2	C355—C354—C353	121.0 (8)
C11—C16—H16	119.2	C355—C354—Cl54	120.5 (6)
N15—N14—C14	115.8 (5)	C353—C354—Cl54	116.5 (8)
N16—N15—N14	172.3 (6)	C354—C355—C356	119.5 (7)
C35—N31—N32	110.0 (3)	C354—C355—H355	120.3

C35—N31—C311	130.9 (3)	C356—C355—H355	120.3
N32—N31—C311	119.1 (3)	C355—C356—C351	119.9 (8)
C33—N32—N31	105.3 (3)	C355—C356—H356	120.0
N32—C33—C34	112.0 (4)	C351—C356—H356	120.0
N32—C33—C331	119.4 (4)	C452—C451—C456	119.5 (9)
C34—C33—C331	128.5 (4)	C451—C452—C453	120.6 (8)
C35—C34—C33	103.7 (3)	C451—C452—Cl62	120.6 (7)
C35—C34—C3	129.5 (3)	C453—C452—Cl62	118.6 (7)
C33—C34—C3	126.8 (4)	C452—C453—C454	117.7 (11)
N31—C35—O351	120.3 (3)	C452—C453—H453	121.1
N31—C35—C34	108.9 (3)	C454—C453—H453	121.1
O351—C35—C34	130.8 (3)	C455—C454—C453	121.0 (10)
C312—C311—C316	120.5 (4)	C455—C454—Cl64	119.3 (9)
C312—C311—N31	121.4 (4)	C453—C454—Cl64	116.8 (11)
C316—C311—N31	118.1 (4)	C456—C455—C454	119.2 (8)
C313—C312—C311	119.2 (4)	C456—C455—H455	120.4
C313—C312—H312	120.4	C454—C455—H455	120.4
C311—C312—H312	120.4	C455—C456—C451	120.0 (9)
C314—C313—C312	120.4 (5)	C455—C456—H456	120.0
C314—C313—H313	119.8	C451—C456—H456	120.0
C312—C313—H313	119.8		
O1—C1—C2—C3	−8.0 (6)	N32—N31—C311—C312	149.6 (4)
C11—C1—C2—C3	171.4 (4)	C35—N31—C311—C316	151.0 (4)
C1—C2—C3—C34	−178.2 (4)	N32—N31—C311—C316	−28.4 (5)
O1—C1—C11—C16	−173.2 (4)	C316—C311—C312—C313	−0.9 (6)
C2—C1—C11—C16	7.4 (6)	N31—C311—C312—C313	−178.8 (4)
O1—C1—C11—C12	6.7 (6)	C311—C312—C313—C314	1.5 (7)
C2—C1—C11—C12	−172.7 (4)	C312—C313—C314—C315	−0.4 (8)
C16—C11—C12—C13	−0.5 (7)	C313—C314—C315—C316	−1.3 (8)
C1—C11—C12—C13	179.6 (4)	C314—C315—C316—C311	1.9 (7)
C11—C12—C13—C14	0.2 (8)	C312—C311—C316—C315	−0.8 (7)
C12—C13—C14—C15	0.2 (8)	N31—C311—C316—C315	177.2 (4)
C12—C13—C14—N14	−179.7 (5)	N31—C35—O351—C351	−102.1 (15)
C13—C14—C15—C16	−0.3 (8)	C34—C35—O351—C351	78.3 (16)
N14—C14—C15—C16	179.6 (5)	C35—O351—C351—C352	−164 (2)
C14—C15—C16—C11	0.0 (7)	C35—O351—C351—C356	0 (4)
C12—C11—C16—C15	0.4 (7)	C356—C351—C352—C353	8 (5)
C1—C11—C16—C15	−179.7 (4)	O351—C351—C352—C353	172 (3)
C13—C14—N14—N15	−172.6 (5)	C356—C351—C352—Cl52	−169 (3)
C15—C14—N14—N15	7.5 (8)	O351—C351—C352—Cl52	−4 (4)
C35—N31—N32—C33	−0.3 (4)	C351—C352—C353—C354	−9 (5)
C311—N31—N32—C33	179.3 (4)	Cl52—C352—C353—C354	168 (3)
N31—N32—C33—C34	0.6 (5)	C352—C353—C354—C355	10 (5)
N31—N32—C33—C331	179.2 (4)	C352—C353—C354—Cl54	174 (3)
N32—C33—C34—C35	−0.6 (5)	C353—C354—C355—C356	−9 (5)
C331—C33—C34—C35	−179.1 (5)	Cl54—C354—C355—C356	−173 (3)
N32—C33—C34—C3	180.0 (4)	C354—C355—C356—C351	8 (5)

C331—C33—C34—C3	1.5 (7)	C352—C351—C356—C355	−8 (5)
C2—C3—C34—C35	5.1 (7)	O351—C351—C356—C355	−171 (3)
C2—C3—C34—C33	−175.7 (4)	C456—C451—C452—C453	−11 (6)
N32—N31—C35—O351	−179.8 (3)	C456—C451—C452—Cl62	164 (3)
C311—N31—C35—O351	0.7 (6)	C451—C452—C453—C454	13 (6)
N32—N31—C35—C34	−0.1 (5)	Cl62—C452—C453—C454	−163 (4)
C311—N31—C35—C34	−179.6 (4)	C452—C453—C454—C455	−13 (6)
C33—C34—C35—N31	0.4 (4)	C452—C453—C454—Cl64	−173 (3)
C3—C34—C35—N31	179.8 (4)	C453—C454—C455—C456	11 (6)
C33—C34—C35—O351	−179.9 (4)	Cl64—C454—C455—C456	171 (3)
C3—C34—C35—O351	−0.5 (7)	C454—C455—C456—C451	−10 (6)
C35—N31—C311—C312	−31.0 (6)	C452—C451—C456—C455	10 (6)

Hydrogen-bond geometry (Å, º)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C356—H356⋯O1[i]	0.93	2.32	3.115 (18)	143
C456—H456⋯O1[i]	0.93	2.47	3.21 (2)	137

Symmetry code: (i) −x+1, −y+2, −z+1.

1-(4-Azidophenyl)-3-[3-methyl-5-(naphthalen-2-yloxy)-1-phenyl-1*H*-pyrazol-4-yl]prop-2-en-1-on (IIe)

Crystal data

$C_{29}H_{21}N_5O_2$	$F(000) = 984$
$M_r = 471.51$	$D_x = 1.324$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 9.8460$ (8) Å	Cell parameters from 4197 reflections
$b = 22.4303$ (18) Å	$\theta = 3.1$–25.1°
$c = 11.0490$ (9) Å	$\mu = 0.09$ mm^{-1}
$\beta = 104.157$ (2)°	$T = 297$ K
$V = 2366.0$ (3) Å^3	Block, orange
$Z = 4$	0.22 × 0.21 × 0.16 mm

Data collection

Bruker APEXII diffractometer	43217 measured reflections
Radiation source: fine focussealed tube	4196 independent reflections
Graphite monochromator	2463 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.092$
Absorption correction: multi-scan (SADABS; Bruker, 2016)	$\theta_{max} = 25.1°$, $\theta_{min} = 3.1°$
$T_{min} = 0.930$, $T_{max} = 0.986$	$h = -11 \rightarrow 11$
	$k = -26 \rightarrow 26$
	$l = -12 \rightarrow 13$

Refinement

Refinement on F^2	Primary atom site location: dual
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.063$	H-atom parameters constrained
$wR(F^2) = 0.154$	$w = 1/[\sigma^2(F_o^2) + (0.0268P)^2 + 3.4386P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	
4196 reflections	$(\Delta/\sigma)_{max} < 0.001$
327 parameters	$\Delta\rho_{max} = 0.23$ e Å^{-3}
0 restraints	

$\Delta\rho_{min} = -0.27$ e Å^{-3}

Extinction correction: SHELXL,
Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Extinction coefficient: 0.0143 (9)

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	0.3799 (4)	0.50783 (16)	0.2247 (3)	0.0478 (9)
O1	0.2646 (3)	0.49934 (13)	0.1526 (2)	0.0673 (8)
C2	0.5060 (4)	0.47779 (16)	0.2070 (3)	0.0489 (9)
H2	0.5904	0.4830	0.2662	0.059*
C3	0.5026 (4)	0.44316 (15)	0.1078 (3)	0.0465 (9)
H3	0.4164	0.4406	0.0502	0.056*
C11	0.3908 (3)	0.54796 (15)	0.3332 (3)	0.0433 (8)
C12	0.2693 (4)	0.56999 (17)	0.3595 (4)	0.0543 (10)
H12	0.1831	0.5588	0.3088	0.065*
C13	0.2725 (4)	0.60784 (17)	0.4580 (4)	0.0552 (10)
H13	0.1896	0.6212	0.4748	0.066*
C14	0.3990 (4)	0.62554 (16)	0.5309 (3)	0.0486 (9)
C15	0.5220 (4)	0.6051 (2)	0.5074 (4)	0.0656 (12)
H15	0.6078	0.6174	0.5572	0.079*
C16	0.5169 (4)	0.56643 (19)	0.4095 (4)	0.0595 (11)
H16	0.6001	0.5524	0.3944	0.071*
N14	0.4149 (4)	0.66594 (16)	0.6335 (3)	0.0655 (10)
N15	0.3037 (4)	0.68821 (16)	0.6481 (3)	0.0621 (9)
N16	0.2126 (4)	0.71190 (18)	0.6714 (4)	0.0877 (13)
N31	0.8165 (3)	0.36482 (13)	0.0799 (3)	0.0495 (8)
N32	0.7208 (3)	0.33982 (14)	−0.0191 (3)	0.0551 (8)
C33	0.6012 (4)	0.36732 (16)	−0.0196 (3)	0.0487 (9)
C34	0.6151 (4)	0.40921 (15)	0.0783 (3)	0.0444 (9)
C35	0.7544 (4)	0.40565 (15)	0.1390 (3)	0.0446 (9)
C311	0.9557 (4)	0.34178 (17)	0.1152 (3)	0.0491 (9)
C312	1.0685 (4)	0.37933 (18)	0.1521 (3)	0.0530 (10)
H312	1.0555	0.4204	0.1536	0.064*
C313	1.2010 (4)	0.3553 (2)	0.1870 (4)	0.0634 (11)
H313	1.2778	0.3805	0.2116	0.076*
C314	1.2215 (5)	0.2949 (2)	0.1861 (4)	0.0762 (13)
H314	1.3115	0.2792	0.2102	0.091*
C315	1.1087 (5)	0.2577 (2)	0.1496 (5)	0.0831 (15)
H315	1.1227	0.2167	0.1496	0.100*
C316	0.9736 (4)	0.28021 (19)	0.1125 (4)	0.0704 (13)
H316	0.8971	0.2549	0.0866	0.084*
C331	0.4703 (4)	0.35114 (19)	−0.1150 (4)	0.0665 (12)

H33A	0.4924	0.3233	−0.1734	0.100*
H33B	0.4298	0.3864	−0.1585	0.100*
H33C	0.4047	0.3333	−0.0743	0.100*
O351	0.8283 (2)	0.42967 (10)	0.2482 (2)	0.0485 (6)
C351	0.8655 (3)	0.51795 (16)	0.3666 (3)	0.0455 (9)
H351	0.8589	0.4953	0.4355	0.055*
C352	0.8523 (3)	0.49169 (15)	0.2527 (3)	0.0423 (8)
C353	0.8651 (4)	0.52368 (17)	0.1473 (3)	0.0498 (9)
H353	0.8558	0.5046	0.0709	0.060*
C354	0.8917 (4)	0.58332 (18)	0.1585 (3)	0.0551 (10)
H354	0.9021	0.6047	0.0893	0.066*
C355	0.9276 (4)	0.67581 (18)	0.2850 (4)	0.0664 (12)
H355	0.9410	0.6977	0.2175	0.080*
C356	0.9309 (5)	0.7037 (2)	0.3951 (5)	0.0732 (13)
H356	0.9446	0.7447	0.4017	0.088*
C357	0.9139 (4)	0.6714 (2)	0.4975 (4)	0.0685 (12)
H357	0.9152	0.6913	0.5716	0.082*
C358	0.8951 (4)	0.61082 (19)	0.4919 (4)	0.0570 (10)
H358	0.8862	0.5897	0.5620	0.068*
C359	0.9037 (4)	0.61317 (16)	0.2727 (3)	0.0498 (9)
C360	0.8896 (3)	0.58065 (16)	0.3779 (3)	0.0437 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.042 (2)	0.052 (2)	0.048 (2)	0.0016 (18)	0.0093 (17)	0.0008 (18)
O1	0.0452 (16)	0.089 (2)	0.0630 (17)	0.0038 (15)	0.0042 (14)	−0.0216 (16)
C2	0.037 (2)	0.058 (2)	0.050 (2)	0.0027 (17)	0.0077 (16)	−0.0056 (19)
C3	0.041 (2)	0.051 (2)	0.047 (2)	0.0013 (17)	0.0080 (16)	0.0021 (18)
C11	0.0349 (19)	0.048 (2)	0.046 (2)	0.0009 (16)	0.0098 (16)	−0.0019 (17)
C12	0.036 (2)	0.064 (3)	0.062 (2)	−0.0024 (18)	0.0092 (18)	−0.011 (2)
C13	0.042 (2)	0.063 (3)	0.061 (2)	−0.0017 (19)	0.0130 (18)	−0.014 (2)
C14	0.048 (2)	0.051 (2)	0.045 (2)	−0.0011 (18)	0.0082 (17)	−0.0036 (18)
C15	0.037 (2)	0.093 (3)	0.061 (3)	0.002 (2)	0.0013 (19)	−0.019 (2)
C16	0.039 (2)	0.082 (3)	0.056 (2)	0.007 (2)	0.0096 (18)	−0.013 (2)
N14	0.054 (2)	0.078 (2)	0.064 (2)	0.0027 (19)	0.0129 (17)	−0.0171 (19)
N15	0.066 (2)	0.058 (2)	0.061 (2)	−0.0092 (19)	0.0140 (19)	−0.0108 (18)
N16	0.076 (3)	0.085 (3)	0.107 (3)	0.002 (2)	0.032 (2)	−0.036 (2)
N31	0.0454 (18)	0.0521 (19)	0.0493 (18)	0.0013 (15)	0.0080 (15)	−0.0150 (15)
N32	0.0519 (19)	0.057 (2)	0.0527 (19)	−0.0057 (16)	0.0061 (15)	−0.0154 (16)
C33	0.048 (2)	0.049 (2)	0.047 (2)	−0.0067 (18)	0.0065 (17)	−0.0048 (18)
C34	0.045 (2)	0.045 (2)	0.042 (2)	−0.0008 (17)	0.0099 (16)	−0.0052 (17)
C35	0.046 (2)	0.044 (2)	0.042 (2)	−0.0012 (17)	0.0079 (17)	−0.0064 (17)
C311	0.046 (2)	0.056 (2)	0.047 (2)	0.0025 (19)	0.0137 (17)	−0.0074 (19)
C312	0.050 (2)	0.056 (2)	0.053 (2)	0.000 (2)	0.0148 (18)	−0.0016 (19)
C313	0.048 (2)	0.084 (3)	0.058 (3)	0.001 (2)	0.0133 (19)	−0.006 (2)
C314	0.054 (3)	0.089 (4)	0.086 (3)	0.017 (3)	0.018 (2)	−0.013 (3)
C315	0.072 (3)	0.069 (3)	0.109 (4)	0.019 (3)	0.024 (3)	−0.021 (3)

C316	0.062 (3)	0.059 (3)	0.093 (3)	0.003 (2)	0.023 (2)	−0.023 (2)
C331	0.060 (3)	0.068 (3)	0.066 (3)	−0.009 (2)	0.004 (2)	−0.014 (2)
O351	0.0554 (15)	0.0442 (14)	0.0417 (14)	−0.0010 (12)	0.0042 (11)	−0.0085 (12)
C351	0.040 (2)	0.053 (2)	0.040 (2)	−0.0020 (17)	0.0057 (16)	−0.0060 (18)
C352	0.0357 (19)	0.043 (2)	0.044 (2)	−0.0003 (16)	0.0026 (15)	−0.0079 (18)
C353	0.052 (2)	0.056 (3)	0.040 (2)	0.0036 (18)	0.0078 (17)	−0.0031 (18)
C354	0.058 (2)	0.060 (3)	0.045 (2)	0.005 (2)	0.0081 (18)	0.005 (2)
C355	0.070 (3)	0.053 (3)	0.071 (3)	0.002 (2)	0.007 (2)	−0.001 (2)
C356	0.074 (3)	0.055 (3)	0.083 (3)	0.006 (2)	0.005 (3)	−0.014 (3)
C357	0.056 (3)	0.072 (3)	0.072 (3)	0.000 (2)	0.007 (2)	−0.029 (3)
C358	0.050 (2)	0.070 (3)	0.049 (2)	−0.003 (2)	0.0093 (18)	−0.018 (2)
C359	0.045 (2)	0.052 (2)	0.047 (2)	0.0021 (18)	0.0028 (17)	−0.0025 (19)
C360	0.0306 (18)	0.055 (2)	0.042 (2)	0.0002 (16)	0.0034 (15)	−0.0121 (18)

Geometric parameters (Å, º)

C1—O1	1.232 (4)	C312—H312	0.9300
C1—C2	1.467 (5)	C313—C314	1.371 (6)
C1—C11	1.482 (5)	C313—H313	0.9300
C2—C3	1.337 (5)	C314—C315	1.368 (6)
C2—H2	0.9300	C314—H314	0.9300
C3—C34	1.446 (5)	C315—C316	1.387 (6)
C3—H3	0.9300	C315—H315	0.9300
C11—C16	1.382 (5)	C316—H316	0.9300
C11—C12	1.389 (5)	C331—H33A	0.9600
C12—C13	1.375 (5)	C331—H33B	0.9600
C12—H12	0.9300	C331—H33C	0.9600
C13—C14	1.367 (5)	O351—C352	1.410 (4)
C13—H13	0.9300	C351—C352	1.367 (5)
C14—C15	1.377 (5)	C351—C360	1.427 (5)
C14—N14	1.430 (5)	C351—H351	0.9300
C15—C16	1.378 (5)	C352—C353	1.400 (5)
C15—H15	0.9300	C353—C354	1.363 (5)
C16—H16	0.9300	C353—H353	0.9300
N14—N15	1.249 (5)	C354—C359	1.407 (5)
N15—N16	1.126 (5)	C354—H354	0.9300
N31—C35	1.353 (4)	C355—C356	1.360 (6)
N31—N32	1.376 (4)	C355—C359	1.426 (5)
N31—C311	1.427 (4)	C355—H355	0.9300
N32—C33	1.327 (4)	C356—C357	1.388 (6)
C33—C34	1.414 (5)	C356—H356	0.9300
C33—C331	1.497 (5)	C357—C358	1.371 (6)
C34—C35	1.374 (5)	C357—H357	0.9300
C35—O351	1.359 (4)	C358—C360	1.419 (5)
C311—C312	1.374 (5)	C358—H358	0.9300
C311—C316	1.394 (5)	C359—C360	1.408 (5)
C312—C313	1.377 (5)		

O1—C1—C2	121.3 (3)	C314—C313—H313	119.5
O1—C1—C11	119.3 (3)	C312—C313—H313	119.5
C2—C1—C11	119.4 (3)	C315—C314—C313	119.6 (4)
C3—C2—C1	121.6 (3)	C315—C314—H314	120.2
C3—C2—H2	119.2	C313—C314—H314	120.2
C1—C2—H2	119.2	C314—C315—C316	121.0 (4)
C2—C3—C34	128.6 (3)	C314—C315—H315	119.5
C2—C3—H3	115.7	C316—C315—H315	119.5
C34—C3—H3	115.7	C315—C316—C311	118.2 (4)
C16—C11—C12	117.2 (3)	C315—C316—H316	120.9
C16—C11—C1	123.5 (3)	C311—C316—H316	120.9
C12—C11—C1	119.3 (3)	C33—C331—H33A	109.5
C13—C12—C11	122.1 (3)	C33—C331—H33B	109.5
C13—C12—H12	119.0	H33A—C331—H33B	109.5
C11—C12—H12	119.0	C33—C331—H33C	109.5
C14—C13—C12	119.2 (3)	H33A—C331—H33C	109.5
C14—C13—H13	120.4	H33B—C331—H33C	109.5
C12—C13—H13	120.4	C35—O351—C352	118.1 (3)
C13—C14—C15	120.6 (4)	C352—C351—C360	118.8 (3)
C13—C14—N14	124.0 (3)	C352—C351—H351	120.6
C15—C14—N14	115.5 (3)	C360—C351—H351	120.6
C14—C15—C16	119.5 (3)	C351—C352—C353	122.5 (3)
C14—C15—H15	120.2	C351—C352—O351	115.8 (3)
C16—C15—H15	120.2	C353—C352—O351	121.7 (3)
C15—C16—C11	121.5 (3)	C354—C353—C352	118.8 (3)
C15—C16—H16	119.3	C354—C353—H353	120.6
C11—C16—H16	119.3	C352—C353—H353	120.6
N15—N14—C14	115.2 (3)	C353—C354—C359	121.4 (4)
N16—N15—N14	172.4 (4)	C353—C354—H354	119.3
C35—N31—N32	110.9 (3)	C359—C354—H354	119.3
C35—N31—C311	129.0 (3)	C356—C355—C359	120.4 (4)
N32—N31—C311	119.6 (3)	C356—C355—H355	119.8
C33—N32—N31	104.3 (3)	C359—C355—H355	119.8
N32—C33—C34	112.7 (3)	C355—C356—C357	120.5 (4)
N32—C33—C331	120.0 (3)	C355—C356—H356	119.7
C34—C33—C331	127.2 (3)	C357—C356—H356	119.7
C35—C34—C33	103.4 (3)	C358—C357—C356	121.5 (4)
C35—C34—C3	130.5 (3)	C358—C357—H357	119.3
C33—C34—C3	126.0 (3)	C356—C357—H357	119.3
N31—C35—O351	119.3 (3)	C357—C358—C360	119.2 (4)
N31—C35—C34	108.6 (3)	C357—C358—H358	120.4
O351—C35—C34	131.6 (3)	C360—C358—H358	120.4
C312—C311—C316	121.1 (4)	C354—C359—C360	119.4 (3)
C312—C311—N31	120.8 (3)	C354—C359—C355	122.0 (4)
C316—C311—N31	118.1 (3)	C360—C359—C355	118.6 (4)
C311—C312—C313	119.0 (4)	C359—C360—C358	119.7 (3)
C311—C312—H312	120.5	C359—C360—C351	119.1 (3)
C313—C312—H312	120.5	C358—C360—C351	121.2 (4)

C314—C313—C312	121.1 (4)		

O1—C1—C2—C3	4.4 (6)	C35—N31—C311—C312	−47.2 (5)
C11—C1—C2—C3	−177.2 (3)	N32—N31—C311—C312	140.9 (3)
C1—C2—C3—C34	−178.0 (3)	C35—N31—C311—C316	132.0 (4)
O1—C1—C11—C16	−171.1 (4)	N32—N31—C311—C316	−39.9 (5)
C2—C1—C11—C16	10.4 (5)	C316—C311—C312—C313	0.0 (6)
O1—C1—C11—C12	7.1 (5)	N31—C311—C312—C313	179.2 (3)
C2—C1—C11—C12	−171.4 (3)	C311—C312—C313—C314	−0.5 (6)
C16—C11—C12—C13	−1.0 (6)	C312—C313—C314—C315	0.3 (7)
C1—C11—C12—C13	−179.3 (4)	C313—C314—C315—C316	0.4 (7)
C11—C12—C13—C14	1.5 (6)	C314—C315—C316—C311	−0.9 (7)
C12—C13—C14—C15	−0.9 (6)	C312—C311—C316—C315	0.7 (6)
C12—C13—C14—N14	178.3 (4)	N31—C311—C316—C315	−178.5 (4)
C13—C14—C15—C16	−0.1 (6)	N31—C35—O351—C352	118.1 (3)
N14—C14—C15—C16	−179.4 (4)	C34—C35—O351—C352	−71.1 (5)
C14—C15—C16—C11	0.6 (7)	C360—C351—C352—C353	1.7 (5)
C12—C11—C16—C15	−0.1 (6)	C360—C351—C352—O351	−179.8 (3)
C1—C11—C16—C15	178.2 (4)	C35—O351—C352—C351	150.9 (3)
C13—C14—N14—N15	−5.0 (6)	C35—O351—C352—C353	−30.7 (4)
C15—C14—N14—N15	174.2 (4)	C351—C352—C353—C354	−0.1 (5)
C35—N31—N32—C33	1.2 (4)	O351—C352—C353—C354	−178.4 (3)
C311—N31—N32—C33	174.5 (3)	C352—C353—C354—C359	−1.2 (5)
N31—N32—C33—C34	−1.1 (4)	C359—C355—C356—C357	1.3 (6)
N31—N32—C33—C331	−179.2 (3)	C355—C356—C357—C358	0.8 (7)
N32—C33—C34—C35	0.5 (4)	C356—C357—C358—C360	−1.6 (6)
C331—C33—C34—C35	178.6 (4)	C353—C354—C359—C360	0.7 (5)
N32—C33—C34—C3	−175.5 (3)	C353—C354—C359—C355	−178.2 (4)
C331—C33—C34—C3	2.5 (6)	C356—C355—C359—C354	176.3 (4)
C2—C3—C34—C35	−3.7 (7)	C356—C355—C359—C360	−2.6 (6)
C2—C3—C34—C33	171.2 (4)	C354—C359—C360—C358	−177.1 (3)
N32—N31—C35—O351	171.8 (3)	C355—C359—C360—C358	1.8 (5)
C311—N31—C35—O351	−0.6 (6)	C354—C359—C360—C351	0.9 (5)
N32—N31—C35—C34	−0.9 (4)	C355—C359—C360—C351	179.9 (3)
C311—N31—C35—C34	−173.4 (3)	C357—C358—C360—C359	0.2 (5)
C33—C34—C35—N31	0.2 (4)	C357—C358—C360—C351	−177.8 (3)
C3—C34—C35—N31	176.0 (3)	C352—C351—C360—C359	−2.1 (5)
C33—C34—C35—O351	−171.3 (4)	C352—C351—C360—C358	175.9 (3)
C3—C34—C35—O351	4.4 (7)		

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
C353—H353···O1[i]	0.93	2.47	3.288 (4)	147
C12—H12···*Cg*3[ii]	0.93	2.93	3.761 (4)	150
C13—H13···*Cg*4[ii]	0.93	2.73	3.547 (4)	148

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