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Different molecular conformations co-exist in each of three 2-aryl-*N*-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)acetamides: hydrogen bonding in zero, one and two dimensions

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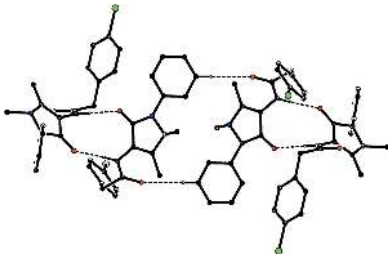
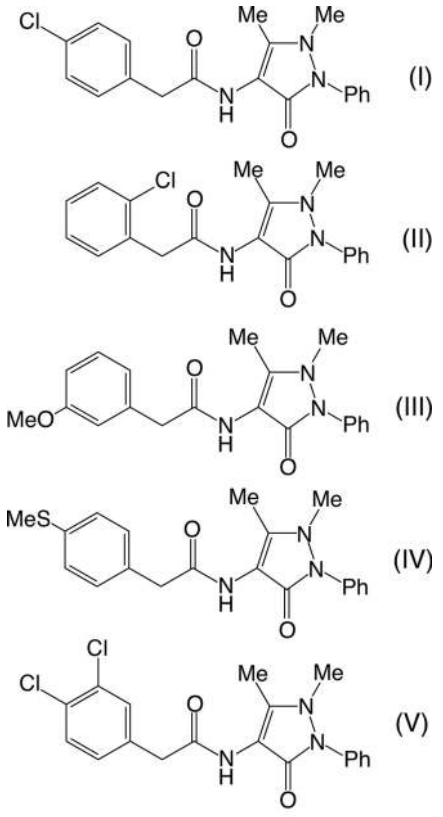
4-Antipyrine [4-amino-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one] and its derivatives exhibit a range of biological activities, including analgesic, antibacterial and anti-inflammatory, and new examples are always of potential interest and value. 2-(4-Chlorophenyl)-*N*-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)acetamide, $C_{19}H_{18}ClN_3O_2$, (I), crystallizes with $Z' = 2$ in the space group $P\overline{1}$, whereas its positional isomer 2-(2-chlorophenyl)-*N*-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)acetamide, (II), crystallizes with $Z' = 1$ in the space group $C2/c$; the molecules of (II) are disordered over two sets of atomic sites having occupancies of 0.6020 (18) and 0.3980 (18). The two independent molecules of (I) adopt different molecular conformations, as do the two disorder components in (II), where the 2-chlorophenyl substituents adopt different orientations. The molecules of (I) are linked by a combination of N—H···O and C—H···O hydrogen bonds to form centrosymmetric four-molecule aggregates, while those of (II) are linked by the same types of hydrogen bonds forming sheets. The related compound *N*-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)-2-(3-methoxyphenyl)acetamide, $C_{20}H_{21}N_3O_3$, (III), is isomorphous with (I) but not strictly isostructural; again the two independent molecules adopt different molecular conformations, and the molecules are linked by N—H···O and C—H···O hydrogen bonds to form ribbons. Comparisons are made with some related structures, indicating that a hydrogen-bonded $R_2^2(10)$ ring is the common structural motif.

1. Introduction

4-Amino-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one, also called 4-aminoantipyrine, and it derivatives exhibit a range of biological activities, including analgesic (Cechinel Filho *et al.*, 1998; Sondhi *et al.*, 1999), antibacterial (Sutcliffe, 2003) and anti-inflammatory (Sondhi *et al.*, 1999). Accordingly, new examples are always of potential interest and value, and we report here the synthesis and the molecular and supramolecular structures of two new derivatives of this class, namely 2-(4-chlorophenyl)-*N*-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)acetamide, (I), and 2-(2-chlorophenyl)-*N*-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)acetamide, (II). We also discuss the recently deposited structure (Cambridge Structural Database; Groom *et al.*, 2016) of the closely related compound 2-(3-methoxyphenyl)-*N*-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)acetamide, (III) (see Scheme 1) (Narayana *et al.*, 2016). The compounds were prepared by the reactions between equi-



molar quantities of 4-aminoantipyrine and the appropriately substituted phenylacetic acid, using 1-ethyl-3-[3-(dimethylamino)propyl]carbodiimide hydrochloride as the coupling reagent.



Scheme 1

2. Experimental

2.1. Synthesis and crystallization

For the synthesis of compounds (I) and (II), equimolar quantities (1 mmol of each component) of 4-aminoantipyrine and the appropriately substituted phenylacetic acid were dissolved in dichloromethane (20 ml) together with 3-[3-(dimethylamino)propyl]-1-ethylcarbodiimide hydrochloride (0.01 mol) as the coupling agent and triethylamine (0.2 mol). The mixtures were stirred at 273 K for 3 h, and then poured with stirring into ice-cold aqueous hydrochloric acid (4 mol dm^{-3}, 100 ml). The aqueous mixtures were extracted exhaustively with dichloromethane and the combined extracts were in each case washed with saturated aqueous sodium hydrogen carbonate solution and then with brine. The solutions were dried over anhydrous sodium sulfate and the solvent was removed under reduced pressure to give compounds (I) and (II) in yields of 75–80%. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation, at ambient temperature and in the presence of air, of solutions in dichloromethane.

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. All H atoms were located in difference maps. H atoms bonded to C atoms were then treated as riding atoms in geometrically idealized positions, with C—H = 0.93 (aromatic), 0.96 (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 bonded to C atoms. For H atoms bonded to N atoms, the atomic coordinates were refined with $U_{iso}(H) = 1.2U_{eq}(N)$, giving the N—H distances shown in Table 3. For compound (II), it was obvious from an early stage that the Cl substituent was disordered over two atomic sites having unequal occupancies and it soon became clear that, in fact, the entire molecule was disordered over two sets of atomic sites having unequal occupancies. For the minor component, the bond lengths and the one-angle nonbonded distances, apart from those involving the Cl atom, were restrained to be the same as the corresponding distances in the major component, subject to s.u. values of 0.005 and 0.01 Å, respectively, and the two independent C—Cl distances were restrained to values of 1.725 (10) Å; in addition, the anisotropic displacement parameters for corresponding pairs of atoms occupying essentially the same physical space were constrained to be identical. Subject to these conditions, the refined occupancies for the two disorder components were 0.6020 (18) and 0.3980 (18).

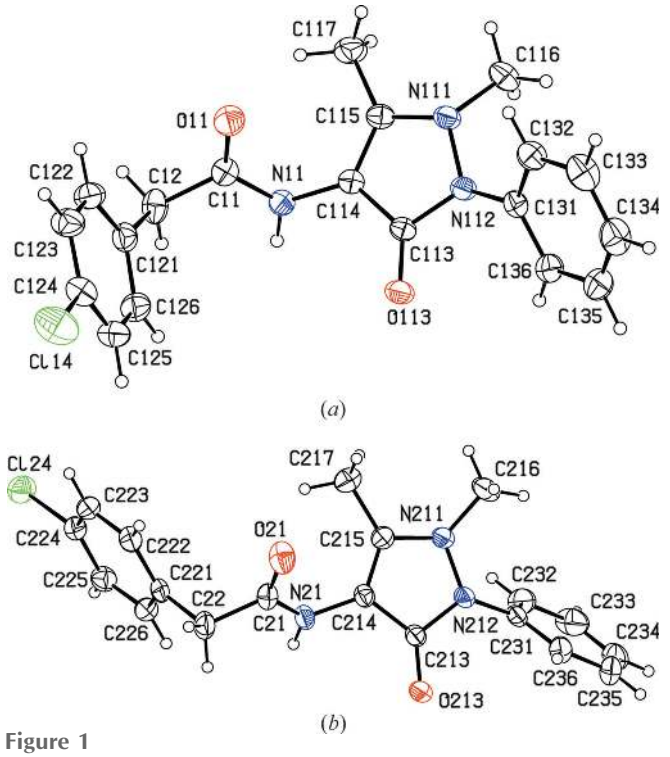


Figure 1
The structures of the two independent molecules in the selected asymmetric unit of compound (I), showing the atom-labelling scheme: (*a*) the type 1 molecule and (*b*) the type 2 molecule. Displacement ellipsoids are drawn at the 30% probability level.

3. Results and discussion

Compounds (I) and (II) are positional isomers, but despite their close relationship, their crystallization characteristics are markedly different. Compound (I) (Fig. 1) crystallizes in the

Table 1
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_{19}H_{18}ClN_3O_2$	$C_{19}H_{18}ClN_3O_2$
M_r	355.81	355.81
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $C2/c$
Temperature (K)	295	295
a, b, c (Å)	10.1018 (4), 10.6099 (5), 18.8129 (11)	23.023 (3), 8.2976 (10), 21.602 (3)
α, β, γ (°)	100.292 (3), 91.881 (3), 116.873 (2)	90, 120.957 (14), 90
V (Å^3)	1754.78 (15)	3538.9 (9)
Z	4	8
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.24	0.23
Crystal size (mm)	0.30 × 0.20 × 0.20	0.40 × 0.30 × 0.20
Data collection		
Diffractometer	Bruker APEXII area-detector	Bruker APEXII area-detector
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 2003)	Multi-scan (*SADABS*; Sheldrick, 2003)
T_{min}, T_{max}	0.788, 0.954	0.767, 0.954
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	32307, 7205, 5041	33715, 4079, 3038
R_{int}	0.031	0.034
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.629	0.651
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.046, 0.133, 1.03	0.051, 0.149, 1.03
No. of reflections	7205	4079
No. of parameters	461	319
No. of restraints	0	64
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.58, −0.63	0.30, −0.41

Computer programs: *APEX2* (Bruker, 2012), *SAINT-Plus* (Bruker, 2012), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

triclinic space group $P\overline{1}$, with $Z' = 2$, and it will be convenient to refer to the molecules of compound (I) containing atoms N11 and N21 as molecules of types 1 and 2, respectively. By contrast, compound (II), which crystallizes in the monoclinic space group $C2/c$, exhibits conformational disorder in which the entire molecule is disordered over two sets of atomic sites having occupancies of 0.6020 (18) and 0.3980 (18), and in which the chlorophenyl ring adopts different orientations in the two disorder components, so that these are, in fact, conformational isomers (Fig. 2).

Compound (III) (Narayana *et al.*, 2016) also crystallizes in the space group $P\overline{1}$, with $Z' = 2$, and its unit-cell dimensions [a = 10.1227 (4), b = 10.6675 (4), c = 19.1679 (10) Å, α = 96.254 (3), β = 93.636 (3) and γ = 118.055 (2)°] are very similar to those of (I), with the corresponding pairs of cell-repeat distances all within 1.5% of one another, and an average difference between the corresponding pairs of inter-axial angles of *ca* 2.3°. In addition, the atomic coordinates for corresponding pairs of atoms in (I) and (III) are very similar. However, while compounds (I) and (III) are thus isomorphous, they cannot be strictly isostructural as (I) contains a 4-chlorophenyl substituent, whereas (III) contains a 3-methoxyphenyl substituent.

In each compound, the reference molecules were selected to have the same sign for the torsion angle Cx13—Cx14—Nx1—Cx1, where x = 1 or 2 (Table 2). On this basis, it is possible to select for (I) an asymmetric unit in which the two independent molecules are linked by two N—H···O hydrogen bonds (Table 3) to form a cyclic dimer. None of the molecules in (I)–(III) exhibits any internal symmetry, so that all are conformationally chiral; the centrosymmetric space groups confirm that they have all crystallized as conformational racemates.

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

For compounds (I) and (III) (Narayana *et al.*, 2016), the indices x (1 or 2) refer to the two independent molecules in the selected asymmetric units; for compound (II), these indices refer to the major and minor disorder components (*cf.* Figs. 1 and 2).

Parameter	(I)		(II)		(III)	
	x = 1	x = 2	x = 1	x = 2	x = 1	x = 2
Cx13—Cx14—Nx1—Cx1	122.2 (2)	129.1 (2)	102.8 (15)	128 (2)	121.0 (2)	136.5 (3)
Cx14—Nx1—Cx1—Cx2	−174.36 (18)	177.29 (19)	−175.3 (8)	162.3 (13)	−170.6 (2)	172.7 (2)
Nx1—Cx1—Cx2—Cx21	114.3 (2)	−89.3 (3)	−154.8 (9)	167.12 (14)	122.2 (2)	−88.9 (3)
Cx1—Cx2—Cx21—Cx22	80.8 (3)	−82.5 (3)	67.3 (7)	96.8 (11)	−84.8 (3)	−84.7 (3)
Nx11—Nx12—Cx31—Cx32	22.6 (3)	−43.9 (3)	22 (2)	31 (3)	26.7 (3)	−34.7 (3)

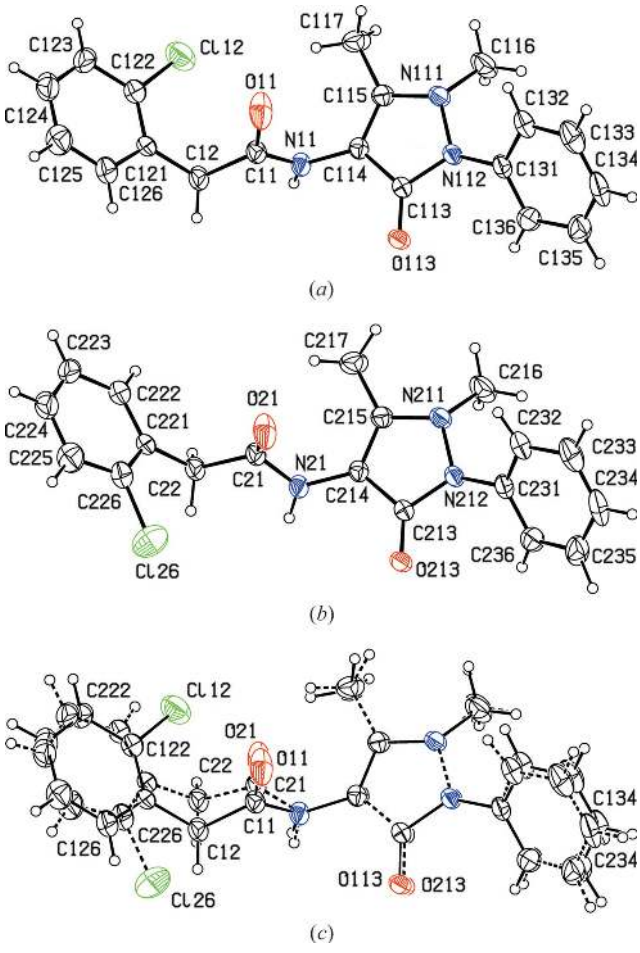


Figure 2
The molecular structures of the disordered conformers in compound (II), showing the atom-labelling scheme: (*a*) the major conformer, (*b*) the minor conformer and (*c*) the two conformers together. Displacement ellipsoids are drawn at the 30% probability level and, for the sake of clarity, many of the atom labels have been omitted from part (*c*).

The two independent molecules in the structure of compound (I) adopt different conformations (Fig. 1) as indicated by the different values of the pairs of torsion angles around the bonds C*x*1—C*x*2, C*x*2—C*x*21 and N*x*12—C*x*31 (Table 2). The two disorder components of compound (II) likewise adopt different conformations (Fig. 2), as indicated not only by the different locations of the chloro substituents in

Table 3
Hydrogen bonds parameters (Å, °) for compounds (I) and (II).

	D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
(I)	N11—H11···O213	0.87 (3)	1.93 (2)	2.781 (2)	169 (2)
	N21—H21···O113	0.84 (2)	2.01 (2)	2.845 (2)	178 (4)
	C133—H133···O11^{i}	0.93	2.52	3.446 (3)	171
(II)	N11—H11···O113^{ii}	0.82 (7)	2.15 (8)	2.94 (2)	162 (6)
	N11—H11···O213^{ii}	0.82 (7)	2.08 (8)	2.88 (2)	168 (6)
	N21—H21···O113^{ii}	0.92 (10)	2.00 (10)	2.91 (2)	161 (7)
	N21—H21···O213^{ii}	0.92 (10)	1.95 (10)	2.82 (3)	158 (7)
	C134—H134···O11^{iii}	0.93	2.57	3.46 (4)	159
	C234—H234···O21^{iii}	0.93	2.49	3.40 (7)	164
	C225—H225···O213^{iv}	0.93	2.39	3.28 (2)	160

Symmetry codes: (i) $-x, -y, -z+1$; (ii) $-x+1, y, -z+\frac{1}{2}$; (iii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (iv) $-x+1, y-1, -z+\frac{1}{2}$.

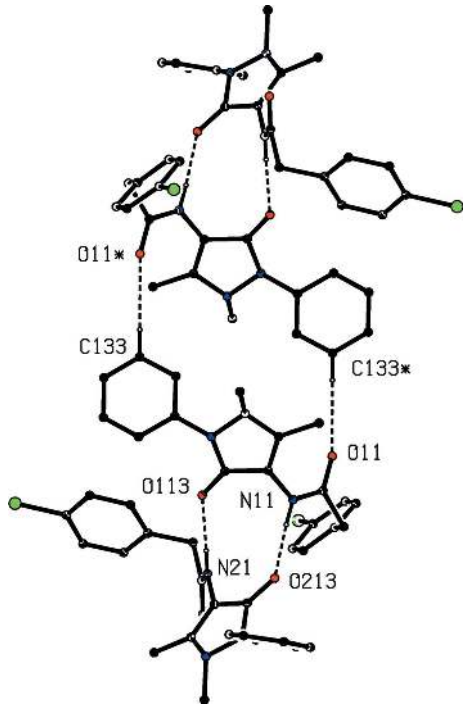


Figure 3
Part of the crystal structure of compound (I), showing the formation of a centrosymmetric four-molecule aggregate built from N—H···O and C—H···O hydrogen bonds, shown as dashed lines. For the sake of clarity, the unit-cell outline and H atoms not involved in the motifs shown have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(-x, -y, -z+1)$.

the two forms, but by the differences in the key pairs of torsion angles exactly as for (I). The same holds for compound (III) so that in each of the structures of (I)–(III) two different conformations co-exist in the same crystal. The related compound (IV) (see Scheme 1) crystallizes with $Z' = 1$ (Fun *et al.*, 2012), but the dichloro derivative (V) crystallizes in the space group $P2_1/c$, with $Z' = 3$ (Mahan *et al.*, 2013); the three independent molecules in (V) show major differences in the torsion angles around the bonds corresponding to C*x*1—C*x*2 and C*x*2—C*x*21 in (I)–(III), such that three very different molecular conformations, all of them chiral, co-exist in crystals of (V).

As noted above, the two molecules in the selected asymmetric unit of compound (I) are linked by N—H···O hydrogen bonds to form a dimeric unit; inversion-related pairs of these units are further linked by C—H···O hydrogen bonds (Table 3) to form a four-molecule aggregate in which a central centrosymmetric $R_2^2(20)$ (Bernstein *et al.*, 1995) ring is flanked by two inversion-related $R_2^2(10)$ rings (Fig. 3). There are no direction-specific interactions between adjacent hydrogen-bonded tetramers, so that the supramolecular aggregation is finite and can thus be regarded as zero-dimensional.

In compound (II), the major and minor conformers exhibit comparable patterns of N—H···O hydrogen bonds (Table 3), in which pairs of molecules related by the twofold rotation axis along $(\frac{1}{2}, y, \frac{1}{4})$ are linked to form an $R_2^2(10)$ dimer (Fig. 4) analogous to that formed in compound (I). However, for such a dimeric unit in which both component molecules are the

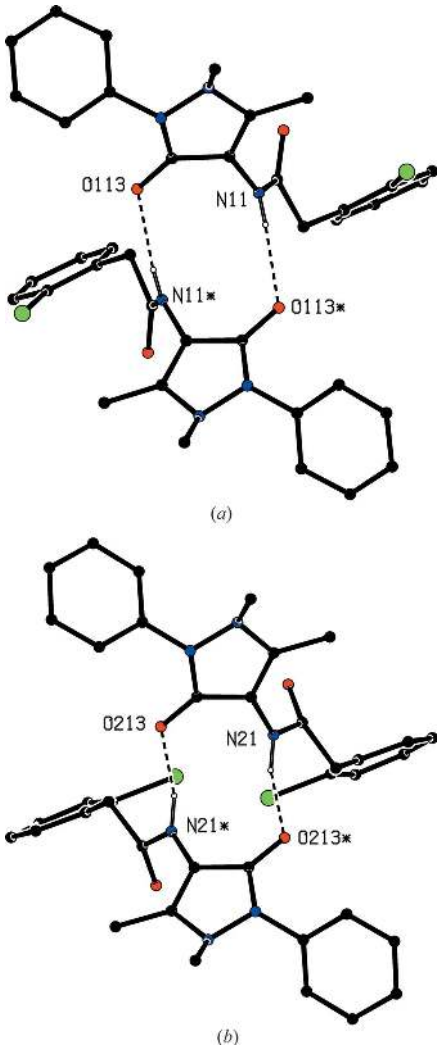


Figure 4
Part of the crystal structure of compound (II), showing the formation of cyclic hydrogen-bonded dimers containing rotation-related pairs of (*a*) the major conformer and (*b*) the minor conformer. Hydrogen bonds are shown as dashed lines and, for the sake of clarity, the unit-cell outline and H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) are at the symmetry position ($-x+1$, y, $-z+\frac{1}{2}$).

minor conformer, the Cl···Cl distance within the dimer would be 2.437 (4) Å, well below the van der Waals contact distance of 3.48 Å (Rowland & Taylor, 1996), suggesting that all such dimeric aggregates consist either of two molecules of the major conformer or of one molecule each of the major and minor forms. The reference dimer lies across the twofold rotation axis along ($\frac{1}{2}$, y, $\frac{1}{4}$) and is linked directly by C—H···O hydrogen bonds to four other dimers, which lie across the rotation axes along (0, y, $\frac{1}{4}$) and (1, y, $\frac{1}{4}$), but displaced from the reference dimer by $\pm\frac{1}{2}y$, so forming a sheet lying parallel to (001) (Fig. 5).

The two independent molecules in the selected asymmetric unit of compound (III) (Narayana *et al.*, 2016) are linked by two N—H···O hydrogen bonds to form an $R_2^2(10)$ dimer, exactly as for compound (I). However, in the structure of (III), these dimers are linked by two C—H···O hydrogen bonds which generate a ribbon running parallel to the [100] direction

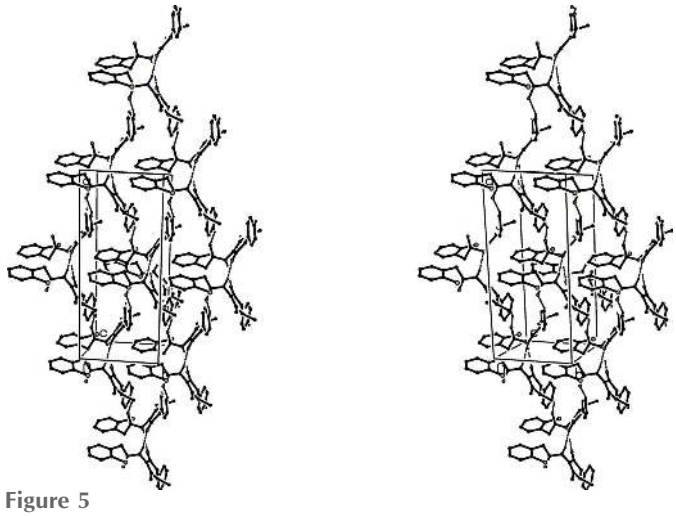


Figure 5
A stereoview of part of the crystal structure of compound (II), showing the formation of a hydrogen-bonded sheet lying parallel to (001). Hydrogen bonds are shown as dashed lines and, for the sake of clarity, only the major conformer is shown and the H atoms not involved in the motifs shown have been omitted.

and in which centrosymmetric $R_2^2(20)$ and $R_4^4(28)$ rings alternate along the centre of the ribbon with $R_2^2(10)$ rings along the edges (Fig. 6). The supramolecular aggregation in compounds (I)–(III) is thus zero-, two- and one-dimensional, respectively.

Inversion-related pairs of molecules in compound (IV) (Fun *et al.*, 2012) are linked by N—H···O hydrogen bonds to form centrosymmetric $R_2^2(10)$ dimers which are further linked by a single C—H···O hydrogen bond to form sheets. Of the three independent molecules in the structure of compound (V) (Mahan *et al.*, 2013), two of them are linked by two independent N—H···O hydrogen bonds to form a cyclic $R_2^2(10)$ dimer as in (I), while inversion-related pairs of the third type of molecule form centrosymmetric dimers as in (IV). Three independent C—H···O hydrogen bonds link the

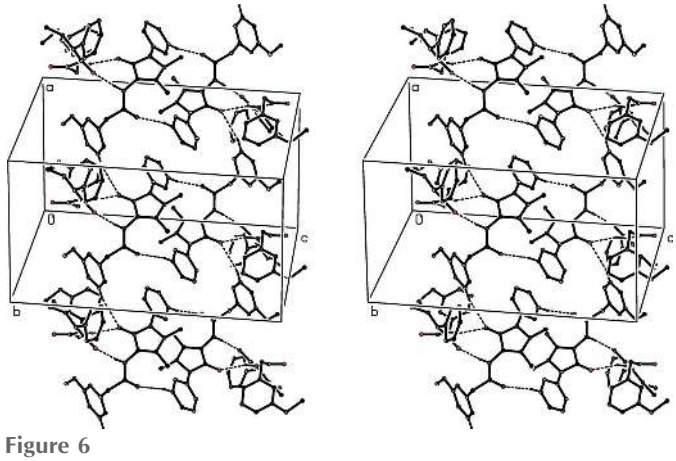


Figure 6
A stereoview of part of the crystal structure of compound (III), showing the formation of a hydrogen-bonded ribbon parallel to [100] containing alternating $R_2^2(20)$ and $R_4^4(28)$ rings along the centre of the ribbon with $R_2^2(10)$ rings along the edges. The original atomic coordinates (Narayana *et al.*, 2016) have been used and hydrogen bonds are shown as dashed lines. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

two types of dimer into chains. The common structural motif in compounds (I)–(V) is the formation of $R_2^2(10)$ dimers and a similar dimeric motif is present in the structure of 2-[(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)amino]-1-methyl-2-oxoethyl pyrrolidine-1-carbodithioate (Akkurt *et al.*, 2010).

The simple precursor compound 4-aminoantipyrine crystallizes in the chiral space group $P6_1/P6_5$ and structures of both enantiomers have been reported (Li *et al.*, 2013; Mnguni & Lemmerer, 2015); a single N—H···O hydrogen bond links the molecules into simple *C*(5) chains.

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supporting information

Acta Cryst. (2016). C**72**, 664-669 [doi:10.1107/S2053229616012870]

Different molecular conformations co-exist in each of three 2-aryl-*N*-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)acetamides: hydrogen bonding in zero, one and two dimensions

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Computing details

For both compounds, data collection: *APEX2* (Bruker, 2012); cell refinement: *APEX2* (Bruker, 2012); data reduction: *SAINT-Plus* (Bruker, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

(I) 2-(4-Chlorophenyl)-*N*-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)acetamide

Crystal data

$C_{19}H_{18}ClN_3O_2$
M_r = 355.81
Triclinic, $P\overline{1}$
a = 10.1018 (4) Å
b = 10.6099 (5) Å
c = 18.8129 (11) Å
α = 100.292 (3)°
β = 91.881 (3)°
γ = 116.873 (2)°
V = 1754.78 (15) Å^3
Z = 4
$F(000)$ = 744
D_x = 1.347 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8317 reflections
θ = 1.1–28.2°
μ = 0.24 mm^{-1}
T = 295 K
Block, colourless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
T_{min} = 0.788, T_{max} = 0.954
32307 measured reflections
7205 independent reflections
5041 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 26.6°, θ_{min} = 2.2°
h = −12→12
k = −13→13
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.133
S = 1.03
7205 reflections
461 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0535P)^2 + 0.8541P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.58$ e Å^{-3}
$\Delta\rho_{min} = -0.63$ e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C11	0.0605 (2)	0.4111 (2)	0.35897 (11)	0.0430 (5)
O11	−0.02706 (17)	0.35666 (18)	0.40064 (9)	0.0587 (4)
N11	0.19775 (19)	0.41816 (17)	0.36076 (9)	0.0393 (4)
H11	0.255 (3)	0.453 (2)	0.3284 (12)	0.047*
C12	0.0218 (3)	0.4743 (2)	0.30052 (14)	0.0545 (6)
H12A	−0.0071	0.5466	0.3224	0.065*
H12B	0.1085	0.5208	0.2760	0.065*
N111	0.2896 (2)	0.27333 (18)	0.50227 (8)	0.0426 (4)
N112	0.33015 (19)	0.20766 (17)	0.44120 (8)	0.0403 (4)
C113	0.2960 (2)	0.2495 (2)	0.37988 (10)	0.0370 (4)
O113	0.30568 (19)	0.19812 (16)	0.31741 (7)	0.0511 (4)
C114	0.2477 (2)	0.35432 (19)	0.40692 (10)	0.0366 (4)
C115	0.2510 (2)	0.3693 (2)	0.48018 (11)	0.0415 (5)
C116	0.3858 (3)	0.3110 (3)	0.57066 (11)	0.0549 (6)
H11A	0.4836	0.3865	0.5682	0.082*
H11B	0.3933	0.2273	0.5784	0.082*
H11C	0.3435	0.3439	0.6103	0.082*
C117	0.2218 (3)	0.4710 (3)	0.53415 (13)	0.0609 (6)
H11D	0.3150	0.5477	0.5600	0.091*
H11E	0.1611	0.4202	0.5680	0.091*
H11F	0.1703	0.5110	0.5094	0.091*
C121	−0.1055 (2)	0.3547 (2)	0.24629 (12)	0.0478 (5)
C122	−0.2525 (3)	0.3030 (3)	0.26009 (13)	0.0568 (6)
H122	−0.2746	0.3487	0.3015	0.068*
C123	−0.3667 (3)	0.1853 (3)	0.21375 (13)	0.0623 (7)
H123	−0.4650	0.1514	0.2238	0.075*
C124	−0.3343 (3)	0.1186 (3)	0.15289 (13)	0.0574 (6)
Cl14	−0.47626 (10)	−0.03248 (9)	0.09513 (4)	0.0943 (3)
C125	−0.1915 (3)	0.1699 (3)	0.13668 (13)	0.0636 (7)
H125	−0.1710	0.1255	0.0943	0.076*
C126	−0.0771 (3)	0.2878 (3)	0.18325 (14)	0.0594 (6)
H126	0.0204	0.3226	0.1720	0.071*
C131	0.3196 (2)	0.0688 (2)	0.43913 (11)	0.0399 (4)
C132	0.2258 (3)	−0.0209 (2)	0.48042 (12)	0.0517 (5)
H132	0.1688	0.0088	0.5101	0.062*
C133	0.2177 (3)	−0.1559 (3)	0.47704 (15)	0.0657 (7)

H133	0.1571	−0.2162	0.5057	0.079*
C134	0.2982 (3)	−0.2006 (3)	0.43200 (16)	0.0695 (7)
H134	0.2911	−0.2918	0.4294	0.083*
C135	0.3892 (3)	−0.1120 (3)	0.39061 (15)	0.0683 (7)
H135	0.4426	−0.1439	0.3594	0.082*
C136	0.4026 (3)	0.0239 (3)	0.39467 (13)	0.0542 (6)
H136	0.4673	0.0851	0.3676	0.065*
C21	0.1899 (2)	0.1687 (2)	0.12171 (13)	0.0476 (5)
O21	0.1859 (2)	0.19074 (18)	0.06084 (9)	0.0670 (5)
N21	0.2985 (2)	0.26017 (18)	0.17678 (10)	0.0444 (4)
H21	0.299 (3)	0.242 (3)	0.2182 (13)	0.053*
C22	0.0736 (3)	0.0327 (2)	0.14195 (15)	0.0583 (6)
H22A	0.0615	0.0549	0.1929	0.070*
H22B	−0.0218	0.0004	0.1131	0.070*
N211	0.6348 (2)	0.54849 (19)	0.14139 (10)	0.0515 (5)
N212	0.59086 (19)	0.62246 (18)	0.19668 (9)	0.0443 (4)
C213	0.4620 (2)	0.5246 (2)	0.21926 (10)	0.0391 (4)
O213	0.40726 (17)	0.55872 (16)	0.27238 (8)	0.0527 (4)
C214	0.4214 (2)	0.3899 (2)	0.17045 (10)	0.0391 (4)
C215	0.5246 (2)	0.4090 (2)	0.12366 (11)	0.0458 (5)
C216	0.7181 (4)	0.6308 (3)	0.08971 (15)	0.0876 (11)
H21A	0.6540	0.6531	0.0613	0.131*
H21B	0.7538	0.5746	0.0580	0.131*
H21C	0.8016	0.7190	0.1156	0.131*
C217	0.5321 (3)	0.3049 (3)	0.06221 (14)	0.0651 (7)
H21D	0.6330	0.3190	0.0628	0.098*
H21E	0.5010	0.3200	0.0171	0.098*
H21F	0.4671	0.2079	0.0668	0.098*
C221	0.1166 (2)	−0.0881 (2)	0.12951 (12)	0.0450 (5)
C222	0.0882 (2)	−0.1749 (2)	0.06067 (12)	0.0485 (5)
H222	0.0452	−0.1566	0.0219	0.058*
C223	0.1228 (2)	−0.2881 (2)	0.04885 (12)	0.0492 (5)
H223	0.1042	−0.3452	0.0023	0.059*
C224	0.1847 (3)	−0.3156 (2)	0.10616 (12)	0.0468 (5)
Cl24	0.22648 (8)	−0.45950 (7)	0.09162 (4)	0.0675 (2)
C225	0.2144 (3)	−0.2317 (3)	0.17520 (12)	0.0540 (6)
H225	0.2562	−0.2515	0.2138	0.065*
C226	0.1811 (3)	−0.1174 (2)	0.18619 (12)	0.0523 (6)
H226	0.2024	−0.0591	0.2326	0.063*
C231	0.7044 (2)	0.7494 (2)	0.24460 (11)	0.0444 (5)
C232	0.8319 (3)	0.7503 (3)	0.27219 (13)	0.0601 (6)
H232	0.8475	0.6699	0.2587	0.072*
C233	0.9370 (3)	0.8729 (4)	0.32045 (15)	0.0794 (9)
H233	1.0248	0.8756	0.3392	0.095*
C234	0.9129 (3)	0.9896 (3)	0.34061 (15)	0.0795 (10)
H234	0.9834	1.0708	0.3739	0.095*
C235	0.7866 (4)	0.9887 (3)	0.31254 (16)	0.0749 (8)
H235	0.7714	1.0693	0.3264	0.090*

C236	0.6803 (3)	0.8674 (2)	0.26328 (13)	0.0583 (6)
H236	0.5942	0.8663	0.2433	0.070*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0410 (11)	0.0343 (10)	0.0495 (12)	0.0144 (9)	0.0053 (9)	0.0084 (9)
O11	0.0444 (9)	0.0690 (10)	0.0657 (10)	0.0240 (8)	0.0183 (8)	0.0263 (8)
N11	0.0394 (9)	0.0358 (9)	0.0434 (9)	0.0155 (7)	0.0108 (7)	0.0151 (7)
C12	0.0465 (13)	0.0462 (12)	0.0732 (16)	0.0207 (11)	0.0031 (11)	0.0221 (11)
N111	0.0517 (10)	0.0448 (9)	0.0316 (8)	0.0232 (8)	0.0060 (7)	0.0067 (7)
N112	0.0505 (10)	0.0397 (9)	0.0339 (8)	0.0230 (8)	0.0086 (7)	0.0096 (7)
C113	0.0387 (10)	0.0339 (10)	0.0354 (10)	0.0130 (8)	0.0076 (8)	0.0104 (8)
O113	0.0757 (11)	0.0528 (9)	0.0352 (8)	0.0376 (8)	0.0142 (7)	0.0118 (6)
C114	0.0363 (10)	0.0320 (9)	0.0371 (10)	0.0119 (8)	0.0060 (8)	0.0078 (8)
C115	0.0443 (11)	0.0365 (10)	0.0384 (11)	0.0152 (9)	0.0066 (9)	0.0050 (8)
C116	0.0614 (14)	0.0546 (13)	0.0390 (11)	0.0204 (12)	−0.0035 (10)	0.0077 (10)
C117	0.0813 (18)	0.0579 (14)	0.0473 (13)	0.0388 (14)	0.0115 (12)	0.0020 (11)
C121	0.0488 (12)	0.0500 (12)	0.0521 (13)	0.0263 (11)	0.0080 (10)	0.0195 (10)
C122	0.0519 (14)	0.0696 (15)	0.0480 (13)	0.0304 (12)	0.0094 (11)	0.0044 (11)
C123	0.0472 (13)	0.0791 (17)	0.0519 (14)	0.0234 (13)	0.0087 (11)	0.0099 (12)
C124	0.0618 (15)	0.0602 (14)	0.0460 (13)	0.0248 (12)	0.0046 (11)	0.0124 (11)
Cl14	0.1013 (6)	0.0752 (5)	0.0688 (5)	0.0169 (4)	−0.0090 (4)	−0.0033 (4)
C125	0.0801 (18)	0.0754 (17)	0.0455 (13)	0.0448 (15)	0.0169 (13)	0.0119 (12)
C126	0.0518 (14)	0.0755 (17)	0.0633 (15)	0.0341 (13)	0.0235 (12)	0.0294 (13)
C131	0.0437 (11)	0.0366 (10)	0.0385 (10)	0.0176 (9)	0.0006 (8)	0.0096 (8)
C132	0.0524 (13)	0.0483 (12)	0.0517 (13)	0.0192 (11)	0.0081 (10)	0.0161 (10)
C133	0.0688 (17)	0.0428 (13)	0.0724 (17)	0.0116 (12)	0.0027 (13)	0.0229 (12)
C134	0.089 (2)	0.0420 (13)	0.0759 (18)	0.0314 (14)	−0.0039 (15)	0.0108 (13)
C135	0.091 (2)	0.0697 (17)	0.0638 (16)	0.0554 (16)	0.0091 (14)	0.0109 (13)
C136	0.0644 (15)	0.0563 (13)	0.0541 (13)	0.0356 (12)	0.0143 (11)	0.0192 (11)
C21	0.0433 (12)	0.0365 (11)	0.0562 (14)	0.0136 (9)	0.0022 (10)	0.0088 (10)
O21	0.0644 (11)	0.0591 (10)	0.0564 (10)	0.0123 (9)	−0.0152 (8)	0.0126 (8)
N21	0.0484 (10)	0.0349 (9)	0.0395 (9)	0.0092 (8)	0.0036 (8)	0.0117 (7)
C22	0.0435 (12)	0.0376 (11)	0.0855 (17)	0.0127 (10)	0.0146 (12)	0.0092 (11)
N211	0.0470 (10)	0.0460 (10)	0.0429 (10)	0.0079 (8)	0.0151 (8)	0.0025 (8)
N212	0.0386 (9)	0.0396 (9)	0.0375 (9)	0.0049 (7)	0.0092 (7)	0.0033 (7)
C213	0.0380 (11)	0.0379 (10)	0.0354 (10)	0.0116 (9)	0.0054 (8)	0.0104 (8)
O213	0.0491 (9)	0.0472 (8)	0.0456 (8)	0.0094 (7)	0.0168 (7)	0.0055 (7)
C214	0.0398 (11)	0.0362 (10)	0.0361 (10)	0.0127 (9)	0.0027 (8)	0.0104 (8)
C215	0.0469 (12)	0.0441 (11)	0.0394 (11)	0.0168 (10)	0.0038 (9)	0.0050 (9)
C216	0.086 (2)	0.0715 (18)	0.0609 (16)	−0.0004 (16)	0.0385 (15)	0.0074 (14)
C217	0.0706 (17)	0.0587 (15)	0.0564 (15)	0.0265 (13)	0.0134 (12)	−0.0007 (12)
C221	0.0347 (10)	0.0320 (10)	0.0564 (13)	0.0050 (8)	0.0115 (9)	0.0096 (9)
C222	0.0465 (12)	0.0457 (12)	0.0472 (12)	0.0155 (10)	0.0029 (9)	0.0131 (10)
C223	0.0520 (13)	0.0463 (12)	0.0408 (11)	0.0178 (10)	0.0053 (10)	0.0037 (9)
C224	0.0511 (13)	0.0412 (11)	0.0464 (12)	0.0192 (10)	0.0087 (10)	0.0115 (9)
Cl24	0.0870 (5)	0.0629 (4)	0.0687 (4)	0.0460 (4)	0.0189 (3)	0.0202 (3)

C225	0.0606 (14)	0.0558 (14)	0.0426 (12)	0.0232 (12)	0.0056 (10)	0.0148 (10)
C226	0.0554 (13)	0.0452 (12)	0.0440 (12)	0.0151 (11)	0.0084 (10)	0.0034 (9)
C231	0.0395 (11)	0.0389 (11)	0.0371 (10)	0.0030 (9)	0.0103 (9)	0.0079 (8)
C232	0.0476 (14)	0.0649 (15)	0.0559 (14)	0.0200 (12)	0.0045 (11)	0.0023 (12)
C233	0.0410 (14)	0.100 (2)	0.0605 (16)	0.0094 (15)	0.0014 (12)	−0.0040 (16)
C234	0.0570 (17)	0.0640 (18)	0.0588 (16)	−0.0143 (14)	0.0115 (13)	−0.0083 (13)
C235	0.087 (2)	0.0383 (13)	0.0740 (18)	0.0097 (14)	0.0189 (16)	0.0042 (12)
C236	0.0616 (15)	0.0444 (13)	0.0578 (14)	0.0148 (11)	0.0101 (11)	0.0122 (11)

Geometric parameters (Å, º)

C11—O11	1.215 (2)	C21—O21	1.212 (3)
C11—N11	1.353 (3)	C21—N21	1.350 (3)
C11—C12	1.511 (3)	C21—C22	1.517 (3)
N11—C114	1.399 (3)	N21—C214	1.405 (2)
N11—H11	0.87 (2)	N21—H21	0.83 (2)
C12—C121	1.510 (3)	C22—C221	1.512 (3)
C12—H12A	0.9700	C22—H22A	0.9700
C12—H12B	0.9700	C22—H22B	0.9700
N111—C115	1.364 (3)	N211—C215	1.360 (3)
N111—N112	1.406 (2)	N211—N212	1.396 (2)
N111—C116	1.463 (3)	N211—C216	1.452 (3)
N112—C113	1.394 (2)	N212—C213	1.387 (2)
N112—C131	1.422 (3)	N212—C231	1.430 (2)
C113—O113	1.231 (2)	C213—O213	1.235 (2)
C113—C114	1.428 (3)	C213—C214	1.425 (3)
C114—C115	1.356 (3)	C214—C215	1.355 (3)
C115—C117	1.483 (3)	C215—C217	1.479 (3)
C116—H11A	0.9600	C216—H21A	0.9600
C116—H11B	0.9600	C216—H21B	0.9600
C116—H11C	0.9600	C216—H21C	0.9600
C117—H11D	0.9600	C217—H21D	0.9600
C117—H11E	0.9600	C217—H21E	0.9600
C117—H11F	0.9600	C217—H21F	0.9600
C121—C126	1.378 (3)	C221—C226	1.383 (3)
C121—C122	1.383 (3)	C221—C222	1.385 (3)
C122—C123	1.376 (3)	C222—C223	1.380 (3)
C122—H122	0.9300	C222—H222	0.9300
C123—C124	1.365 (3)	C223—C224	1.368 (3)
C123—H123	0.9300	C223—H223	0.9300
C124—C125	1.360 (4)	C224—C225	1.374 (3)
C124—Cl14	1.733 (3)	C224—Cl24	1.740 (2)
C125—C126	1.380 (4)	C225—C226	1.382 (3)
C125—H125	0.9300	C225—H225	0.9300
C126—H126	0.9300	C226—H226	0.9300
C131—C136	1.376 (3)	C231—C232	1.368 (3)
C131—C132	1.380 (3)	C231—C236	1.369 (3)
C132—C133	1.387 (3)	C232—C233	1.382 (4)

C132—H132	0.9300	C232—H232	0.9300
C133—C134	1.361 (4)	C233—C234	1.359 (5)
C133—H133	0.9300	C233—H233	0.9300
C134—C135	1.365 (4)	C234—C235	1.361 (5)
C134—H134	0.9300	C234—H234	0.9300
C135—C136	1.373 (3)	C235—C236	1.388 (4)
C135—H135	0.9300	C235—H235	0.9300
C136—H136	0.9300	C236—H236	0.9300
O11—C11—N11	122.9 (2)	O21—C21—N21	123.5 (2)
O11—C11—C12	121.8 (2)	O21—C21—C22	122.3 (2)
N11—C11—C12	115.31 (18)	N21—C21—C22	114.1 (2)
C11—N11—C114	123.23 (17)	C21—N21—C214	124.54 (18)
C11—N11—H11	120.1 (15)	C21—N21—H21	121.6 (16)
C114—N11—H11	116.2 (15)	C214—N21—H21	113.9 (16)
C121—C12—C11	109.27 (17)	C221—C22—C21	112.18 (18)
C121—C12—H12A	109.8	C221—C22—H22A	109.2
C11—C12—H12A	109.8	C21—C22—H22A	109.2
C121—C12—H12B	109.8	C221—C22—H22B	109.2
C11—C12—H12B	109.8	C21—C22—H22B	109.2
H12A—C12—H12B	108.3	H22A—C22—H22B	107.9
C115—N111—N112	106.81 (15)	C215—N211—N212	107.21 (16)
C115—N111—C116	121.99 (17)	C215—N211—C216	125.02 (19)
N112—N111—C116	114.89 (18)	N212—N211—C216	116.1 (2)
C113—N112—N111	108.88 (16)	C213—N212—N211	109.36 (15)
C113—N112—C131	123.24 (16)	C213—N212—C231	123.33 (16)
N111—N112—C131	118.59 (15)	N211—N212—C231	118.30 (16)
O113—C113—N112	123.48 (18)	O213—C213—N212	122.90 (18)
O113—C113—C114	131.26 (18)	O213—C213—C214	132.45 (18)
N112—C113—C114	105.24 (16)	N212—C213—C214	104.62 (17)
C115—C114—N11	129.18 (19)	C215—C214—N21	128.16 (19)
C115—C114—C113	108.43 (18)	C215—C214—C213	109.06 (17)
N11—C114—C113	122.26 (17)	N21—C214—C213	122.61 (18)
C114—C115—N111	110.00 (17)	C214—C215—N211	109.27 (18)
C114—C115—C117	129.6 (2)	C214—C215—C217	130.3 (2)
N111—C115—C117	120.36 (19)	N211—C215—C217	120.4 (2)
N111—C116—H11A	109.5	N211—C216—H21A	109.5
N111—C116—H11B	109.5	N211—C216—H21B	109.5
H11A—C116—H11B	109.5	H21A—C216—H21B	109.5
N111—C116—H11C	109.5	N211—C216—H21C	109.5
H11A—C116—H11C	109.5	H21A—C216—H21C	109.5
H11B—C116—H11C	109.5	H21B—C216—H21C	109.5
C115—C117—H11D	109.5	C215—C217—H21D	109.5
C115—C117—H11E	109.5	C215—C217—H21E	109.5
H11D—C117—H11E	109.5	H21D—C217—H21E	109.5
C115—C117—H11F	109.5	C215—C217—H21F	109.5
H11D—C117—H11F	109.5	H21D—C217—H21F	109.5
H11E—C117—H11F	109.5	H21E—C217—H21F	109.5

C126—C121—C122	118.0 (2)	C226—C221—C222	118.4 (2)
C126—C121—C12	120.4 (2)	C226—C221—C22	121.4 (2)
C122—C121—C12	121.5 (2)	C222—C221—C22	120.2 (2)
C123—C122—C121	121.3 (2)	C223—C222—C221	120.9 (2)
C123—C122—H122	119.4	C223—C222—H222	119.5
C121—C122—H122	119.4	C221—C222—H222	119.5
C124—C123—C122	119.3 (2)	C224—C223—C222	119.4 (2)
C124—C123—H123	120.3	C224—C223—H223	120.3
C122—C123—H123	120.3	C222—C223—H223	120.3
C125—C124—C123	120.7 (2)	C223—C224—C225	121.3 (2)
C125—C124—Cl14	119.4 (2)	C223—C224—Cl24	119.50 (17)
C123—C124—Cl14	119.9 (2)	C225—C224—Cl24	119.24 (18)
C124—C125—C126	119.9 (2)	C224—C225—C226	118.8 (2)
C124—C125—H125	120.1	C224—C225—H225	120.6
C126—C125—H125	120.1	C226—C225—H225	120.6
C121—C126—C125	120.8 (2)	C225—C226—C221	121.3 (2)
C121—C126—H126	119.6	C225—C226—H226	119.4
C125—C126—H126	119.6	C221—C226—H226	119.4
C136—C131—C132	120.3 (2)	C232—C231—C236	121.3 (2)
C136—C131—N112	119.24 (18)	C232—C231—N212	120.3 (2)
C132—C131—N112	120.46 (19)	C236—C231—N212	118.4 (2)
C131—C132—C133	119.1 (2)	C231—C232—C233	118.8 (3)
C131—C132—H132	120.5	C231—C232—H232	120.6
C133—C132—H132	120.5	C233—C232—H232	120.6
C134—C133—C132	120.3 (2)	C234—C233—C232	120.4 (3)
C134—C133—H133	119.9	C234—C233—H233	119.8
C132—C133—H133	119.9	C232—C233—H233	119.8
C133—C134—C135	120.3 (2)	C233—C234—C235	120.6 (2)
C133—C134—H134	119.9	C233—C234—H234	119.7
C135—C134—H134	119.9	C235—C234—H234	119.7
C134—C135—C136	120.5 (3)	C234—C235—C236	120.0 (3)
C134—C135—H135	119.7	C234—C235—H235	120.0
C136—C135—H135	119.7	C236—C235—H235	120.0
C135—C136—C131	119.5 (2)	C231—C236—C235	118.8 (3)
C135—C136—H136	120.2	C231—C236—H236	120.6
C131—C136—H136	120.2	C235—C236—H236	120.6

O11—C11—N11—C114	4.9 (3)	O21—C21—N21—C214	−1.4 (4)
C12—C11—N11—C114	−174.36 (18)	C22—C21—N21—C214	177.29 (19)
O11—C11—C12—C121	−65.0 (3)	O21—C21—C22—C221	89.4 (3)
N11—C11—C12—C121	114.3 (2)	N21—C21—C22—C221	−89.3 (3)
C115—N111—N112—C113	−8.2 (2)	C215—N211—N212—C213	7.2 (2)
C116—N111—N112—C113	−146.82 (17)	C216—N211—N212—C213	152.4 (2)
C115—N111—N112—C131	−155.98 (17)	C215—N211—N212—C231	155.52 (19)
C116—N111—N112—C131	65.4 (2)	C216—N211—N212—C231	−59.3 (3)
N111—N112—C113—O113	−172.76 (18)	N211—N212—C213—O213	172.7 (2)
C131—N112—C113—O113	−26.8 (3)	C231—N212—C213—O213	26.3 (3)
N111—N112—C113—C114	5.8 (2)	N211—N212—C213—C214	−5.5 (2)

C131—N112—C113—C114	151.75 (18)	C231—N212—C213—C214	−151.9 (2)
C11—N11—C114—C115	−53.2 (3)	C21—N21—C214—C215	−56.2 (3)
C11—N11—C114—C113	122.2 (2)	C21—N21—C214—C213	129.1 (2)
O113—C113—C114—C115	177.1 (2)	O213—C213—C214—C215	−176.1 (2)
N112—C113—C114—C115	−1.3 (2)	N212—C213—C214—C215	1.8 (2)
O113—C113—C114—N11	0.9 (3)	O213—C213—C214—N21	−0.5 (4)
N112—C113—C114—N11	−177.47 (16)	N212—C213—C214—N21	177.38 (18)
N11—C114—C115—N111	171.97 (18)	N21—C214—C215—N211	−172.6 (2)
C113—C114—C115—N111	−3.9 (2)	C213—C214—C215—N211	2.6 (3)
N11—C114—C115—C117	−8.9 (4)	N21—C214—C215—C217	6.2 (4)
C113—C114—C115—C117	175.3 (2)	C213—C214—C215—C217	−178.5 (2)
N112—N111—C115—C114	7.4 (2)	N212—N211—C215—C214	−6.0 (2)
C116—N111—C115—C114	142.4 (2)	C216—N211—C215—C214	−147.2 (3)
N112—N111—C115—C117	−171.84 (19)	N212—N211—C215—C217	175.0 (2)
C116—N111—C115—C117	−36.8 (3)	C216—N211—C215—C217	33.8 (4)
C11—C12—C121—C126	−95.2 (3)	C21—C22—C221—C226	99.2 (3)
C11—C12—C121—C122	80.8 (3)	C21—C22—C221—C222	−82.5 (3)
C126—C121—C122—C123	2.2 (4)	C226—C221—C222—C223	0.2 (3)
C12—C121—C122—C123	−173.9 (2)	C22—C221—C222—C223	−178.14 (19)
C121—C122—C123—C124	−0.2 (4)	C221—C222—C223—C224	0.6 (3)
C122—C123—C124—C125	−1.9 (4)	C222—C223—C224—C225	−0.6 (3)
C122—C123—C124—Cl14	178.4 (2)	C222—C223—C224—Cl24	179.27 (17)
C123—C124—C125—C126	2.0 (4)	C223—C224—C225—C226	−0.2 (3)
Cl14—C124—C125—C126	−178.3 (2)	Cl24—C224—C225—C226	179.87 (17)
C122—C121—C126—C125	−2.1 (4)	C224—C225—C226—C221	1.1 (3)
C12—C121—C126—C125	174.0 (2)	C222—C221—C226—C225	−1.1 (3)
C124—C125—C126—C121	0.0 (4)	C22—C221—C226—C225	177.2 (2)
C113—N112—C131—C136	58.8 (3)	C213—N212—C231—C232	99.7 (3)
N111—N112—C131—C136	−158.29 (19)	N211—N212—C231—C232	−43.9 (3)
C113—N112—C131—C132	−120.3 (2)	C213—N212—C231—C236	−78.7 (3)
N111—N112—C131—C132	22.6 (3)	N211—N212—C231—C236	137.7 (2)
C136—C131—C132—C133	0.7 (3)	C236—C231—C232—C233	0.7 (3)
N112—C131—C132—C133	179.8 (2)	N212—C231—C232—C233	−177.7 (2)
C131—C132—C133—C134	−1.8 (4)	C231—C232—C233—C234	0.7 (4)
C132—C133—C134—C135	1.0 (4)	C232—C233—C234—C235	−1.3 (4)
C133—C134—C135—C136	1.0 (4)	C233—C234—C235—C236	0.5 (4)
C134—C135—C136—C131	−2.1 (4)	C232—C231—C236—C235	−1.5 (3)
C132—C131—C136—C135	1.2 (3)	N212—C231—C236—C235	176.9 (2)
N112—C131—C136—C135	−177.9 (2)	C234—C235—C236—C231	0.9 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H11···O213	0.87 (3)	1.93 (2)	2.781 (2)	169 (2)
N21—H21···O113	0.84 (2)	2.01 (2)	2.845 (2)	178 (4)
C133—H133···O11^{i}	0.93	2.52	3.446 (3)	171

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

(II) 2-(2-Chlorophenyl)-*N*-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)acetamide

Crystal data

$C_{19}H_{18}ClN_3O_2$
$M_r = 355.81$
Monoclinic, $C2/c$
$a = 23.023$ (3) Å
$b = 8.2976$ (10) Å
$c = 21.602$ (3) Å
$\beta = 120.957$ (14)°
$V = 3538.9$ (9) Å^3
$Z = 8$
$F(000) = 1488$
$D_x = 1.336$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6192 reflections
$\theta = 2.1$–$32.2°$
$\mu = 0.23$ mm^{-1}
$T = 295$ K
Block, colourless
0.40 × 0.30 × 0.20 mm

Data collection

Bruker APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.767$, $T_{max} = 0.954$
33715 measured reflections
4079 independent reflections
3038 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 27.6°$, $\theta_{min} = 2.1°$
$h = -29 \rightarrow 29$
$k = -8 \rightarrow 10$
$l = -28 \rightarrow 28$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.149$
$S = 1.03$
4079 reflections
319 parameters
64 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0639P)^2 + 3.3163P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.007$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0042 (5)

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)
C11	0.5797 (2)	0.6114 (5)	0.3652 (2)	0.0430 (10)	0.6020 (18)
O11	0.6397 (10)	0.563 (3)	0.404 (2)	0.093 (3)	0.6020 (18)
N11	0.5674 (5)	0.7661 (13)	0.3420 (9)	0.0447 (8)	0.6020 (18)
H11	0.532 (3)	0.811 (7)	0.332 (3)	0.054*	0.6020 (18)
C12	0.51822 (15)	0.5266 (4)	0.3591 (2)	0.0521 (7)	0.6020 (18)
H12A	0.4917	0.6049	0.3675	0.063*	0.6020 (18)
H12B	0.4902	0.4862	0.3103	0.063*	0.6020 (18)
N111	0.7060 (7)	1.016 (3)	0.3680 (6)	0.0452 (7)	0.6020 (18)

N112	0.6676 (5)	1.0041 (18)	0.2923 (6)	0.0418 (9)	0.6020 (18)
C113	0.6138 (4)	0.8990 (11)	0.2722 (5)	0.0327 (15)	0.6020 (18)
O113	0.5739 (5)	0.8552 (12)	0.2095 (5)	0.0432 (15)	0.6020 (18)
C114	0.6156 (6)	0.8630 (13)	0.3379 (5)	0.0365 (13)	0.6020 (18)
C115	0.6693 (13)	0.939 (5)	0.3931 (5)	0.0435 (6)	0.6020 (18)
C116	0.734 (2)	1.177 (4)	0.3956 (12)	0.0678 (8)	0.6020 (18)
H11A	0.7651	1.1711	0.4467	0.102*	0.6020 (18)
H11B	0.6974	1.2492	0.3863	0.102*	0.6020 (18)
H11C	0.7566	1.2161	0.3720	0.102*	0.6020 (18)
C117	0.6917 (11)	0.943 (3)	0.4710 (5)	0.0647 (10)	0.6020 (18)
H11D	0.6630	0.8747	0.4797	0.097*	0.6020 (18)
H11E	0.6889	1.0517	0.4848	0.097*	0.6020 (18)
H11F	0.7377	0.9062	0.4990	0.097*	0.6020 (18)
C121	0.5354 (3)	0.3900 (4)	0.41070 (17)	0.0420 (10)	0.6020 (18)
C122	0.5669 (4)	0.4185 (6)	0.4834 (2)	0.0549 (11)	0.6020 (18)
C123	0.5817 (8)	0.2935 (9)	0.5341 (3)	0.0690 (13)	0.6020 (18)
H123	0.6027	0.3151	0.5832	0.083*	0.6020 (18)
C124	0.5641 (15)	0.1393 (9)	0.5081 (4)	0.0723 (14)	0.6020 (18)
H124	0.5723	0.0553	0.5401	0.087*	0.6020 (18)
C125	0.5348 (10)	0.1072 (6)	0.4367 (4)	0.0722 (13)	0.6020 (18)
H125	0.5256	0.0011	0.4205	0.087*	0.6020 (18)
C126	0.5187 (4)	0.2301 (5)	0.3883 (3)	0.0604 (11)	0.6020 (18)
H126	0.4961	0.2066	0.3392	0.073*	0.6020 (18)
C131	0.7008 (5)	1.034 (3)	0.2530 (7)	0.0450 (5)	0.6020 (18)
C132	0.7707 (5)	1.0201 (12)	0.2868 (6)	0.0567 (18)	0.6020 (18)
H132	0.7963	0.9930	0.3355	0.068*	0.6020 (18)
C133	0.8017 (6)	1.047 (2)	0.2473 (9)	0.077 (3)	0.6020 (18)
H133	0.8481	1.0310	0.2688	0.092*	0.6020 (18)
C134	0.7653 (8)	1.0974 (18)	0.1777 (8)	0.076 (2)	0.6020 (18)
H134	0.7866	1.1159	0.1516	0.091*	0.6020 (18)
C135	0.6974 (9)	1.120 (4)	0.1464 (7)	0.075 (3)	0.6020 (18)
H135	0.6730	1.1608	0.0997	0.091*	0.6020 (18)
C136	0.6634 (8)	1.084 (4)	0.1829 (8)	0.0616 (14)	0.6020 (18)
H136	0.6166	1.0947	0.1601	0.074*	0.6020 (18)
Cl12	0.59006 (6)	0.6106 (2)	0.51896 (6)	0.0847 (4)	0.6020 (18)
C21	0.5859 (3)	0.6482 (9)	0.3872 (4)	0.0430 (10)	0.3980 (18)
O21	0.6348 (15)	0.564 (4)	0.395 (4)	0.093 (3)	0.3980 (18)
N21	0.5662 (7)	0.779 (2)	0.3433 (13)	0.0447 (8)	0.3980 (18)
H21	0.520 (4)	0.793 (11)	0.317 (4)	0.054*	0.3980 (18)
C22	0.5300 (2)	0.5857 (6)	0.3992 (3)	0.0521 (7)	0.3980 (18)
H22A	0.5240	0.6613	0.4297	0.063*	0.3980 (18)
H22B	0.4878	0.5817	0.3531	0.063*	0.3980 (18)
N211	0.7063 (11)	1.023 (4)	0.3698 (9)	0.0452 (7)	0.3980 (18)
N212	0.6664 (8)	1.018 (3)	0.2941 (8)	0.0418 (9)	0.3980 (18)
C213	0.6083 (7)	0.928 (2)	0.2732 (7)	0.0327 (15)	0.3980 (18)
O213	0.5674 (8)	0.887 (2)	0.2103 (8)	0.0432 (15)	0.3980 (18)
C214	0.6123 (10)	0.884 (2)	0.3393 (8)	0.0365 (13)	0.3980 (18)
C215	0.6706 (19)	0.943 (7)	0.3952 (8)	0.0435 (6)	0.3980 (18)

C216	0.736 (3)	1.181 (6)	0.3999 (18)	0.0678 (8)	0.3980 (18)
H21A	0.7561	1.2248	0.3745	0.102*	0.3980 (18)
H21B	0.7694	1.1699	0.4501	0.102*	0.3980 (18)
H21C	0.7006	1.2526	0.3949	0.102*	0.3980 (18)
C217	0.6953 (16)	0.939 (5)	0.4738 (8)	0.0647 (10)	0.3980 (18)
H21D	0.6740	1.0236	0.4853	0.097*	0.3980 (18)
H21E	0.7435	0.9541	0.5007	0.097*	0.3980 (18)
H21F	0.6844	0.8367	0.4861	0.097*	0.3980 (18)
C221	0.5440 (5)	0.4227 (7)	0.4331 (4)	0.0420 (10)	0.3980 (18)
C222	0.5713 (7)	0.4054 (11)	0.5057 (4)	0.0549 (11)	0.3980 (18)
H222	0.5848	0.4970	0.5346	0.066*	0.3980 (18)
C223	0.5797 (14)	0.2525 (14)	0.5387 (5)	0.0690 (13)	0.3980 (18)
H223	0.5995	0.2424	0.5884	0.083*	0.3980 (18)
C224	0.558 (2)	0.1199 (13)	0.4944 (7)	0.0723 (14)	0.3980 (18)
H224	0.5629	0.0180	0.5146	0.087*	0.3980 (18)
C225	0.5279 (16)	0.1345 (10)	0.4219 (7)	0.0722 (13)	0.3980 (18)
H225	0.5089	0.0448	0.3923	0.087*	0.3980 (18)
C226	0.5260 (8)	0.2811 (9)	0.3921 (4)	0.0604 (11)	0.3980 (18)
C231	0.6996 (7)	1.036 (5)	0.2540 (10)	0.0450 (5)	0.3980 (18)
C232	0.7656 (8)	0.982 (2)	0.2826 (10)	0.0567 (18)	0.3980 (18)
H232	0.7876	0.9274	0.3265	0.068*	0.3980 (18)
C233	0.7983 (10)	1.010 (4)	0.2448 (14)	0.077 (3)	0.3980 (18)
H233	0.8447	0.9931	0.2671	0.092*	0.3980 (18)
C234	0.7630 (13)	1.062 (3)	0.1754 (13)	0.076 (2)	0.3980 (18)
H234	0.7846	1.0769	0.1494	0.091*	0.3980 (18)
C235	0.6956 (13)	1.093 (6)	0.1443 (11)	0.075 (3)	0.3980 (18)
H235	0.6702	1.1153	0.0951	0.091*	0.3980 (18)
C236	0.6638 (11)	1.093 (7)	0.1849 (12)	0.0616 (14)	0.3980 (18)
H236	0.6197	1.1306	0.1654	0.074*	0.3980 (18)
Cl26	0.4895 (2)	0.2868 (4)	0.2999 (2)	0.1328 (11)	0.3980 (18)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0348 (12)	0.049 (2)	0.050 (3)	−0.0007 (12)	0.0253 (18)	0.0039 (16)
O11	0.043 (2)	0.0891 (13)	0.151 (9)	0.0239 (15)	0.053 (5)	0.066 (2)
N11	0.0298 (7)	0.0521 (17)	0.0548 (10)	0.0040 (8)	0.0236 (7)	0.0137 (14)
C12	0.0339 (12)	0.060 (2)	0.0599 (19)	−0.0029 (12)	0.0222 (15)	0.0149 (14)
N111	0.0397 (7)	0.0557 (18)	0.0398 (9)	−0.0131 (8)	0.0201 (7)	−0.0099 (8)
N112	0.0371 (7)	0.051 (2)	0.0396 (9)	−0.0126 (10)	0.0211 (6)	−0.0059 (10)
C113	0.0284 (15)	0.029 (4)	0.0435 (9)	0.003 (2)	0.0202 (9)	−0.0015 (16)
O113	0.0343 (18)	0.052 (4)	0.0404 (7)	−0.007 (2)	0.0170 (9)	−0.0066 (18)
C114	0.0312 (12)	0.038 (3)	0.0442 (9)	0.0035 (18)	0.0222 (8)	0.0033 (13)
C115	0.0362 (11)	0.0553 (19)	0.0418 (10)	0.0001 (8)	0.0221 (10)	−0.0007 (12)
C116	0.066 (2)	0.070 (2)	0.067 (3)	−0.0303 (13)	0.033 (3)	−0.0273 (16)
C117	0.050 (2)	0.1021 (19)	0.0416 (12)	−0.003 (2)	0.0234 (13)	−0.0033 (14)
C121	0.0298 (16)	0.051 (2)	0.042 (2)	−0.0077 (15)	0.017 (2)	0.0010 (18)
C122	0.0437 (16)	0.0665 (17)	0.054 (3)	−0.0084 (13)	0.025 (3)	0.004 (2)

C123	0.0453 (15)	0.098 (4)	0.0595 (15)	−0.005 (4)	0.0238 (14)	0.0260 (19)
C124	0.051 (5)	0.068 (2)	0.086 (3)	0.000 (3)	0.027 (6)	0.029 (2)
C125	0.069 (4)	0.047 (2)	0.092 (4)	0.009 (4)	0.036 (5)	0.014 (2)
C126	0.053 (2)	0.066 (3)	0.0685 (16)	−0.011 (3)	0.0349 (15)	0.004 (2)
C131	0.0470 (9)	0.0461 (11)	0.0512 (10)	−0.0155 (8)	0.0320 (8)	−0.0093 (8)
C132	0.0483 (17)	0.055 (5)	0.0749 (18)	−0.014 (3)	0.0372 (15)	−0.005 (3)
C133	0.0643 (18)	0.082 (9)	0.111 (2)	−0.022 (3)	0.0634 (19)	−0.019 (4)
C134	0.105 (2)	0.066 (7)	0.102 (2)	−0.050 (4)	0.086 (2)	−0.041 (3)
C135	0.101 (2)	0.087 (9)	0.0580 (14)	−0.037 (3)	0.0546 (15)	−0.018 (3)
C136	0.0594 (12)	0.083 (4)	0.0477 (12)	−0.0184 (13)	0.0316 (10)	−0.0078 (18)
Cl12	0.0808 (7)	0.0924 (8)	0.0814 (7)	−0.0292 (6)	0.0420 (6)	−0.0263 (6)
C21	0.0348 (12)	0.049 (2)	0.050 (3)	−0.0007 (12)	0.0253 (18)	0.0039 (16)
O21	0.043 (2)	0.0891 (13)	0.151 (9)	0.0239 (15)	0.053 (5)	0.066 (2)
N21	0.0298 (7)	0.0521 (17)	0.0548 (10)	0.0040 (8)	0.0236 (7)	0.0137 (14)
C22	0.0339 (12)	0.060 (2)	0.0599 (19)	−0.0029 (12)	0.0222 (15)	0.0149 (14)
N211	0.0397 (7)	0.0557 (18)	0.0398 (9)	−0.0131 (8)	0.0201 (7)	−0.0099 (8)
N212	0.0371 (7)	0.051 (2)	0.0396 (9)	−0.0126 (10)	0.0211 (6)	−0.0059 (10)
C213	0.0284 (15)	0.029 (4)	0.0435 (9)	0.003 (2)	0.0202 (9)	−0.0015 (16)
O213	0.0343 (18)	0.052 (4)	0.0404 (7)	−0.007 (2)	0.0170 (9)	−0.0066 (18)
C214	0.0312 (12)	0.038 (3)	0.0442 (9)	0.0035 (18)	0.0222 (8)	0.0033 (13)
C215	0.0362 (11)	0.0553 (19)	0.0418 (10)	0.0001 (8)	0.0221 (10)	−0.0007 (12)
C216	0.066 (2)	0.070 (2)	0.067 (3)	−0.0303 (13)	0.033 (3)	−0.0273 (16)
C217	0.050 (2)	0.1021 (19)	0.0416 (12)	−0.003 (2)	0.0234 (13)	−0.0033 (14)
C221	0.0298 (16)	0.051 (2)	0.042 (2)	−0.0077 (15)	0.017 (2)	0.0010 (18)
C222	0.0437 (16)	0.0665 (17)	0.054 (3)	−0.0084 (13)	0.025 (3)	0.004 (2)
C223	0.0453 (15)	0.098 (4)	0.0595 (15)	−0.005 (4)	0.0238 (14)	0.0260 (19)
C224	0.051 (5)	0.068 (2)	0.086 (3)	0.000 (3)	0.027 (6)	0.029 (2)
C225	0.069 (4)	0.047 (2)	0.092 (4)	0.009 (4)	0.036 (5)	0.014 (2)
C226	0.053 (2)	0.066 (3)	0.0685 (16)	−0.011 (3)	0.0349 (15)	0.004 (2)
C231	0.0470 (9)	0.0461 (11)	0.0512 (10)	−0.0155 (8)	0.0320 (8)	−0.0093 (8)
C232	0.0483 (17)	0.055 (5)	0.0749 (18)	−0.014 (3)	0.0372 (15)	−0.005 (3)
C233	0.0643 (18)	0.082 (9)	0.111 (2)	−0.022 (3)	0.0634 (19)	−0.019 (4)
C234	0.105 (2)	0.066 (7)	0.102 (2)	−0.050 (4)	0.086 (2)	−0.041 (3)
C235	0.101 (2)	0.087 (9)	0.0580 (14)	−0.037 (3)	0.0546 (15)	−0.018 (3)
C236	0.0594 (12)	0.083 (4)	0.0477 (12)	−0.0184 (13)	0.0316 (10)	−0.0078 (18)
Cl26	0.150 (2)	0.183 (3)	0.0636 (11)	−0.0293 (19)	0.0536 (13)	0.0027 (13)

Geometric parameters (Å, º)

C11—O11	1.257 (15)	C21—O21	1.258 (16)
C11—N11	1.353 (5)	C21—N21	1.355 (5)
C11—C12	1.524 (3)	C21—C22	1.528 (4)
N11—C114	1.410 (3)	N21—C214	1.410 (3)
N11—H11	0.82 (7)	N21—H21	0.91 (8)
C12—C121	1.493 (3)	C22—C221	1.492 (4)
C12—H12A	0.9700	C22—H22A	0.9700
C12—H12B	0.9700	C22—H22B	0.9700
N111—C115	1.373 (3)	N211—C215	1.373 (4)

N111—N112	1.407 (3)	N211—N212	1.406 (4)
N111—C116	1.467 (3)	N211—C216	1.467 (4)
N112—C113	1.389 (3)	N212—C213	1.389 (4)
N112—C131	1.427 (3)	N212—C231	1.427 (4)
C113—O113	1.237 (3)	C213—O213	1.237 (3)
C113—C114	1.430 (3)	C213—C214	1.430 (4)
C114—C115	1.353 (5)	C214—C215	1.353 (5)
C115—C117	1.487 (4)	C215—C217	1.487 (4)
C116—H11A	0.9600	C216—H21A	0.9600
C116—H11B	0.9600	C216—H21B	0.9600
C116—H11C	0.9600	C216—H21C	0.9600
C117—H11D	0.9600	C217—H21D	0.9600
C117—H11E	0.9600	C217—H21E	0.9600
C117—H11F	0.9600	C217—H21F	0.9600
C121—C122	1.370 (4)	C221—C222	1.365 (5)
C121—C126	1.397 (4)	C221—C226	1.399 (5)
C122—C123	1.417 (4)	C222—C223	1.418 (5)
C122—Cl12	1.730 (5)	C222—H222	0.9300
C123—C124	1.372 (5)	C223—C224	1.372 (5)
C123—H123	0.9300	C223—H223	0.9300
C124—C125	1.354 (5)	C224—C225	1.355 (5)
C124—H124	0.9300	C224—H224	0.9300
C125—C126	1.368 (5)	C225—C226	1.366 (5)
C125—H125	0.9300	C225—H225	0.9300
C126—H126	0.9300	C226—Cl26	1.719 (7)
C131—C136	1.366 (4)	C231—C236	1.366 (4)
C131—C132	1.388 (5)	C231—C232	1.388 (6)
C132—C133	1.385 (4)	C232—C233	1.386 (5)
C132—H132	0.9300	C232—H232	0.9300
C133—C134	1.356 (5)	C233—C234	1.357 (5)
C133—H133	0.9300	C233—H233	0.9300
C134—C135	1.360 (5)	C234—C235	1.360 (5)
C134—H134	0.9300	C234—H234	0.9300
C135—C136	1.401 (7)	C235—C236	1.402 (8)
C135—H135	0.9300	C235—H235	0.9300
C136—H136	0.9300	C236—H236	0.9300

O11—C11—N11	119.8 (9)	O21—C21—N21	119.6 (12)
O11—C11—C12	124.4 (4)	O21—C21—C22	124.0 (6)
N11—C11—C12	113.4 (3)	N21—C21—C22	112.5 (4)
C11—N11—C114	122.9 (5)	C21—N21—C214	123.1 (6)
C11—N11—H11	121 (4)	C21—N21—H21	114 (6)
C114—N11—H11	116 (4)	C214—N21—H21	123 (6)
C121—C12—C11	114.1 (3)	C221—C22—C21	113.8 (4)
C121—C12—H12A	108.7	C221—C22—H22A	108.8
C11—C12—H12A	108.7	C21—C22—H22A	108.8
C121—C12—H12B	108.7	C221—C22—H22B	108.8
C11—C12—H12B	108.7	C21—C22—H22B	108.8

H12A—C12—H12B	107.6	H22A—C22—H22B	107.7
C115—N111—N112	106.2 (2)	C215—N211—N212	106.5 (4)
C115—N111—C116	119.4 (4)	C215—N211—C216	119.3 (6)
N112—N111—C116	114.8 (4)	N212—N211—C216	114.7 (6)
C113—N112—N111	109.5 (3)	C213—N212—N211	109.7 (3)
C113—N112—C131	126.0 (4)	C213—N212—C231	125.9 (6)
N111—N112—C131	118.1 (4)	N211—N212—C231	118.0 (6)
O113—C113—N112	124.5 (3)	O213—C213—N212	124.4 (5)
O113—C113—C114	130.7 (3)	O213—C213—C214	130.4 (5)
N112—C113—C114	104.8 (2)	N212—C213—C214	104.9 (3)
C115—C114—N11	127.0 (4)	C215—C214—N21	126.9 (6)
C115—C114—C113	108.9 (2)	C215—C214—C213	108.9 (3)
N11—C114—C113	124.1 (3)	N21—C214—C213	123.8 (5)
C114—C115—N111	109.8 (4)	C214—C215—N211	109.9 (3)
C114—C115—C117	129.5 (3)	C214—C215—C217	129.3 (6)
N111—C115—C117	120.7 (4)	N211—C215—C217	120.7 (5)
N111—C116—H11A	109.5	N211—C216—H21A	109.5
N111—C116—H11B	109.5	N211—C216—H21B	109.5
H11A—C116—H11B	109.5	H21A—C216—H21B	109.5
N111—C116—H11C	109.5	N211—C216—H21C	109.5
H11A—C116—H11C	109.5	H21A—C216—H21C	109.5
H11B—C116—H11C	109.5	H21B—C216—H21C	109.5
C115—C117—H11D	109.5	C215—C217—H21D	109.5
C115—C117—H11E	109.5	C215—C217—H21E	109.5
H11D—C117—H11E	109.5	H21D—C217—H21E	109.5
C115—C117—H11F	109.5	C215—C217—H21F	109.5
H11D—C117—H11F	109.5	H21D—C217—H21F	109.5
H11E—C117—H11F	109.5	H21E—C217—H21F	109.5
C122—C121—C126	116.8 (3)	C222—C221—C226	116.8 (4)
C122—C121—C12	120.3 (3)	C222—C221—C22	121.0 (5)
C126—C121—C12	122.9 (3)	C226—C221—C22	122.1 (5)
C121—C122—C123	122.3 (3)	C221—C222—C223	122.3 (5)
C121—C122—Cl12	121.9 (3)	C221—C222—H222	118.9
C123—C122—Cl12	115.7 (3)	C223—C222—H222	118.9
C124—C123—C122	117.6 (3)	C224—C223—C222	117.5 (5)
C124—C123—H123	121.2	C224—C223—H223	121.3
C122—C123—H123	121.2	C222—C223—H223	121.3
C125—C124—C123	121.3 (4)	C225—C224—C223	121.3 (5)
C125—C124—H124	119.4	C225—C224—H224	119.3
C123—C124—H124	119.4	C223—C224—H224	119.3
C124—C125—C126	120.2 (4)	C224—C225—C226	119.9 (6)
C124—C125—H125	119.9	C224—C225—H225	120.0
C126—C125—H125	119.9	C226—C225—H225	120.0
C125—C126—C121	121.7 (4)	C225—C226—C221	121.5 (5)
C125—C126—H126	119.2	C225—C226—Cl26	116.6 (6)
C121—C126—H126	119.2	C221—C226—Cl26	121.3 (5)
C136—C131—C132	120.6 (4)	C236—C231—C232	120.4 (6)
C136—C131—N112	119.1 (4)	C236—C231—N212	119.3 (5)

C132—C131—N112	120.2 (3)	C232—C231—N212	120.1 (5)
C133—C132—C131	119.2 (4)	C233—C232—C231	119.0 (6)
C133—C132—H132	120.4	C233—C232—H232	120.5
C131—C132—H132	120.4	C231—C232—H232	120.5
C134—C133—C132	120.8 (4)	C234—C233—C232	120.5 (5)
C134—C133—H133	119.6	C234—C233—H233	119.8
C132—C133—H133	119.6	C232—C233—H233	119.8
C133—C134—C135	119.4 (3)	C233—C234—C235	119.3 (5)
C133—C134—H134	120.3	C233—C234—H234	120.4
C135—C134—H134	120.3	C235—C234—H234	120.4
C134—C135—C136	121.6 (4)	C234—C235—C236	121.3 (6)
C134—C135—H135	119.2	C234—C235—H235	119.3
C136—C135—H135	119.2	C236—C235—H235	119.3
C131—C136—C135	118.2 (5)	C231—C236—C235	118.0 (7)
C131—C136—H136	120.9	C231—C236—H236	121.0
C135—C136—H136	120.9	C235—C236—H236	121.0

O11—C11—N11—C114	21 (3)	O21—C21—N21—C214	−39 (4)
C12—C11—N11—C114	−175.3 (8)	C22—C21—N21—C214	162.3 (13)
O11—C11—C12—C121	8 (3)	O21—C21—C22—C221	9 (4)
N11—C11—C12—C121	−154.8 (9)	N21—C21—C22—C221	167.1 (14)
C115—N111—N112—C113	−9 (2)	C215—N211—N212—C213	−3 (3)
C116—N111—N112—C113	−144 (2)	C216—N211—N212—C213	−137 (3)
C115—N111—N112—C131	−163 (2)	C215—N211—N212—C231	−157 (3)
C116—N111—N112—C131	62.8 (19)	C216—N211—N212—C231	69 (3)
N111—N112—C113—O113	−174.6 (13)	N211—N212—C213—O213	−171 (2)
C131—N112—C113—O113	−23.6 (15)	C231—N212—C213—O213	−20 (3)
N111—N112—C113—C114	7.2 (13)	N211—N212—C213—C214	3 (2)
C131—N112—C113—C114	158.2 (11)	C231—N212—C213—C214	153.7 (18)
C11—N11—C114—C115	−79 (3)	C21—N21—C214—C215	−44 (5)
C11—N11—C114—C113	102.8 (15)	C21—N21—C214—C213	128 (2)
O113—C113—C114—C115	180 (2)	O213—C213—C214—C215	172 (4)
N112—C113—C114—C115	−2 (2)	N212—C213—C214—C215	−1 (4)
O113—C113—C114—N11	−1 (2)	O213—C213—C214—N21	−1 (3)
N112—C113—C114—N11	176.6 (11)	N212—C213—C214—N21	−173.9 (18)
N11—C114—C115—N111	177.5 (16)	N21—C214—C215—N211	172 (3)
C113—C114—C115—N111	−4 (3)	C213—C214—C215—N211	−1 (5)
N11—C114—C115—C117	−1 (6)	N21—C214—C215—C217	−12 (9)
C113—C114—C115—C117	178 (4)	C213—C214—C215—C217	175 (6)
N112—N111—C115—C114	8 (3)	N212—N211—C215—C214	2 (5)
C116—N111—C115—C114	140 (3)	C216—N211—C215—C214	134 (5)
N112—N111—C115—C117	−174 (3)	N212—N211—C215—C217	−174 (5)
C116—N111—C115—C117	−42 (3)	C216—N211—C215—C217	−42 (5)
C11—C12—C121—C122	67.3 (7)	C21—C22—C221—C222	96.8 (11)
C11—C12—C121—C126	−115.0 (6)	C21—C22—C221—C226	−87.3 (11)
C126—C121—C122—C123	−0.4 (14)	C226—C221—C222—C223	−2 (2)
C12—C121—C122—C123	177.3 (10)	C22—C221—C222—C223	174.4 (17)
C126—C121—C122—Cl12	−179.5 (6)	C221—C222—C223—C224	−2 (4)

C12—C121—C122—Cl12	−1.7 (10)	C222—C223—C224—C225	−1 (6)
C121—C122—C123—C124	1 (3)	C223—C224—C225—C226	7 (7)
Cl12—C122—C123—C124	179.9 (19)	C224—C225—C226—C221	−11 (5)
C122—C123—C124—C125	1 (4)	C224—C225—C226—Cl26	178 (3)
C123—C124—C125—C126	−4 (4)	C222—C221—C226—C225	8 (3)
C124—C125—C126—C121	4 (3)	C22—C221—C226—C225	−168.2 (19)
C122—C121—C126—C125	−1.9 (15)	C222—C221—C226—Cl26	178.6 (11)
C12—C121—C126—C125	−179.6 (12)	C22—C221—C226—Cl26	2.6 (17)
C113—N112—C131—C136	56 (3)	C213—N212—C231—C236	56 (4)
N111—N112—C131—C136	−155 (2)	N211—N212—C231—C236	−155 (4)
C113—N112—C131—C132	−127.2 (19)	C213—N212—C231—C232	−118 (3)
N111—N112—C131—C132	22 (2)	N211—N212—C231—C232	31 (3)
C136—C131—C132—C133	−4 (3)	C236—C231—C232—C233	10 (5)
N112—C131—C132—C133	179.1 (13)	N212—C231—C232—C233	−176 (2)
C131—C132—C133—C134	4 (2)	C231—C232—C233—C234	−12 (4)
C132—C133—C134—C135	0 (3)	C232—C233—C234—C235	3 (5)
C133—C134—C135—C136	−4 (4)	C233—C234—C235—C236	9 (6)
C132—C131—C136—C135	1 (4)	C232—C231—C236—C235	1 (7)
N112—C131—C136—C135	177 (2)	N212—C231—C236—C235	−173 (4)
C134—C135—C136—C131	4 (5)	C234—C235—C236—C231	−10 (7)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H11···O113^{i}	0.82 (7)	2.15 (8)	2.94 (2)	162 (6)
N11—H11···O213^{i}	0.82 (7)	2.08 (8)	2.88 (2)	168 (6)
N21—H21···O113^{i}	0.92 (10)	2.00 (10)	2.91 (2)	161 (7)
N21—H21···O213^{i}	0.92 (10)	1.95 (10)	2.82 (3)	158 (7)
C134—H134···O11^{ii}	0.93	2.57	3.46 (4)	159
C234—H234···O21^{ii}	0.93	2.49	3.40 (7)	164
C225—H225···O213^{iii}	0.93	2.39	3.28 (2)	160

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