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Four 2-aryl-8,8-dimethyl-6,7,8,9-tetrahydropyrazolo[2,3-*a*]quinazolin-6-ones: isolated molecules, hydrogen-bonded dimers, and π-stacked chains of hydrogen-bonded dimers

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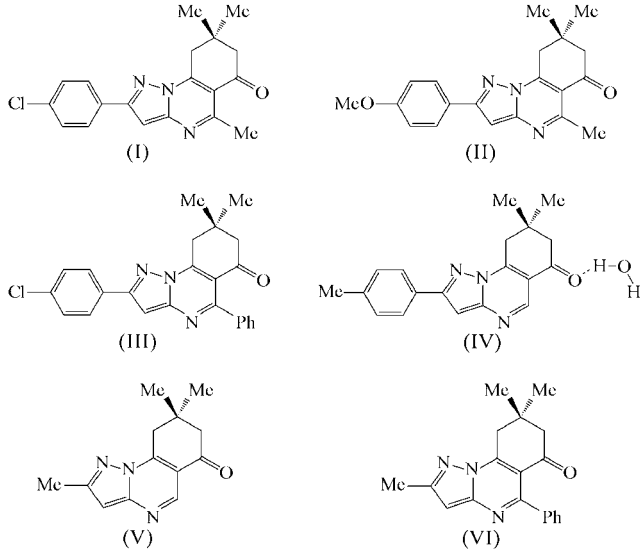
In each of the three compounds 2-(4-chlorophenyl)-5,8,8-trimethyl-6,7,8,9-tetrahydropyrazolo[2,3-*a*]quinazolin-6-one, $C_{19}H_{18}ClN_3O$, (I), 2-(4-methoxyphenyl)-5,8,8-trimethyl-6,7,8,9-tetrahydropyrazolo[2,3-*a*]quinazolin-6-one, $C_{20}H_{21}N_3O_2$, (II), and 8,8-dimethyl-2-(4-methylphenyl)-6,7,8,9-tetrahydropyrazolo[2,3-*a*]quinazolin-6-one monohydrate, $C_{19}H_{19}N_3O\cdot H_2O$, (IV), the non-aromatic carbocyclic ring adopts a half-chair conformation, while in 2-(4-chlorophenyl)-8,8-dimethyl-5-phenyl-6,7,8,9-tetrahydropyrazolo[2,3-*a*]quinazolin-6-one, $C_{24}H_{20}ClN_3O$, (III), the corresponding ring adopts a conformation intermediate between the envelope and screw–boat forms. The structure of (I) consists of isolated molecules, while that of (II) contains dimers formed by C—H···O hydrogen bonds. In (III), dimers formed by C—H···O hydrogen bonds are linked into chains by means of an aromatic π–π stacking interaction, while in the monohydrate, (IV), the heterocyclic molecules and the water molecules are linked by O—H···O and O—H···N hydrogen bonds to form centrosymmetric four-component aggregates.

Comment

As part of our synthetic study of fused pyrazole systems, we are now focusing on pyrazoloquinazoline derivatives, which are important pharmacophores (Fry *et al.*, 1994). We have previously reported different methods for the preparation of this class of compound *via* solvent-free procedures under microwave irradiation, *via* both three-component cyclocondensation using a 5-amino-1*H*-pyrazole, 5,5-dimethylcyclohexane-1,3-dione (dimedone) and formaldehyde (Low *et al.*, 2004*a*), and a two-component cyclocondensation with the corresponding 5-amino-1*H*-pyrazole and 2-acetyl-1-tetralone (Low, Cobo, Quiroga *et al.*, 2004; Portilla *et al.*, 2005). Here, we



report the molecular and supramolecular structures of four new substituted 6,7,8,9-tetrahydropyrazolo[2,3-*a*]quinazolin-6-ones, namely 2-(4-chlorophenyl)-5,8,8-trimethyl-6,7,8,9-tetrahydropyrazolo[2,3-*a*]quinazolin-6-one, (I), 2-(4-meth-

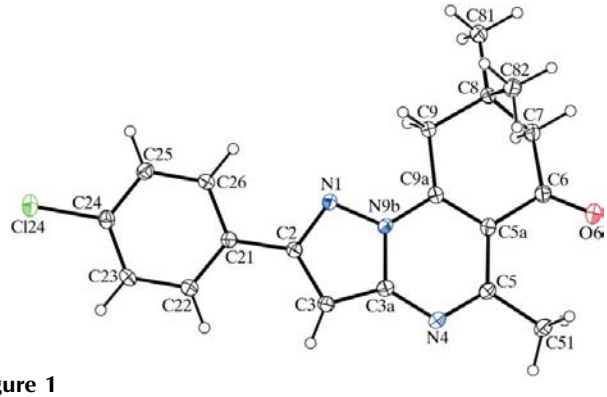


Figure 1
A view of the molecule of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

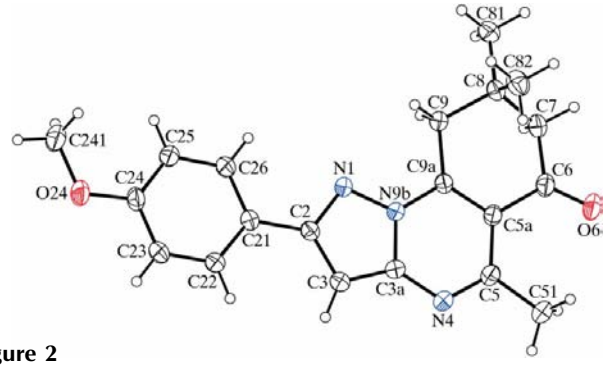


Figure 2
A view of the molecule of compound (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

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oxyphenyl)-5,8,8-trimethyl-6,7,8,9-tetrahydropyrazolo[2,3-*a*]-quinazolin-6-one, (II), 2-(4-chlorophenyl)-8,8-dimethyl-5-phenyl-6,7,8,9-tetrahydropyrazolo[2,3-*a*]quinazolin-6-one, (III), and 8,8-dimethyl-2-(4-methylphenyl)-6,7,8,9-tetrahydropyrazolo[2,3-*a*]quinazolin-6-one monohydrate, (IV) (Figs. 1–4). Compounds (I), (III) and (IV) were prepared using microwave irradiation of three-component reaction mixtures containing a 5-amino-3-aryl-1*H*-pyrazole, dimedone and an orthoester, but in the absence of any solvent, while compound (II) was prepared similarly using a solvent-free two-component cyclocondensation of a 5-amino-3-aryl-1*H*-pyrazole with 2-acetyldimedone. Each of compounds (I)–(IV) has an aryl substituent at position C2, and we briefly compare the structures of (I)–(IV) with those of (V) and (VI) (Low *et al.*, 2004*a*,*b*), each of which has a methyl substituent at C2.

In each of compounds (I)–(IV), there is considerable bond fixation in the heterocyclic rings, with rather little variation in the bond distances from one compound to another (Table 1), indicating that the classical representation shown in the scheme is appropriate. The ring-puckering parameters (Cremer & Pople, 1975) for the non-aromatic carbocylic rings, calculated for the atom sequence C5a/C6–C9/C9a in each case (Table 1), indicate that, in compounds (I), (II) and (IV), this ring adopts a half-chair conformation, for which the idealized parameters are $\theta = 50.8^\circ$ and $\varphi = (60n + 30)/5$. By contrast, in compound (III), the conformation of the non-aromatic carbocyclic ring is best described as intermediate between screw–boat and envelope forms; for these conformations, the idealized parameters are $\theta = 67.5$ and 54.7°, and $\varphi = (60n + 30)$ and $60n^\circ$, respectively. The corresponding rings in compounds (V) and (VI) both adopt envelope conformations (Low *et al.*, 2004*a*,*b*). The aryl rings at the C2 positions deviate only modestly from being coplanar with the adjacent pyrazole rings, as shown by the dihedral angles between these rings (Table 1), but the 5-phenyl ring in compound (III) is significantly rotated about C5—C51 out of the plane of the adjacent pyrimidine ring, possibly because of a repulsive intramolecular interaction between atoms H52 and O6 (Fig. 3).

There are no direction-specific interactions of any kind between the molecules in compound (I), but the molecules in compound (II) are linked into centrosymmetric $R_2^2(8)$ (Bernstein *et al.*, 1995) dimers (Fig. 5) by paired C—H···O hydrogen bonds, in which both the donor and the acceptor are parts of the 4-methoxyphenyl substituent (Table 2). There are no direction-specific interactions between these dimers.

In compound (III), the molecules are again linked into centrosymmetric $R_2^2(16)$ dimers by means of paired C—H···O hydrogen bonds, where now the donor lies in the unsub-

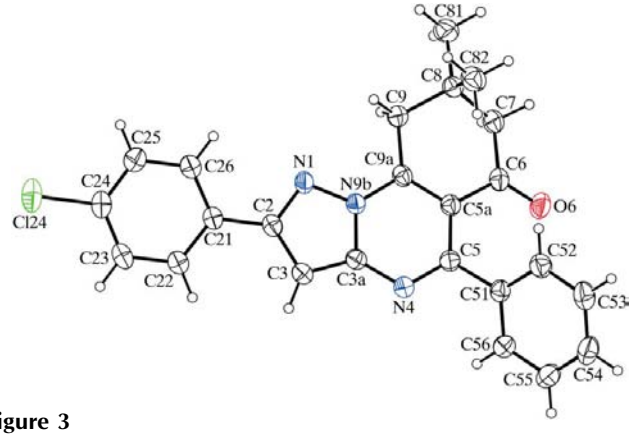

Figure 3
A view of the molecule of compound (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

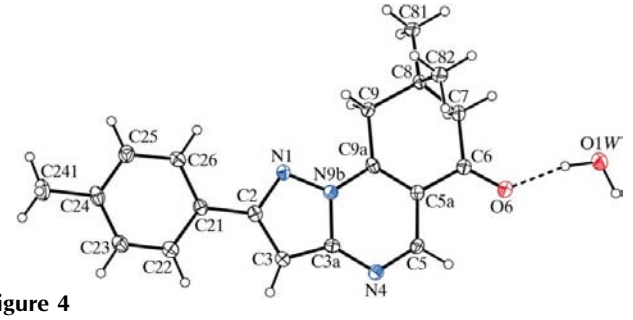

Figure 4
The independent molecular components of compound (IV), showing the atom-labelling scheme and the O—H···O hydrogen bond (dashed line) within the selected asymmetric unit. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

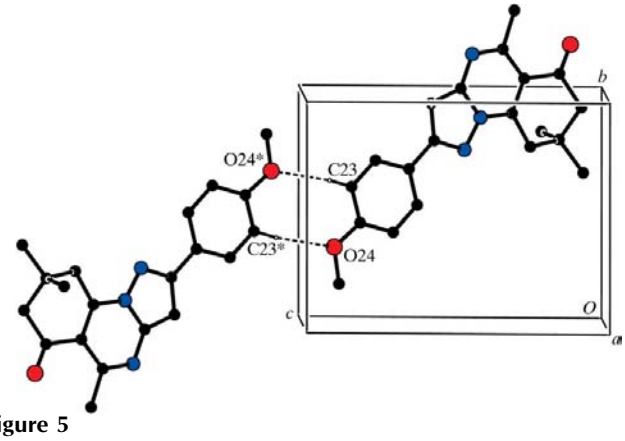

Figure 5
Part of the crystal structure of compound (II), showing the formation of a centrosymmetric $R_2^2(8)$ dimer. Hydrogen bonds are shown as dashed lines. For the sake of clarity, H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(1 - x, 1 - y, 2 - z)$.

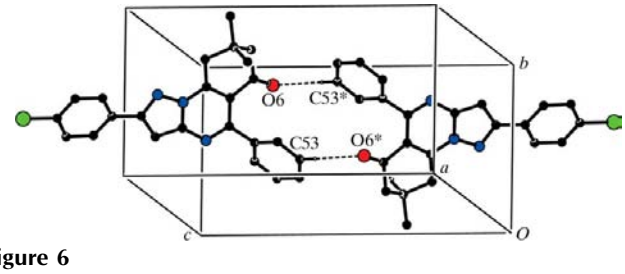

Figure 6
Part of the crystal structure of compound (III), showing the formation of a centrosymmetric $R_2^2(16)$ dimer. Hydrogen bonds are shown as dashed lines. For the sake of clarity, H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(1 - x, 1 - y, 1 - z)$.

stituted 5-phenyl ring and the acceptor is carbonyl atom O6 (Table 3 and Fig. 6). These hydrogen-bonded dimers are linked by a single aromatic π–π stacking interaction. The substituted phenyl rings (C21–C26) in the molecules at (x, y, z) and $(3-x, -y, 2-z)$, which lie in the hydrogen-bonded dimers centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ and $(\frac{5}{2}, -\frac{1}{2}, \frac{3}{2})$, respectively, are strictly parallel, with an interplanar spacing of 3.619 (2) Å and a ring centroid separation of 3.765 (2) Å. Propagation by inversion of this stacking interaction then links the hydrogen-bonded dimers into chains running parallel to the $[2\bar{1}1]$ direction (Fig. 7). There are no direction-specific interactions between adjacent chains.

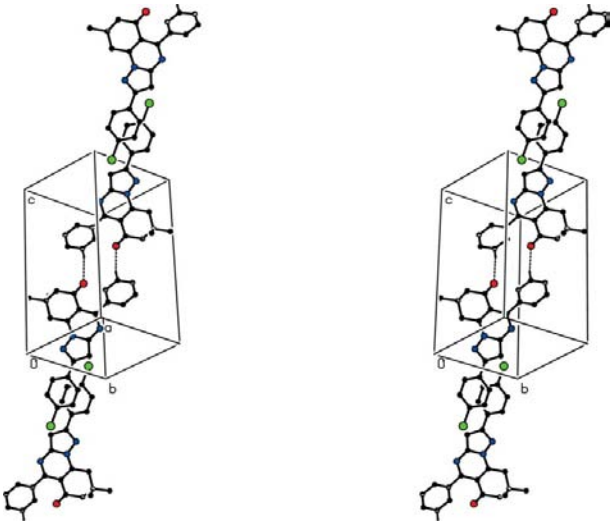

Figure 7
A stereoview of part of the crystal structure of compound (III), showing the formation of a π-stacked $[2\bar{1}1]$ chain of hydrogen-bonded dimers. Hydrogen bonds are shown as dashed lines. For the sake of clarity, H atoms not involved in the motif shown have been omitted.

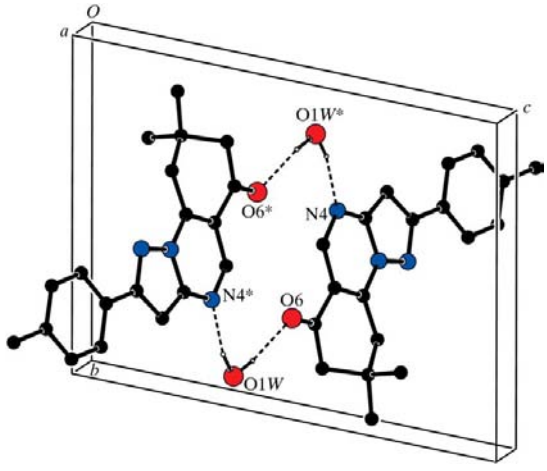

Figure 8
Part of the crystal structure of compound (IV), showing the formation of a centrosymmetric $R_4^4(16)$ aggregate. Hydrogen bonds are shown as dashed lines. For the sake of clarity, H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(3-x, 1-y, 1-z)$.

Unlike compounds (I)–(III), which all crystallize in solvent-free forms, compound (IV) crystallizes as a stoichiometric monohydrate (Fig. 4), and the molecules are linked by a combination of O—H···O and O—H···N hydrogen bonds (Table 4) to form a centrosymmetric four-molecule aggregate characterized by an $R_4^4(16)$ motif (Fig. 8). As in compound (II), there are no direction-specific interactions between the hydrogen-bonded units in (IV).

The different supramolecular structures of compounds (I)–(IV) may be contrasted with those of the analogues (V) and (VI). The molecules of (V) are linked by paired C—H···N hydrogen bonds into $R_2^2(6)$ dimers, which are further linked into chains by means of a π–π stacking interaction (Low *et al.*, 2004*a*), while the molecules of (VI) are linked by a single C—H···N hydrogen bond into simple $C(10)$ chains (Low *et al.*, 2004*b*). There are no C—H···O hydrogen bonds in the structures of (V) and (VI), and no C—H···N hydrogen bonds in any of (I)–(IV).

Experimental

For the syntheses of compounds (I), (III) and (IV), equimolar mixtures (1 mmol of each component), comprising a 5-amino-3-aryl-1*H*-pyrazole, where the aryl group is 4-chlorophenyl for (I) and (III) and 4-methylphenyl for (IV), 5,5-dimethylcyclohexane-1,3-dione (dimedone) and a triethyl orthoester, *viz*. acetate for (I), benzoate for (III) and formate for (IV), were placed in open Pyrex glass vessels in the absence of solvent and then irradiated in a domestic microwave oven at 600 W for 4 min for (I), 5 min for (III) or 2 min for (IV). The resulting solid products were washed with ethanol, dried and then recrystallized from ethanol to provide crystals suitable for single-crystal X-ray diffraction. For (I), m.p. 492 K, yield 60%; for (III), m.p. 498–499 K, yield 62%; for (IV), m.p. 466 K, yield 60%. For the synthesis of compound (II), an equimolar mixture (1 mmol of each component) of 5-amino-3-(4-methoxyphenyl)-1*H*-pyrazole and 2-acetyldimedone was placed in an open Pyrex glass vessel in the absence of solvent and irradiated in a domestic microwave oven at 600 W for 1.5 min. The reaction mixture was extracted with ethanol and, after removing the solvent, the solid product was recrystallized from dimethylformamide to give crystals suitable for single-crystal X-ray diffraction; m.p. 477–478 K, yield 80%.

Table 1
Selected geometric parameters (Å, °) for compounds (I)–(IV).

θ and φ are ring-puckering parameters (Cremer & Pople, 1975).

Parameter	(I)	(II)	(III)	(IV)
N1—C2	1.342 (3)	1.348 (2)	1.339 (2)	1.348 (4)
C2—C3	1.405 (3)	1.399 (2)	1.397 (2)	1.396 (4)
C3—C3a	1.381 (3)	1.373 (2)	1.370 (2)	1.376 (4)
C3a—N4	1.362 (3)	1.358 (2)	1.358 (2)	1.350 (4)
N4—C5	1.320 (3)	1.315 (2)	1.316 (2)	1.312 (4)
C5—C5a	1.447 (3)	1.442 (2)	1.445 (2)	1.422 (4)
C5a—C9a	1.376 (3)	1.379 (2)	1.380 (2)	1.371 (4)
C9a—N9b	1.357 (3)	1.359 (2)	1.362 (2)	1.360 (4)
N9b—N1	1.357 (2)	1.3651 (18)	1.3613 (18)	1.358 (4)
C3a—N9b	1.394 (3)	1.390 (2)	1.392 (2)	1.415 (4)
(Pyrazole)–(C21–C26)	5.6 (2)	6.3 (2)	14.2 (2)	3.6 (2)
(Pyrimidine)–(C51–C56)			44.7 (2)	
θ	51.8 (3)	52.7 (2)	65.0 (2)	52.4 (4)
φ	150.0 (4)	156.8 (3)	169.7 (2)	160.1 (5)

Compound (I)

Crystal data

$C_{19}H_{18}ClN_3O$
M_r = 339.81
Triclinic, $P\overline{1}$
a = 8.5280 (12) Å
b = 8.8610 (14) Å
c = 11.7340 (16) Å
α = 100.992 (10)°
β = 93.118 (15)°
γ = 110.524 (9)°
V = 808.1 (2) Å^3
Z = 2
D_x = 1.397 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 120 (2) K
Plate, colourless
0.4 × 0.3 × 0.2 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan, *EVALCCD* (Duisenberg *et al.*, 2003) and *SADABS* (Sheldrick, 2003)
T_{min} = 0.861, T_{max} = 0.952
17119 measured reflections
3562 independent reflections
2542 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.152
S = 1.08
3562 reflections
220 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0677P)^2 + 0.7985P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Compound (II)

Crystal data

$C_{20}H_{21}N_3O_2$
M_r = 335.40
Monoclinic, $P2_1/c$
a = 17.7659 (6) Å
b = 8.5730 (2) Å
c = 11.3982 (3) Å
β = 101.094 (2)°
V = 1703.56 (8) Å^3
Z = 4
D_x = 1.308 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 120 (2) K
Plate, colourless
0.40 × 0.22 × 0.08 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.956, T_{max} = 0.993
14346 measured reflections
3886 independent reflections
2825 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.153
S = 1.07
3886 reflections
231 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0712P)^2 + 0.3779P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.025 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
C23—H23···O24^{i}	0.93	2.46	3.385 (2)	173

Symmetry code: (i) $-x+1, -y+1, -z+2$.

Compound (III)

Crystal data

$C_{24}H_{20}ClN_3O$
M_r = 401.88
Triclinic, $P\overline{1}$
a = 7.6778 (2) Å
b = 8.1298 (3) Å
c = 16.6417 (5) Å
α = 89.080 (2)°
β = 85.123 (2)°
γ = 72.490 (2)°
V = 987.06 (5) Å^3
Z = 2
D_x = 1.352 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 298 (2) K
Plate, colourless
0.45 × 0.40 × 0.10 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.927, T_{max} = 0.979
17614 measured reflections
4513 independent reflections
2799 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.134
S = 1.03
4513 reflections
262 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0639P)^2 + 0.1379P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C53—H53···O6^{i}	0.93	2.48	3.358 (2)	158

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

Compound (IV)

Crystal data

$C_{19}H_{19}N_3O{\cdot}H_2O$
M_r = 323.39
Triclinic, $P\overline{1}$
a = 5.8190 (7) Å
b = 10.4640 (13) Å
c = 13.847 (2) Å
α = 78.447 (12)°
β = 79.647 (9)°
γ = 84.882 (12)°
V = 811.37 (19) Å^3
Z = 2
D_x = 1.324 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 120 (2) K
Plate, colourless
0.4 × 0.3 × 0.2 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan, *EVALCCD* (Duisenberg *et al.*, 2003) and (*SADABS*; Sheldrick, 2003)
T_{min} = 0.956, T_{max} = 0.986
16071 measured reflections
3593 independent reflections
1683 reflections with $I > 2\sigma(I)$
R_{int} = 0.105
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.223
S = 1.04
3593 reflections
220 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1104P)^2 + 0.257P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1W···N4^{i}	0.96	2.00	2.944 (4)	167
O1W—H2W···O6	0.96	1.95	2.879 (3)	163
C5—H5···O6^{i}	0.95	2.58	3.465 (4)	154

Symmetry code: (i) $-x+3, -y+1, -z+1$.

For compound (II), the space group $P2_1/c$ was uniquely assigned from the systematic absences. Crystals of each of (I), (II) and (IV) are triclinic, and in each case the space group $P\overline{1}$ was selected and then confirmed by the structure analysis. All H atoms were located in difference maps. H atoms bonded to C atoms were treated as riding, with C—H distances of 0.95 (aromatic), 0.98 (CH_3) or 0.99 Å (CH_2), and with U_{iso}(H) = 1.2U_{eq}(C), or 1.5U_{eq}(C) for methyl groups. The H atoms of the water molecule in (IV) were permitted to ride at the positions identified from a difference map, giving O—H distances of 0.96 Å, with U_{iso}(H) = 1.5U_{eq}(O).

For all compounds, data collection: *COLLECT* (Nonius, 1999). Cell refinement: *DIRAX/LSQ* (Duisenberg *et al.*, 2000) for (I) and (IV); *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* for (II) and (III). Data reduction: *EVALCCD* (Duisenberg *et al.*, 2003) for (I) and (IV); *DENZO* and *COLLECT* for (II) and (III). Program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999) and *WinGX* (Farrugia, 1999) for (I) and (IV); *SIR2004* (Burla *et al.*, 2005) and *WinGX* for (II) and (III). For all compounds, program(s) used to refine structure: *OSCAIL* (McArdle, 2003) and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK3026). Services for accessing these data are described at the back of the journal.

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