

2,3-Dimethoxybenzaldehyde isonicotinoylhydrazone chloroform monosolvate, and the mono- and dihydrates of 3,4,5-trimethoxybenzaldehyde isonicotinoylhydrazone: hydrogen-bonded supramolecular structures in one, two and three dimensions

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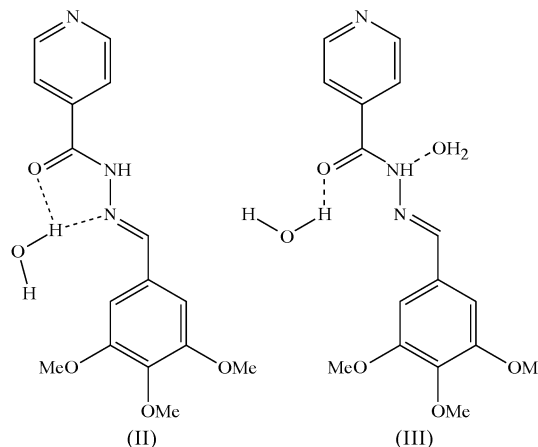
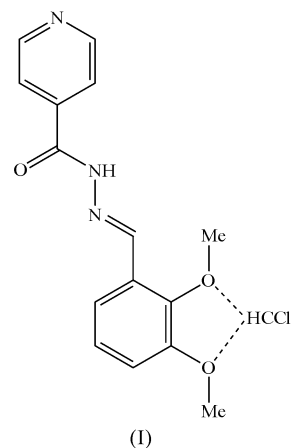
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In 2,3-dimethoxybenzaldehyde isonicotinoylhydrazone chloroform monosolvate, $C_{15}H_{15}N_3O_3 \cdot CHCl_3$, the hydrazone molecules are linked by a combination of $N-H \cdots N$ and $C-H \cdots N$ hydrogen bonds into chains from which the chloroform molecules are pendent. 3,4,5-Trimethoxybenzaldehyde isonicotinoylhydrazone forms two stoichiometric hydrates. In the monohydrate, $C_{16}H_{17}N_3O_4 \cdot H_2O$, the components are linked into sheets by a combination of $O-H \cdots O$, $O-H \cdots N$ and $N-H \cdots N$ hydrogen bonds, and in the dihydrate, $C_{16}H_{17}N_3O_4 \cdot 2H_2O$, a combination of $O-H \cdots O$, $O-H \cdots N$ and $N-H \cdots O$ hydrogen bonds links the components into a three-dimensional framework structure.

Comment

We report here the molecular and supramolecular structures of three methoxy-substituted benzaldehyde isonicotinoylhydrazones, namely 2,3-dimethoxybenzaldehyde isonicotinoylhydrazone which crystallizes as a chloroform monosolvate, (I), and the mono- and dihydrates of 3,4,5-trimethoxybenzaldehyde isonicotinoylhydrazone, (II) and (III), respectively (Figs. 1–3). We have undertaken this work as part of a more general study of isonicotinoylhydrazones (Wardell, de Souza, Ferreira *et al.*, 2005; Wardell, de Souza, Wardell *et al.*, 2005; Wardell *et al.*, 2006; Low *et al.*, 2006). The structure of the

dihydrate, (III), has been reported previously (Bhagiratha *et al.*, 2000), determined from diffraction data collected at ambient temperature, but the description of the supramolecular aggregation differs markedly from that deduced here.



In the organic components in each of compounds (I)–(III), the central spacer unit between atoms C14 and C21 (Figs. 1–3) is effectively planar, with an all-*trans* chain-extended conformation, as shown by the relevant torsion angles (Table 1). The two independent rings are only slightly twisted out of the plane of the central spacer unit, although with no evident pattern in the torsion angles defining the ring orientations. Methoxy atom C231 in (I), and the corresponding atoms C231 and C251 in the two hydrates (II) and (III), are all almost coplanar with the adjacent aryl rings, whereas the C/O/C planes containing atoms C221 in (I) and C241 in (II) and (III) are almost orthogonal to the planes of the adjacent rings. In general, isolated methoxy groups bonded to aryl rings exhibit effective coplanarity with the aryl ring (Seip & Seip, 1973; Ferguson *et al.*, 1996; Gallagher *et al.*, 2001, 2004). Thus, the methoxy groups in compounds (I)–(III), where the C/O/C planes are approximately normal to the planes of the adjacent aryl rings, are anomalous. If the two methoxy groups in compound (I) were both approximately coplanar with the aryl ring, with atoms C221 and C231 maximally distant from one another to minimize close repulsive $H \cdots O$ contacts involving the H atoms of one substituent and the O atom of the other,

there would be close repulsive contacts between the H atoms bonded to atoms C221 and C27, hence the orthogonal conformation of the methoxy group at C22. Rather similar considerations govern the conformations adopted by the 4-methoxy groups in each of (II) and (III). As usual, the two exocyclic C—C—O angles for the orthogonal methoxy groups are very similar, whereas these two angles consistently differ by 8–10° for the coplanar methoxy groups. The organic components of hydrates (II) and (III) show no significant differences, apart from minor differences in conformation (Table 1).

The supramolecular aggregation in compound (I) is extremely simple, even though the structure contains three types of hydrogen bond (Table 2). Atoms N17 and C27 in the hydrazone molecule at (x, y, z) both act as hydrogen-bond donors to pyridyl atom N11 of the hydrazone molecule at $(\frac{1}{2} - x, -\frac{1}{2} + y,$

$\frac{3}{2} - z)$, so forming a $C(7)C(9)[R_2^1(6)]$ (Bernstein *et al.*, 1995) chain of rings running parallel to the [010] direction and generated by the 2_1 screw axis along $(\frac{1}{4}, y, \frac{3}{4})$ (Fig. 4). The chloroform molecules are pendent from the chain, to which they are weakly linked *via* a planar three-centre C—H...O₂ hydrogen bond (Fig. 1 and Table 2). Two chains of this type, related to one another by inversion, pass through each unit cell, but there are no direction-specific interactions between the chains, so that the supramolecular structure is strictly one-dimensional.

Within the selected asymmetric unit of compound (II), the water molecule is linked to the hydrazone component *via* a three-centre O—H...N,O hydrogen bond (Fig. 2 and Table 3). Two further hydrogen bonds, one each of O—H...O and N—H...N types, link these bimolecular aggregates into sheets. Water atom O2 at (x, y, z) acts as hydrogen-bond donor to atom N27 and methoxy atom O24 at $(-x, -\frac{1}{2} + y, \frac{1}{2} - z)$, so

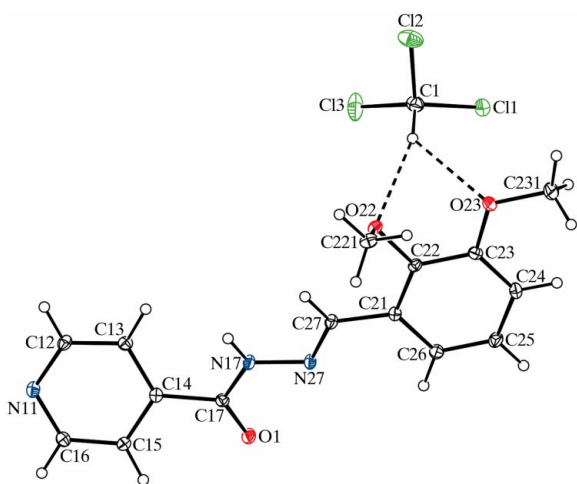


Figure 1
The independent molecular components of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

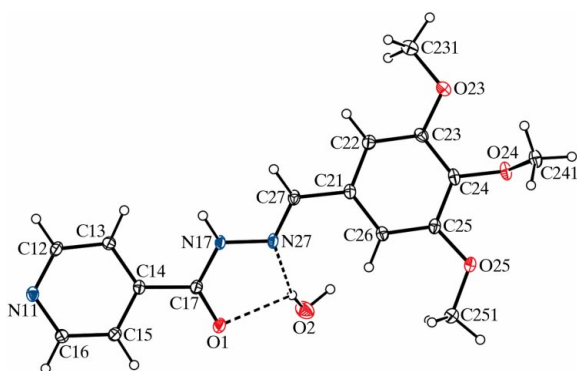


Figure 2
The independent molecular components of compound (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

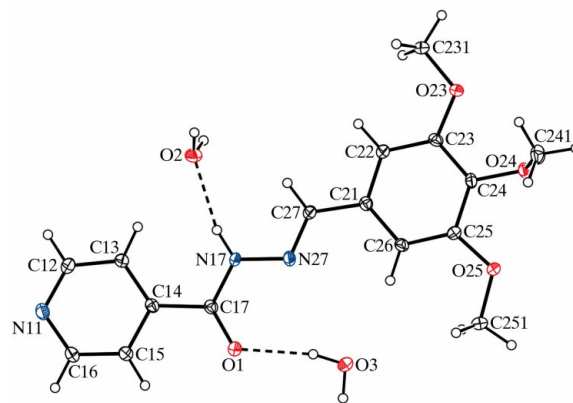


Figure 3
The independent molecular components of compound (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

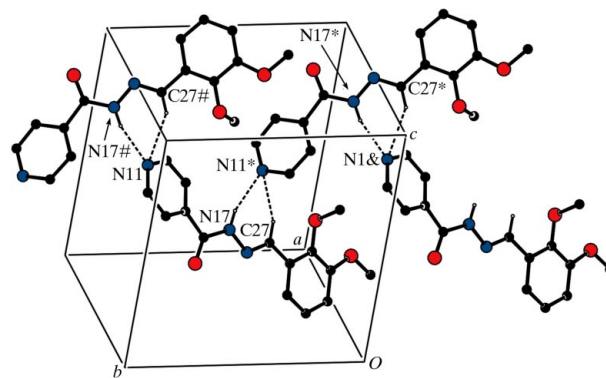


Figure 4
Part of the crystal structure of compound (I), showing the formation of a hydrogen-bonded chain of rings along [010]. For the sake of clarity, the chloroform molecules and H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*), a hash (#) or an ampersand (&) are at the symmetry positions $(\frac{1}{2} - x, -\frac{1}{2} + y, \frac{3}{2} - z)$, $(\frac{1}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z)$ and $(x, -1 + y, z)$, respectively.

forming a $C_2^2(12)$ chain running parallel to the $[010]$ direction and generated by the 2_1 screw axis along $(0, y, \frac{1}{4})$. In addition, amino atom N17 at (x, y, z) acts as hydrogen-bond donor to pyridyl atom N11 at $(1 - x, \frac{1}{2} + y, \frac{3}{2} - z)$, so forming a second motif running parallel to the $[010]$ direction, this time of $C(7)$ type and generated by the 2_1 screw axis along $(\frac{1}{2}, y, \frac{3}{4})$. The combination of these two chain motifs generates a sheet of $R_1^2(5)$ and $R_6^0(35)$ rings parallel to $(10\bar{1})$ (Fig. 5). Two inversion-related sheets pass through each unit cell, but there are no direction-specific interactions between adjacent sheets, so that the supramolecular structure is strictly two-dimensional.

The asymmetric unit of the dihydrate compound, (III), has been selected such that the components are joined by one $O-H\cdots O$ hydrogen bond and one $N-H\cdots O$ hydrogen bond (Fig. 3 and Table 4). These three-component aggregates are linked by two-centre $O-H\cdots N$ and $O-H\cdots O$ hydrogen bonds and by a three-centre $O-H\cdots(O)_2$ hydrogen bond to form a three-dimensional framework, whose formation is readily analysed in terms of simple substructures of low dimensionality. In the first such substructure, water atom O2 at (x, y, z) acts as hydrogen-bond donor, *via* atom H2A, to

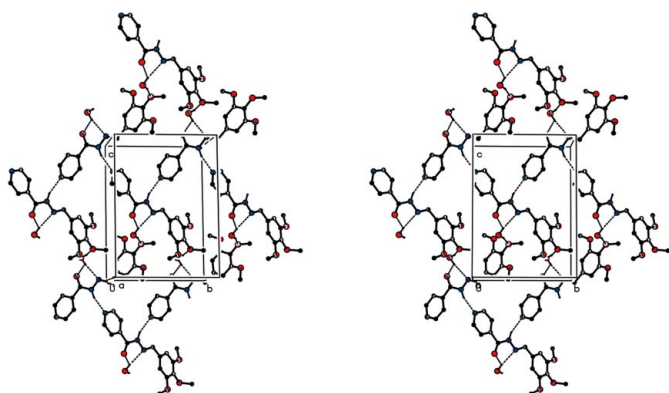


Figure 5
A stereoview of part of the crystal structure of compound (II), showing the formation of a hydrogen-bonded sheet of $R_1^2(5)$ and $R_6^0(35)$ rings parallel to $(10\bar{1})$. For the sake of clarity, H atoms bonded to C atoms have been omitted.

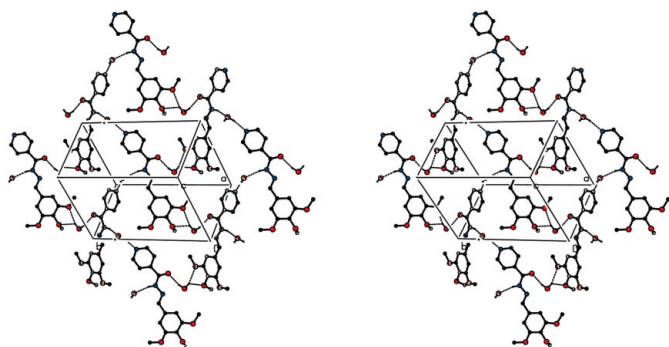


Figure 6
A stereoview of part of the crystal structure of compound (III), showing the formation of a hydrogen-bonded sheet of $R_1^2(5)$ and $R_6^0(40)$ rings parallel to $(10\bar{1})$. For the sake of clarity, H atoms bonded to C atoms have been omitted.

pyridyl atom N11 at $(-\frac{1}{2} + x, \frac{1}{2} - y, -\frac{1}{2} + z)$, so forming a $C_2^2(9)$ chain running parallel to the $[101]$ direction and generated by the n -glide plane at $y = \frac{3}{4}$. In the second substructure, water atom O3 at (x, y, z) acts as hydrogen-bond donor, *via* atom H3B, to methoxy atoms O24 and O25, both at $(\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z)$, thus forming a $C_2^2(12)C_2^2(13)[R_1^2(5)]$ chain of rings, again parallel to the $[101]$ direction but now generated by the n -glide plane at $y = \frac{1}{4}$. The combination of these two substructures then generates a sheet parallel to $(10\bar{1})$ (Fig. 6).

Two sheets of this type, related to one another by inversion, pass through each unit cell, and adjacent sheets are linked by

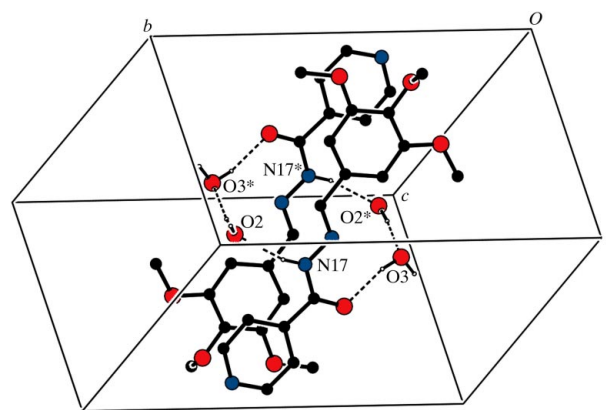


Figure 7
Part of the crystal structure of compound (III), showing the formation of the centrosymmetric $R_6^0(16)$ motif linking the $(10\bar{1})$ sheets. For the sake of clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(1 - x, 1 - y, 1 - z)$.

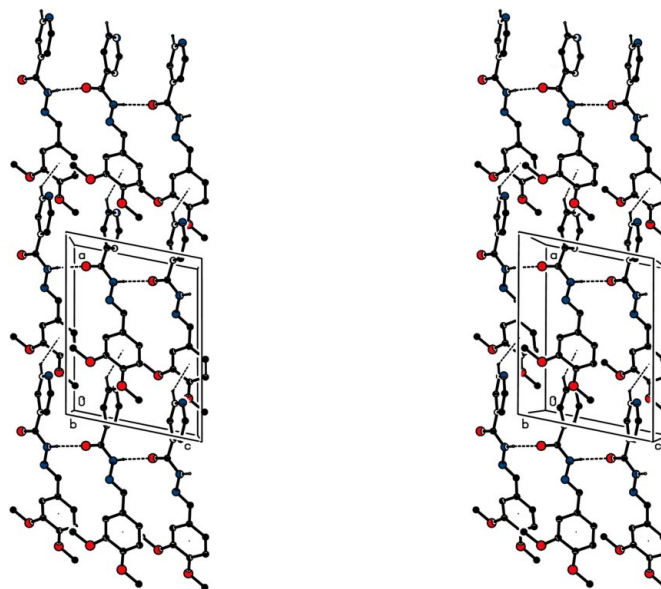


Figure 8
A stereoview of part of the crystal structure of compound (IV), showing the formation of a sheet parallel to (010) built from $N-H\cdots O$ and $C-H\cdots\pi(\text{arene})$ hydrogen bonds. The original atomic coordinates (Chen *et al.*, 1997) have been used. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

the final substructural motif. Water atom O2 at (x, y, z) acts as hydrogen-bond donor, *via* atom H2B, to water atom O3 at $(1 - x, 1 - y, 1 - z)$, so generating by inversion an $R_6^6(16)$ motif (Fig. 7). Propagation of this motif by the space group links each $(10\bar{1})$ sheet to the two neighbouring sheets, so linking all of the molecular components into a single three-dimensional framework of considerable complexity.

The structure deduced here for dihydrate (III) differs markedly from that recently reported for this compound at ambient temperature, where the structure was described in terms of sheets parallel to (010) formed by O—H...O and O—H...N hydrogen bonds (Bhagiratha *et al.*, 2000). Although the unit-cell dimensions, space group and atomic coordinates reported earlier show that there has been no phase change between ambient temperature and 120 K, we find no combination of any subset of the hydrogen bonds present which can generate a sheet parallel to (010). In any event, as noted above, the supramolecular structure of (III) is three-dimensional, not two-dimensional.

It is of interest at this point briefly to compare the structures of compounds (I) and the isomeric solvent-free hydrazone (IV), which was crystallized from acetonitrile (Chen *et al.*, 1997). In both methoxy groups of compound (IV), the methyl C atoms are essentially coplanar with the adjacent aryl ring. The supramolecular structure of (IV) was described in terms of simple chains formed by a single N—H...O hydrogen bond (Chen *et al.*, 1997). In fact, these chains, which are of $C(4)$ type, are linked into sheets by a C—H... π (arene) hydrogen bond, although this interaction was not mentioned in the original report. The parameters are $H1...Cg^i = 2.88 \text{ \AA}$, $C1...Cg^i = 3.675(3) \text{ \AA}$ and $C1-H1...Cg^i = 144^\circ$, where the original atom numbers have been used and Cg represents the centroid of the aryl ring [symmetry code: (i) $1 + x, y, z$]. The resulting supramolecular structure of (IV) then takes the form of sheets parallel to (010) (Fig. 8).

Experimental

Equimolar quantities (1 mmol) of the appropriate arylaldehyde [2,3-dimethoxybenzaldehyde for the synthesis of (I), and 3,4,5-trimethoxybenzaldehyde for (II) and (III)] and isonicotinoylhydrazine were dissolved, respectively, in ethanol (10 ml) and water (10 ml). These solutions were mixed and each mixture was stirred at room temperature until reaction was complete, as shown by thin-layer chromatography. Each reaction mixture was concentrated under reduced pressure. The residues were washed successively with cold ethanol and diethyl ether, and then recrystallized from ethanol. Analysis for 2,3-dimethoxybenzaldehyde isonicotinoylhydrazine: yield 90%, m.p. 413–414 K; $^1\text{H NMR}$ (DMSO- d_6): δ 12.08 (*s*, 1H, NH), 8.78 (*d*, 2H, $J = 6.0$ Hz, H12 and H16), 8.75 (*s*, 1H, H27), 7.85 (*d*, 2H, $J = 6.0$ Hz, H13 and H15), 7.48 (*dd*, 1H, $J = 6.5$ and 3.5 Hz, H24), 7.16–7.14 (*m*, 2H, H25 and H26), 3.83 (*s*, 3H, OCH₃), 3.80 (*s*, 3H, OCH₃); IR (KBr disc, ν , cm^{-1}): 1671 (CO). Crystals grown from solution in ethanol were found to be unsuitable for single-crystal X-ray diffraction, as were crystals obtained by slow recrystallization from both methanol and acetonitrile. Crystals obtained by slow evaporation of a solution in chloroform, *viz.* the chloroform monosolvate (I), were found to be suitable for single-crystal X-ray diffraction. Analysis for 3,4,5-trimethoxybenzaldehyde isonicoti-

noylhydrazine: yield 88%; $^1\text{H NMR}$ (DMSO- d_6): δ 12.06 (*s*, 1H, NH), 8.79 (*d*, 2H, $J = 5.5$ Hz, H12 and H16), 8.40 (*s*, 1H, H27), 7.84 (*d*, 2H, $J = 5.5$ Hz, H13 and H15), 7.81 (*s*, 2H, H22 and H26), 3.84 (*s*, 6H, 2 \times OCH₃), 3.17 (*s*, 3H, OCH₃). Recrystallization from ethanol gave the dihydrate, (III): m.p. 466–468 K; IR (KBr disc, ν , cm^{-1}) 1664 (CO). Further recrystallization from chloroform–propan-2-ol (1:1 *v/v*) produced the monohydrate, (II): IR (KBr disc, ν , cm^{-1}) 1664 (CO).

Compound (I)

Crystal data

$C_{15}H_{15}N_3O_3 \cdot CHCl_3$	$Z = 4$
$M_r = 404.67$	$D_x = 1.492 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 12.7400(4) \text{ \AA}$	$\mu = 0.53 \text{ mm}^{-1}$
$b = 10.8595(3) \text{ \AA}$	$T = 120(2) \text{ K}$
$c = 13.9187(4) \text{ \AA}$	Plate, colourless
$\beta = 110.7280(10)^\circ$	$0.18 \times 0.13 \times 0.03 \text{ mm}$
$V = 1801.01(9) \text{ \AA}^3$	

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer	20128 measured reflections
φ and ω scans	4128 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	3388 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.932, T_{\max} = 0.984$	$R_{\text{int}} = 0.047$
	$\theta_{\max} = 27.5^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.02P)^2 + 1.8708P]$
$R[F^2 > 2\sigma(F^2)] = 0.041$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.090$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 1.02$	$\Delta\rho_{\max} = 0.40 \text{ e \AA}^{-3}$
4128 reflections	$\Delta\rho_{\min} = -0.60 \text{ e \AA}^{-3}$
228 parameters	
H-atom parameters constrained	

Table 1

Selected torsion and bond angles ($^\circ$) for compounds (I)–(III).

	(I)	(II)	(III)
C13–C14–C17–N17	27.7 (2)	18.5 (2)	7.9 (3)
C14–C17–N17–N27	–176.86 (15)	–177.52 (14)	178.43 (17)
C17–N17–N27–C27	175.15 (16)	176.37 (16)	174.09 (19)
N17–N27–C27–C21	–178.11 (16)	–178.87 (14)	177.30 (19)
N27–C27–C21–C22	172.79 (17)	159.37 (17)	–177.6 (2)
C21–C22–O22	120.19 (16)		
C23–C22–O22	118.71 (16)		
C21–C22–O22–C221	98.85 (19)		
C22–C23–O23	115.64 (16)	124.14 (16)	124.62 (19)
C24–C23–O23	125.14 (17)	115.97 (14)	116.02 (18)
C22–C23–O23–C231	179.89 (18)	–12.3 (2)	–2.9 (3)
C23–C24–O24		120.76 (15)	120.48 (18)
C25–C24–O24		118.85 (15)	118.96 (19)
C23–C24–O24–C241		87.82 (19)	81.1 (2)
C24–C25–O25		115.24 (14)	114.09 (18)
C26–C25–O25		124.59 (15)	125.19 (19)
C26–C25–O25–C251		8.2 (2)	3.1 (3)

Table 2

Hydrogen-bond geometry ($\text{\AA}, ^\circ$) for (I).

$D-H...A$	$D-H$	$H...A$	$D...A$	$D-H...A$
N17–H17...N11 ⁱ	0.88	2.17	3.015 (2)	162
C1–H1...O22	1.00	2.41	3.363 (2)	159
C1–H1...O23	1.00	2.59	3.371 (3)	134
C27–H27...N11 ⁱ	0.95	2.60	3.411 (2)	144

Symmetry code: (i) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$.

Compound (II)

Crystal data

C₁₆H₁₇N₃O₄·H₂O
M_r = 333.34
 Monoclinic, *P*₂₁/*c*
a = 10.8081 (6) Å
b = 10.3597 (3) Å
c = 14.4270 (7) Å
 β = 95.281 (2)°
V = 1608.52 (13) Å³

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
T_{min} = 0.967, *T_{max}* = 0.995

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.047
wR [*F*²] = 0.133
S = 1.03
 3686 reflections
 220 parameters
 H-atom parameters constrained

Table 3

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

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
N17–H17...N11 ⁱ	0.88	2.12	2.947 (2)	157
O2–H2A...O1	0.90	2.41	3.015 (2)	125
O2–H2A...N27	0.90	2.32	3.206 (2)	166
O2–H2B...O24 ⁱⁱ	0.90	1.95	2.823 (2)	162

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

Compound (III)

Crystal data

C₁₆H₁₇N₃O₄·2H₂O
M_r = 351.36
 Monoclinic, *P*₂₁/*n*
a = 8.9930 (5) Å
b = 16.2713 (10) Å
c = 11.6311 (6) Å
 β = 90.354 (3)°
V = 1701.92 (17) Å³

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
T_{min} = 0.966, *T_{max}* = 0.999

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.065
wR [*F*²] = 0.121
S = 1.01
 3890 reflections
 229 parameters
 H-atom parameters constrained

For compounds (I) and (III), the space group *P*₂₁/*n* was uniquely assigned from the systematic absences, and space group *P*₂₁/*c* was similarly assigned for compound (II). All H atoms were located in difference maps and then treated as riding atoms, with C–H = 0.95

Table 4

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

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
N17–H17...O2	0.88	2.02	2.871 (2)	163
O2–H2A...N11 ⁱ	0.87	1.99	2.843 (2)	166
O2–H2B...O3 ⁱⁱ	0.87	1.88	2.745 (2)	174
O3–H3A...O1	0.87	2.01	2.864 (2)	166
O3–H3B...O24 ⁱⁱⁱ	0.87	2.24	2.874 (2)	130
O3–H3B...O25 ⁱⁱⁱ	0.87	2.33	3.157 (2)	159

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

(aromatic and –CH=N–), 0.98 (methyl) and 1.00 Å (aliphatic CH), N–H = 0.88 Å and O–H = 0.87–0.90 Å, and with *U*_{iso}(H) = *kU*_{eq}(C,N,O), where *k* = 1.5 for the methyl groups and the water molecules or 1.2 for all other H atoms.

For all compounds, data collection: COLLECT (Nonius, 1999); cell refinement: DENZO (Otwinowski & Minor, 1997) and COLLECT; data reduction: DENZO and COLLECT; program(s) used to solve structure: OSCAIL (McArdle, 2003) and SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: OSCAIL 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: GG3058). Services for accessing these data are described at the back of the journal.

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