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π-Stacked hydrogen-bonded dimers in 2-(2-nitrophenylaminocarbonyl)-benzoic acid, and hydrogen-bonded sheets in orthorhombic and monoclinic polymorphs of 2-(4-nitrophenylaminocarbonyl)benzoic acid

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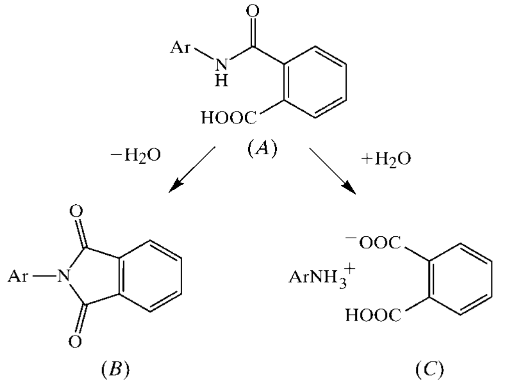
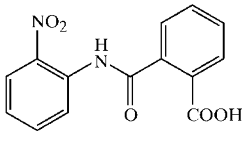
Molecules of 2-(2-nitrophenylaminocarbonyl)benzoic acid, $C_{14}H_{10}N_2O_5$, are linked into centrosymmetric $R_2^2(8)$ dimers by a single O—H···O hydrogen bond [H···O = 1.78 Å, O···O = 2.623 (2) Å and O—H···O = 178°] and these dimers are linked into sheets by a single aromatic π–π stacking interaction. The isomeric compound 2-(4-nitrophenylaminocarbonyl)benzoic acid crystallizes in two polymorphic forms. In the orthorhombic form (space group $P2_12_12_1$ with Z' = 1, crystallized from ethanol), the molecules are linked into sheets of $R_4^4(22)$ rings by a combination of one N—H···O hydrogen bond [H···O = 1.96 Å, N···O = 2.833 (3) Å and N—H···O = 171°] and one O—H···O hydrogen bond [H···O = 1.78 Å, O···O = 2.614 (3) Å and O—H···O = 173°]. In the monoclinic form (space group $P2_1/n$ with Z' = 2, crystallized from acetone), the molecules are linked by a combination of two N—H···O hydrogen bonds [H···O = 2.09 and 2.16 Å, N···O = 2.873 (4) and 2.902 (3) Å, and N—H···O = 147 and 141°] and two O—H···O hydrogen bonds [H···O = 1.84 and 1.83 Å, O···O = 2.664 (3) and 2.666 (3) Å, and O—H···O = 166 and 174°] into sheets of some complexity. These sheets are linked into a three-dimensional framework by a single C—H···O hydrogen bond [H···O = 2.45 Å, C···O = 3.355 (4) Å and C—H···O = 160°].

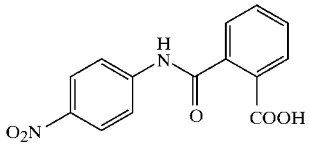
Comment

The reaction of C-substituted anilines with phthalic anhydride initially yields *N*-arylaminocarbonylbenzoic acids, (*A*) (see scheme). Dehydration of this type of intermediate yields *N*-arylphthalimides, (*B*), while hydrolysis yields arylammonium hydrogenphthalates, (*C*). We have recently reported the supramolecular structure of a type (*C*) salt, 4-iodoanilinium 3-nitrophthalate(1−) (Glidewell *et al.*, 2003), and we report here the molecular and supramolecular structures of two examples of the type (*A*) intermediate, namely 2-(2-nitrophenylaminocarbonyl)benzoic acid, (I) (Fig. 1), and two polymorphs of 2-(4-nitrophenylaminocarbonyl)benzoic acid, *viz.* orthorhombic, (II) (Fig. 2), and monoclinic, (III) (Fig. 3).





(I)



(II) orthorhombic
(III) monoclinic

In each of compounds (I)–(III), the central C11—C17(O17)—N1—C21 fragment of the molecule is essentially planar, with a *trans* amide conformation. The C11–C16 and C21–C26 rings are considerably rotated away from this plane, as indicated by the key torsion angles (Tables 1, 3 and 5). In (I), the carboxyl and nitro groups make dihedral angles of 29.9 (2) and 36.5 (2)°, respectively, with the adjacent aryl rings, and in (II), the corresponding angles are 12.2 (2) and 11.1 (2)°, respectively. In (III), these dihedral angles are 7.7 (2) and 13.0 (2)°, respectively, in molecule *A*, and 10.1 (2) and 6.5 (1)° in molecule *B*. The C—O distances in the carboxyl groups are consistent with the location of the fully ordered carboxyl H atoms, as identified from difference maps. The other bond distances and angles show no exceptional features.

In compound (I) (Fig. 1), there is an intramolecular N—H···O hydrogen bond forming an *S*(6) motif (Bernstein *et al.*, 1995). The supramolecular aggregation is dominated by a single O—H···O hydrogen bond, propagated by aromatic π–π stacking interactions. Carboxyl atom O11 in the molecule at (*x*, *y*, *z*) acts as hydrogen-bond donor to carboxyl atom O12 in the molecule at (1 − *x*, 1 − *y*, 1 − *z*), so generating a centrosymmetric dimer characterized by the usual $R_2^2(8)$ motif and centred at ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$) (Fig. 4).

The π–π stacking interaction involves the nitro-substituted ring of the molecule at (*x*, *y*, *z*), which is part of the dimer

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centred at ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$), and the corresponding rings of the molecules at ($\frac{1}{2}+x$, y, $\frac{3}{2}-z$) and ($x-\frac{1}{2}$, y, $\frac{3}{2}-z$), which themselves are components of the dimers centred at (1, $\frac{1}{2}$, 1) and (0, $\frac{1}{2}$, 1), respectively. The interplanar angle between adjacent rings of this type is *ca* 9.5°, with a centroid separation of 3.793 (2) Å and an interplanar spacing of *ca* 3.42 Å. Propagation by inversion of this interaction also links the ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$) dimer to those centred at (0, $\frac{1}{2}$, 0) and (1, $\frac{1}{2}$, 0), and hence the $R_2^2(8)$ dimers are linked into an (010) sheet (Fig. 5).

Whereas the supramolecular structure of (I) is controlled by a combination of O—H···O hydrogen bonds and aromatic π–π stacking interactions, with the N—H bond participating only in an intramolecular hydrogen bond, the supramolecular aggregation in polymorphic compounds (II) and (III) is controlled by a combination of N—H···O and O—H···O hydrogen bonds. While π–π stacking interactions and X—H···π(arene) hydrogen bonds (X is C, N or O) are all absent from the structures of (II) and (III), there are two C—H···O hydrogen bonds present in (III).

In compound (II), the amidic atom N1 in the molecule at (x, y, z) acts as hydrogen-bond donor to carboxyl atom O12 in the molecule at ($x-1$, y, z), so generating by translation a $C(7)$ chain running parallel to the [100] direction. At the same time, carboxyl atom O11 at (x, y, z) acts as hydrogen-bond donor to amidic atom O17 in the molecule at ($1-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$), so producing a spiral $C(7)$ chain running parallel to the [010] direction and generated by the 2_1 screw axis along ($\frac{1}{2}$, y, $\frac{1}{4}$). It is noteworthy that the carboxyl group in (II) forms neither of the motifs so characteristic of simple carboxylic acids, namely the $R_2^2(8)$ dimer [*cf.* compound (I)] and the $C(4)$ chain. Likewise, the amide group in (II) does not form the $C(4)$ chain often found in simple carboxylic amides, but instead each of the two hydrogen bonds in (II) utilizes a donor and an acceptor from

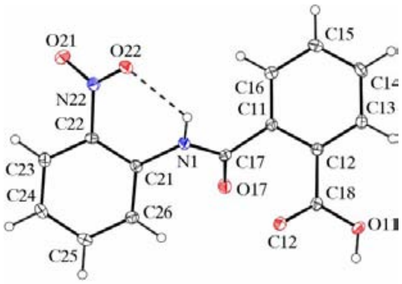

Figure 1
The molecule of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

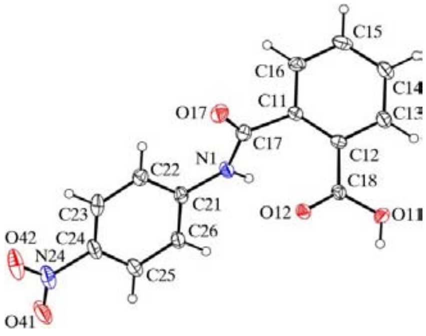

Figure 2
The molecule of compound (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

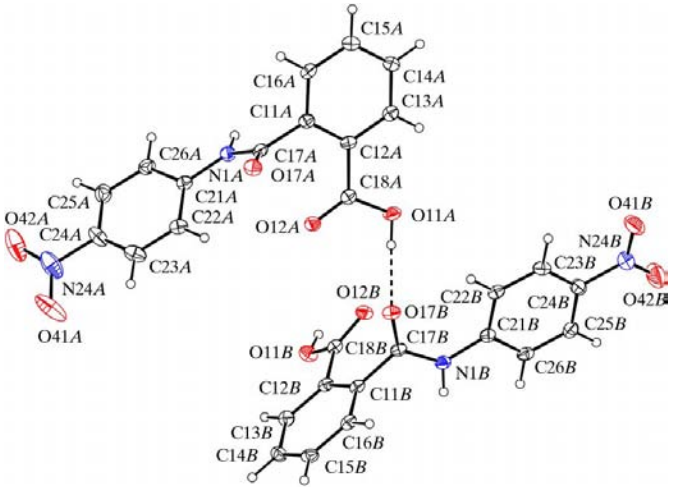

Figure 3
The two independent molecules in compound (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

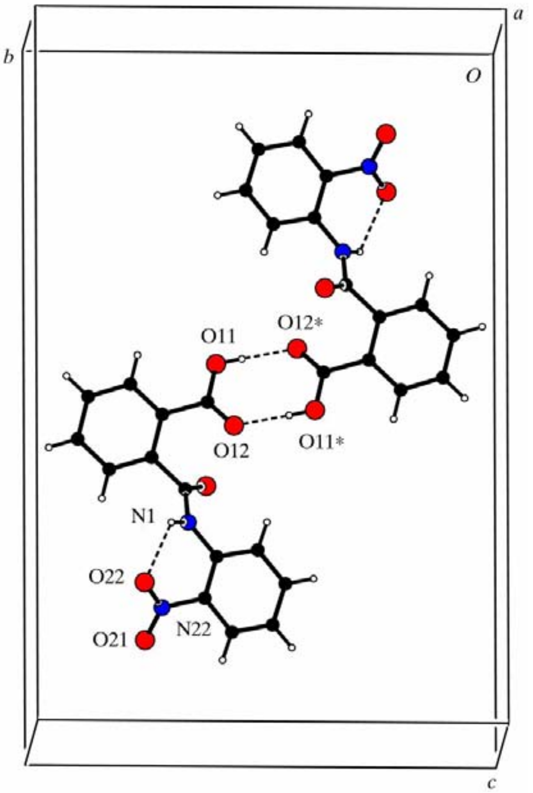

Figure 4
Part of the crystal structure of (I), showing the formation of a centrosymmetric $R_2^2(8)$ dimer. Atoms marked with an asterisk (*) are at the symmetry position ($1-x$, $1-y$, $1-z$).

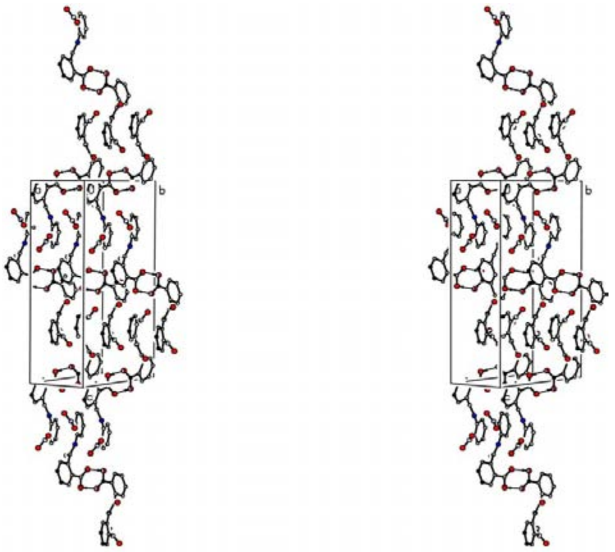

Figure 5
Stereoview of part of the crystal structure of (I), showing the linking of the hydrogen-bonded dimers into an (010) sheet by means of aromatic π–π stacking interactions. For the sake of clarity, H atoms bonded to C or N atoms have been omitted.

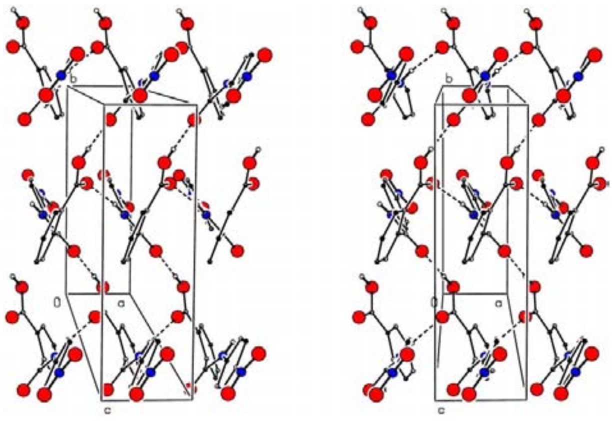

Figure 6
Stereoview of part of the crystal structure of (II), showing the formation of an (001) sheet of $R_4^4(22)$ rings. For the sake of clarity, H atoms bonded to C atoms have been omitted.

the different functional groups (acid and amide) within the molecule.

The combination of the two $C(7)$ chains in (II) generates an (001) sheet in the form of a hydrogen-bonded (4,4)-net (Batten & Robson, 1998) built from a single type of $R_4^4(22)$ ring (Fig. 6). This sheet lies in the domain $-0.03 < z < 0.53$ and a second such sheet lies in the domain $0.47 < z < 1.03$, but there are no direction-specific interactions between adjacent sheets.

The supramolecular structure of the monoclinic form, (III), consists of sheets generated by a combination of O—H$\cdots$O and N—H$\cdots$O hydrogen bonds, linked into a three-dimensional framework by a single rather strong C—H$\cdots$O hydrogen bond (Table 6). Carboxyl atom O11*A* acts as hydrogen-bond donor to amidic atom O17*B* within the asymmetric unit (Fig. 3) and, in a similar fashion, carboxyl atom O11*B* in the type *B* molecule at (x, y, z) acts as hydrogen-bond donor to amidic atom O17*A* in the type *A* molecule at (x, $1 + y$, z). These two hydrogen bonds thus generate by translation a $C_2^2(14)$ chain running parallel to the [010] direction (Fig. 7). These translational chains are then linked into (001) sheets by the two N—H$\cdots$O hydrogen bonds.

Amino atom N1*A* in the type *A* molecule at (x, y, z) acts as hydrogen-bond donor to carboxyl atom O12*B* in the type *B* molecule at ($\frac{1}{2} - x$, $y - \frac{1}{2}$, $\frac{1}{2} - z$), so producing a second $C_2^2(14)$

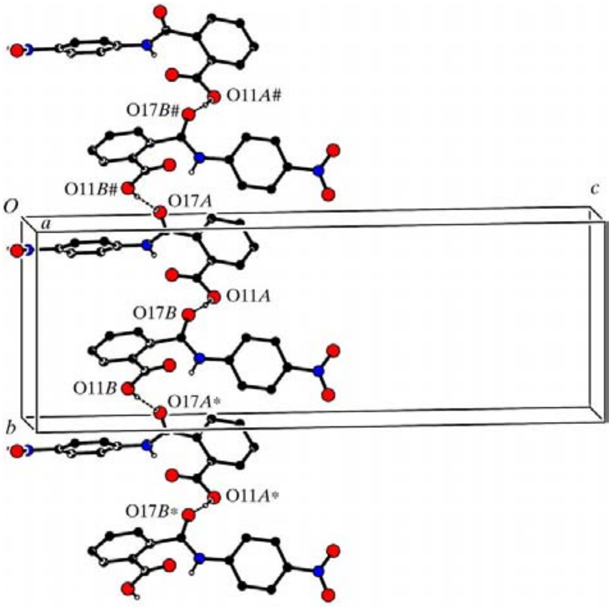

Figure 7
Part of the crystal structure of (III), showing the $C_2^2(14)$ chain generated by translation. For the sake of clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*) or hash (#) are at the symmetry positions (x, $1 + y$, z) and (x, $y - 1$, z), respectively.

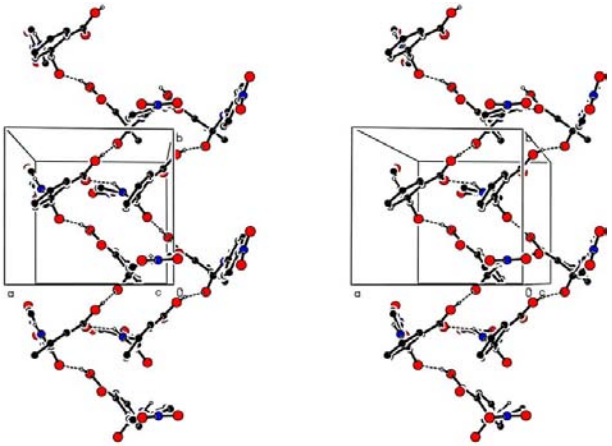

Figure 8
Stereoview of part of the crystal structure of (III), showing the $C_2^2(14)$ chains generated by translation and by the 2_1 screw axis along ($\frac{1}{4}$, y, $\frac{1}{4}$). For the sake of clarity, H atoms bonded to C atoms have been omitted.

chain parallel to [010], this time generated by the 2_1 screw axis along ($\frac{1}{4}$, y, $\frac{1}{4}$) (Fig. 8). Similarly, amino atom N1*B* at (x, y, z) acts as hydrogen-bond donor to carboxyl atom O12*A* at ($\frac{3}{2} - x$, $\frac{1}{2} + y$, $\frac{1}{2} - z$), so producing a third $C_2^2(14)$ chain, this time generated by the 2_1 screw axis along ($\frac{3}{4}$, y, $\frac{1}{4}$).

The combination of these three $C_2^2(14)$ chains generates an (001) sheet lying in the domain $-0.02 < z < 0.52$, and a second such sheet, related to the first by inversion, lies in the domain $0.48 < z < 1.02$. The sheet is reinforced by a C—H···O hydrogen bond (Table 6) and adjacent sheets are linked by a second C—H···O hydrogen bond. Atom C23*B* in the type *B* molecule at (x, y, z), which lies in the $-0.02 < z < 0.52$ sheet, acts as hydrogen-bond donor to nitro atom O41*A* in the type *A* molecule at ($\frac{1}{2} + x$, $\frac{1}{2} - y$, $\frac{1}{2} + z$), which forms part of the sheet in the domain $0.48 < z < 1.02$. Propagation of this interaction by the space group then generates a single three-dimensional framework.

Experimental

Samples of both acids were prepared by heating under reflux chloroform solutions containing equimolar mixtures of phthalic anhydride and the appropriate nitroaniline. The reactions were monitored by thin-layer chromatography and, when complete, the mixtures were cooled and the solvent was removed. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of solutions in ethanol for (I) and (II), and in acetone for (III). Melting points: for (I), 426–428 K; for (II), 475–477 K, with liberation of water, followed rapidly by re-solidification on formation of *N*-(4-nitrophenyl)phthalimide and subsequent re-melting at 528–531 K; for (III), 471–473 K, with liberation of water, followed rapidly by re-solidification as for (II).

Compound (I)

Crystal data

$C_{14}H_{10}N_2O_5$
$M_r = 286.24$
Orthorhombic, *Pbca*
$a = 7.2761$ (2) Å
$b = 14.8881$ (5) Å
$c = 23.0407$ (6) Å
$V = 2495.94$ (13) Å^3
$Z = 8$
$D_x = 1.523$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 2848 reflections
$\theta = 3.2$–27.5°
$\mu = 0.12$ mm^{-1}
$T = 120$ (2) K
Block, colourless
0.36 × 0.36 × 0.22 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995, 1997)
$T_{min} = 0.955$, $T_{max} = 0.975$
15 224 measured reflections
2848 independent reflections
1876 reflections with $I > 2\sigma(I)$
$R_{int} = 0.072$
$\theta_{max} = 27.5°$
$h = -7 \rightarrow 9$
$k = -19 \rightarrow 19$
$l = -27 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.115$
$S = 0.98$
2848 reflections
191 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0595P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

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

C18—O11	1.312 (2)	C18—O12	1.227 (2)
C12—C11—C17—N1	127.5 (2)	C11—C12—C18—O11	153.6 (2)
C11—C17—N1—C21	177.8 (2)	C21—C22—N22—O21	143.3 (2)
C17—N1—C21—C22	−139.1 (2)		

Table 2
Hydrogen-bonding geometry (Å, °) for (I).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O22	0.88	2.14	2.719 (2)	123
O11—H11···O12^{i}	0.84	1.78	2.623 (2)	178

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

Compound (II)

Crystal data

$C_{14}H_{10}N_2O_5$
$M_r = 286.24$
Orthorhombic, $P2_12_12_1$
$a = 3.9188$ (2) Å
$b = 12.6896$ (6) Å
$c = 27.7029$ (16) Å
$V = 1377.61$ (12) Å^3
$Z = 4$
$D_x = 1.380$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 1820 reflections
$\theta = 3.2$–27.5°
$\mu = 0.11$ mm^{-1}
$T = 120$ (2) K
Needle, colourless
0.25 × 0.08 × 0.04 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995, 1997)
$T_{min} = 0.969$, $T_{max} = 0.996$
4875 measured reflections
1820 independent reflections
1237 reflections with $I > 2\sigma(I)$
$R_{int} = 0.052$
$\theta_{max} = 27.5°$
$h = -5 \rightarrow 5$
$k = -16 \rightarrow 16$
$l = -35 \rightarrow 35$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.123$
$S = 1.09$
1820 reflections
191 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0583P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Table 3
Selected geometric parameters (Å, °) for (II).

C18—O11	1.324 (3)	C18—O12	1.219 (3)
C12—C11—C17—N1	−64.8 (5)	C11—C12—C18—O11	171.2 (3)
C11—C17—N1—C21	179.1 (3)	C23—C24—N24—O41	168.8 (4)
C17—N1—C21—C22	17.7 (6)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O12^{i}	0.88	1.96	2.833 (3)	171
O11—H11···O17^{ii}	0.84	1.78	2.614 (3)	173

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

Compound (III)

Crystal data

$C_{14}H_{10}N_2O_5$
$M_r = 286.24$
Monoclinic, $P2_1/n$
$a = 10.2098$ (10) Å
$b = 9.3749$ (8) Å
$c = 27.409$ (2) Å
$\beta = 100.578$ (4)°
$V = 2578.9$ (4) Å^3
$Z = 8$

$D_x = 1.474$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5503 reflections
$\theta = 3.0$–27.4°
$\mu = 0.11$ mm^{-1}
$T = 120$ (2) K
Plate, colourless
0.22 × 0.18 × 0.10 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995, 1997)
$T_{\min} = 0.965$, $T_{\max} = 0.989$
19 240 measured reflections

5503 independent reflections
2662 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.095$
$\theta_{\max} = 27.4°$
$h = -11 \rightarrow 12$
$k = -12 \rightarrow 11$
$l = -35 \rightarrow 35$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.069$
$wR(F^2) = 0.166$
$S = 0.99$
5503 reflections
381 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0716P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.30$ e Å^{-3}
$\Delta\rho_{\min} = -0.26$ e Å^{-3}

Table 5
Selected geometric parameters (Å, °) for (III).

C18*A*—O11*A*	1.321 (3)	C18*B*—O11*B*	1.324 (3)
C18*A*—O12*A*	1.219 (3)	C18*B*—O12*B*	1.213 (3)
C12*A*—C11*A*—C17*A*—N1*A*	−85.3 (4)	C12*B*—C11*B*—C17*B*—N1*B*	−88.7 (4)
C11*A*—C17*A*—N1*A*—C21*A*	172.5 (3)	C11*B*—C17*B*—N1*B*—C21*B*	176.7 (3)
C17*A*—N1*A*—C21*A*—C22*A*	−26.3 (4)	C17*B*—N1*B*—C21*B*—C22*B*	10.8 (5)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H1*A*···O12*B*$^{\rm i}$	0.88	2.09	2.873 (4)	147
N1*B*—H1*B*···O12*A*$^{\rm ii}$	0.88	2.16	2.902 (3)	141
O11*A*—H11*A*···O17*B*	0.84	1.84	2.664 (3)	166
O11*B*—H11*B*···O17*A*$^{\rm iii}$	0.84	1.83	2.666 (3)	174
C23*B*—H23*B*···O41*A*$^{\rm iv}$	0.95	2.45	3.355 (4)	160

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

For compounds (I), (II) and (III), the space groups *Pbca*, $P2_12_12_1$ and $P2_1/n$, respectively, were uniquely assigned from the systematic absences. All H atoms were located from difference maps and then treated as riding atoms, with distances C—H = 0.95 Å, N—H = 0.88 Å and O—H = 0.84 Å. In the absence of significant anomalous scattering, the absolute configuration of (II) could not be established. However, this has no chemical significance and the Friedel equivalents were merged. Conventional refinement of (II) led to $R = 0.095$ with a maximum residual density of 1.63 e Å^{-3}, and examination of the refined structure of (II) using *PLATON* (Spek, 2003) revealed two voids, each of volume *ca* 89 Å^3, centred at approximately $(0, \frac{1}{4}, \frac{1}{2})$ and $(0, \frac{3}{4}, 0)$. The electron density within these voids could not be interpreted in terms of any sensible model of solvent molecules, and accordingly the reflection data were subjected to the *SQUEEZE* option in *PLATON* before the final refinement. This procedure suggested the presence of only 6–7 electrons per unit cell within the voids. Examination of the refined structure of (III) using *PLATON* also revealed two symmetry-related void spaces per unit cell, each of approximate volume 63 Å^3, centred at $(0, \frac{1}{2}, 0)$ and $(\frac{1}{2}, 0, \frac{1}{2})$, although the residual densities from the refinement were very low. Accordingly, a supernumerary O atom was placed near the centre of the void and its site-occupancy factor was refined, giving a value of 0.045 (6), indicative of negligible electron density within the void.

For all three compounds, data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*. For compounds (I) and (II), program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). For compound (III), program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS*97; program(s) used to refine structure: *OSCAIL* and *SHELXL*97. For all three compounds, molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PRPKAPPA* (Ferguson, 1999).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GG1204). Services for accessing these data are described at the back of the journal.

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