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3-(Acetylamino)benzoic acid

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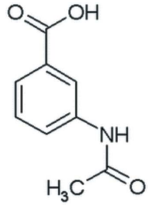
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.052; wR factor = 0.037; data-to-parameter ratio = 7.8.

The title compound, $C_9H_9NO_3$, was crystallized from methanol. The monoclinic structure features one molecule in the asymmetric unit. The topology of the O—H···O and N—H···O hydrogen-bond network can be characterized by the graph-set assignments $C(9)$ and $R_2^2(14)$.

Related literature

For the crystal structures of the *ortho*- and *para*-isomers of the title compound, see: Feeder & Jones (1992); Kashino *et al.* (1986); Kovalevsky (1999); Mascarenhas *et al.* (1980); Rajnikant & Deshmukh (2004).

For related literature, see: Etter (1990).



Experimental

Crystal data

$C_9H_9NO_3$
M_r = 179.17
Monoclinic, $P2_1/n$
a = 3.9522 (15) Å
b = 10.699 (4) Å
c = 19.831 (7) Å
β = 93.393 (8)°
V = 837.1 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293.1 K
0.60 × 0.10 × 0.05 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.937, T_{max} = 0.995
9339 measured reflections
2325 independent reflections
1168 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.037
S = 1.50
1168 reflections
152 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H7···O1^{i}	1.02 (3)	1.71 (3)	2.714 (2)	164 (2)
N1—H1···O2^{ii}	1.02 (2)	2.01 (2)	3.022 (2)	173.3 (18)

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku, 2005); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CrystalStructure*; software used to prepare material for publication: *CrystalStructure*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2363).

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Betteridge, P. W., Carruthers, J. R., Cooper, R. L., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
Feeder, N. & Jones, W. (1992). *Mol. Cryst. Liq. Cryst. Sci. Technol. Sect. A*, **211**, 111–124.
Jacobson, R. (1998). Private communication to the Rigaku Corporation.
Kashino, S., Matsushita, T., Iwamoto, T., Yamaguchi, K. & Haisa, M. (1986). *Acta Cryst.* C**42**, 457–462.
Kovalevsky, A. Yu. (1999). *Acta Cryst.* C55, IUC9900093.
Larson, A. C. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 291–294. Copenhagen: Munksgaard.
Mascarenhas, Y. P., de Almeida, V. N., Lechat, J. R. & Barelli, N. (1980). *Acta Cryst.* B**36**, 502–504.
Rajnikant, D. K. & Deshmukh, M. B. (2004). *J. Chem. Crystallogr.* **34**, 471–475.
Rigaku (2005). *CrystalStructure* (Version 3.7.0) and *CrystalClear* . Rigaku Corporation, Tokyo, Japan.

supplementary materials

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3-(Acetylamino)benzoic acid

L. K. Hansen, G. L. Perlovich and A. Bauer-Brandl

Comment

The crystal structures of 2-acetylaminobenzoic acid, (I) (Kovalevsky, 1999; Mascarenhas *et al.*, 1980; Rajnikant & Deshmukh, 2004), and 4-acetylaminobenzoic acid, (III) (Kashino *et al.*, 1986; Feeder & Jones, 1992), have been described in the literature. The crystal structure of the title compound (II) has not been solved to date. Therefore, in the present work, we have attempted to fill this gap (Fig. 1, Table 1).

The dihedral angle in (II) between the mean plane of the aromatic ring and the plane of atoms C7/O3/O1 is 6.5 (3)°. The corresponding angle between the ring and the acetyl group C2/C1/N1/C8 is 6.3 (3)°.

The unit-cell packing of (II) is shown in Figs. 2 and 3. The molecules form layers held together by N—H···O and O—H···O hydrogen bonds. The layers interact with each other by van der Waals forces. The topology of the hydrogen-bond network of (II) can be described by the graph-set assignments introduced by Etter (1990) as a C(9) chain along the c axis and an $R_2^2(14)$ intermolecular ring. The hydrogen-bond networks in (I) and (III) can be characterized as S(6) and C(8), and C(4) and $R_2^2(8)$, respectively.

Experimental

A commercial sample of 3-acetylaminobenzoic acid was used. Single crystals of (II) were grown from a water–methanol solution by vapour diffusion of water.

Refinement

The crystals of (II) were of poor quality and weakly diffracting, which accounts for the low fraction of measured reflections. All H atoms, except for those attached to the C9 methyl group, were located in difference maps and their positions and U_{iso} values were refined freely. The C9 H atoms were positioned geometrically over two orientations of equal occupancy and refined as riding, with C—H = 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C9)$.

Figures

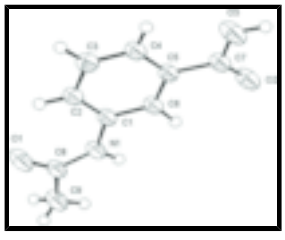


Fig. 1. A view of (II), showing displacement ellipsoids drawn at the 40% probability level (arbitrary spheres for the H atoms).

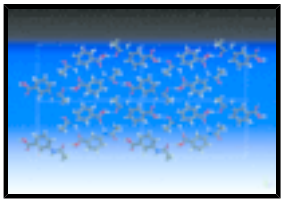


Fig. 2. A projection of the molecular packing of (II) along the a axis.

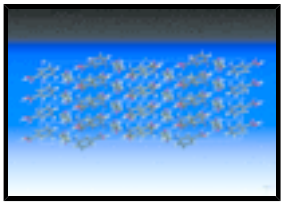


Fig. 3. A projection of the molecular packing of (II) along the b axis.

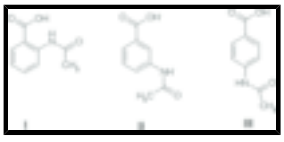


Fig. 4. Compounds (I), (II) and (III).

3-(Acetylamino)benzoic acid

Crystal data

$C_9H_9NO_3$	F_{000} = 376.00
M_r = 179.17	D_x = 1.422 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71070 Å
Hall symbol: -P 2yn	Cell parameters from 1899 reflections
a = 3.9522 (15) Å	θ = 2.8–30.5º
b = 10.699 (4) Å	μ = 0.11 mm^{-1}
c = 19.831 (7) Å	T = 293.1 K
β = 93.393 (8)º	Prism, colourless
V = 837.1 (5) Å^3	0.60 × 0.10 × 0.05 mm
Z = 4	

Data collection

Rigaku Saturn CCD area-detector diffractometer	1168 reflections with $F^2 > 2\sigma(F^2)$
Detector resolution: 7.31 pixels mm^{-1}	R_{int} = 0.031
ω scans	θ_{max} = 32.0º
Absorption correction: multi-scan (Jacobson, 1998)	h = −5→4
T_{min} = 0.937, T_{max} = 0.995	k = −15→14
9339 measured reflections	l = −27→26
2325 independent reflections	

Refinement

Refinement on F	$(\Delta/\sigma)_{max}$ = 0.012

$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.037$
$S = 1.50$
1187 reflections
152 parameters
H atoms treated by a mixture of independent and constrained refinement
Weighting scheme based on measured s.u.'s $w = 1/\sigma^2(F_o)$

$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Extinction correction: Larson (1970), equation 22
Extinction coefficient: 50 (6)

Special details

Refinement. Refinement using reflections with $F^2 > 2.0$ sigma(F^2). The weighted R-factor(wR), goodness of fit (S) and R-factor (gt) are based on F, with F set to zero for negative F. The threshold expression of $F^2 > 2.0$ sigma(F^2) is used only for calculating R-factor (gt).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.4578 (5)	0.19844 (17)	0.24639 (8)	0.0669 (6)	
O2	0.9126 (4)	0.11335 (16)	−0.10208 (8)	0.0619 (6)	
O3	0.7144 (5)	0.30147 (17)	−0.12845 (10)	0.0696 (7)	
N1	0.6495 (5)	0.09474 (19)	0.15195 (10)	0.0443 (6)	
C1	0.5574 (5)	0.1793 (2)	0.10074 (11)	0.0388 (7)	
C2	0.6738 (6)	0.1521 (2)	0.03496 (12)	0.0403 (7)	
C3	0.6057 (5)	0.2333 (2)	−0.01719 (11)	0.0393 (7)	
C4	0.4122 (6)	0.3398 (2)	−0.00383 (12)	0.0448 (8)	
C5	0.2922 (6)	0.3649 (2)	0.06125 (13)	0.0477 (8)	
C6	0.3639 (6)	0.2857 (2)	0.11430 (12)	0.0436 (7)	
C7	0.7576 (6)	0.2074 (2)	−0.08637 (12)	0.0462 (8)	
C8	0.5988 (6)	0.1067 (2)	0.21953 (12)	0.0477 (8)	
C9	0.7234 (6)	−0.0009 (2)	0.25989 (12)	0.0614 (9)	
H1	0.780 (5)	0.021 (2)	0.1341 (11)	0.077 (9)*	
H2	0.822 (4)	0.0766 (18)	0.0236 (10)	0.044 (6)*	
H4	0.377 (5)	0.396 (2)	−0.0423 (11)	0.056 (7)*	
H5	0.147 (5)	0.4411 (19)	0.0738 (10)	0.055 (7)*	
H6	0.272 (4)	0.3037 (17)	0.1606 (10)	0.042 (6)*	
H7	0.821 (7)	0.286 (2)	−0.1736 (17)	0.120 (11)*	
H8	0.7098	−0.0743	0.2329	0.073*	0.50
H9	0.5888	−0.0111	0.2976	0.073*	0.50
H10	0.9525	0.0133	0.2752	0.073*	0.50
H11	0.9227	−0.0622	0.2293	0.073*	0.50
H12	0.8177	0.0348	0.3013	0.073*	0.50
H13	0.5133	−0.0634	0.2731	0.073*	0.50

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1056 (16)	0.0619 (13)	0.0334 (10)	0.0159 (11)	0.0067 (10)	−0.0059 (9)
O2	0.0922 (14)	0.0543 (12)	0.0395 (11)	0.0146 (11)	0.0056 (9)	−0.0051 (9)
O3	0.1094 (17)	0.0633 (14)	0.0367 (11)	0.0188 (11)	0.0093 (11)	0.0114 (10)
N1	0.0594 (13)	0.0427 (13)	0.0305 (11)	0.0021 (11)	0.0009 (9)	−0.0005 (10)
C1	0.0475 (15)	0.0381 (14)	0.0305 (13)	−0.0041 (12)	0.0005 (11)	0.0006 (11)
C2	0.0507 (15)	0.0382 (14)	0.0320 (14)	0.0001 (13)	0.0012 (11)	−0.0035 (12)
C3	0.0471 (14)	0.0395 (14)	0.0308 (14)	−0.0066 (12)	−0.0006 (11)	−0.0008 (12)
C4	0.0528 (17)	0.0443 (16)	0.0367 (15)	−0.0042 (13)	−0.0024 (13)	0.0028 (13)
C5	0.0507 (16)	0.0421 (16)	0.0502 (17)	0.0055 (13)	0.0027 (13)	−0.0021 (14)
C6	0.0527 (16)	0.0437 (15)	0.0347 (14)	0.0001 (13)	0.0052 (12)	−0.0052 (13)
C7	0.0576 (17)	0.0468 (16)	0.0334 (15)	−0.0053 (14)	−0.0042 (12)	0.0007 (13)
C8	0.0571 (17)	0.0521 (17)	0.0339 (15)	−0.0091 (14)	0.0027 (12)	0.0007 (14)
C9	0.079 (2)	0.0635 (19)	0.0407 (17)	0.0043 (16)	−0.0060 (15)	0.0121 (15)

Geometric parameters (Å, °)

O1—C8	1.262 (3)	C5—C6	1.368 (3)
O2—C7	1.228 (3)	C8—C9	1.471 (3)
O3—C7	1.312 (3)	O3—H7	1.02 (3)
N1—C1	1.392 (2)	N1—H1	1.02 (2)
N1—C8	1.373 (3)	C2—H2	1.031 (19)
C1—C2	1.438 (3)	C4—H4	0.97 (2)
C1—C6	1.406 (3)	C5—H5	1.04 (2)
C2—C3	1.365 (3)	C6—H6	1.03 (2)
C3—C4	1.406 (3)	C9—H8	0.950
C3—C7	1.555 (3)	C9—H9	0.950
C4—C5	1.427 (3)	C9—H10	0.950
C1—N1—C8	127.2 (2)	C1—C2—H2	125.0 (11)
N1—C1—C2	116.5 (2)	C3—C2—H2	115.0 (11)
N1—C1—C6	120.5 (2)	C3—C4—H4	113.8 (13)
C2—C1—C6	122.9 (2)	C5—C4—H4	123.7 (13)
C1—C2—C3	120.0 (2)	C4—C5—H5	125.8 (12)
C2—C3—C4	117.2 (2)	C6—C5—H5	113.3 (12)
C2—C3—C7	119.1 (2)	C1—C6—H6	123.2 (10)
C4—C3—C7	123.6 (2)	C5—C6—H6	120.3 (10)
C3—C4—C5	122.5 (2)	C8—C9—H8	109.6
C4—C5—C6	120.9 (2)	C8—C9—H9	109.5
C1—C6—C5	116.4 (2)	C8—C9—H10	109.4
O2—C7—O3	121.0 (2)	C8—C9—H11	110.9
O2—C7—C3	126.5 (2)	C8—C9—H12	104.7
O3—C7—C3	112.5 (2)	C8—C9—H13	111.7
O1—C8—N1	125.3 (2)	H8—C9—H9	109.5
O1—C8—C9	121.4 (2)	H8—C9—H10	109.5
N1—C8—C9	113.2 (2)	H9—C9—H10	109.5

C7—O3—H7	112.8 (16)	H11—C9—H12	114.3
C1—N1—H1	111.4 (13)	H11—C9—H13	108.0
C8—N1—H1	121.3 (13)	H12—C9—H13	107.2
C1—N1—C8—O1	−0.1 (2)	C1—C2—C3—C7	175.3 (2)
C1—N1—C8—C9	179.2 (2)	C2—C3—C4—C5	0.7 (3)
C8—N1—C1—C2	172.9 (2)	C2—C3—C7—O2	7.3 (3)
C8—N1—C1—C6	−6.3 (3)	C2—C3—C7—O3	−170.5 (2)
N1—C1—C2—C3	−177.3 (2)	C4—C3—C7—O2	−175.8 (2)
N1—C1—C6—C5	178.5 (2)	C4—C3—C7—O3	6.4 (3)
C2—C1—C6—C5	−0.7 (3)	C7—C3—C4—C5	−176.3 (2)
C6—C1—C2—C3	1.9 (3)	C3—C4—C5—C6	0.5 (3)
C1—C2—C3—C4	−1.8 (3)	C4—C5—C6—C1	−0.5 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H7···O1^{i}	1.02 (3)	1.71 (3)	2.714 (2)	164 (2)
N1—H1···O2^{ii}	1.02 (2)	2.01 (2)	3.022 (2)	173.3 (18)

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

Fig. 1

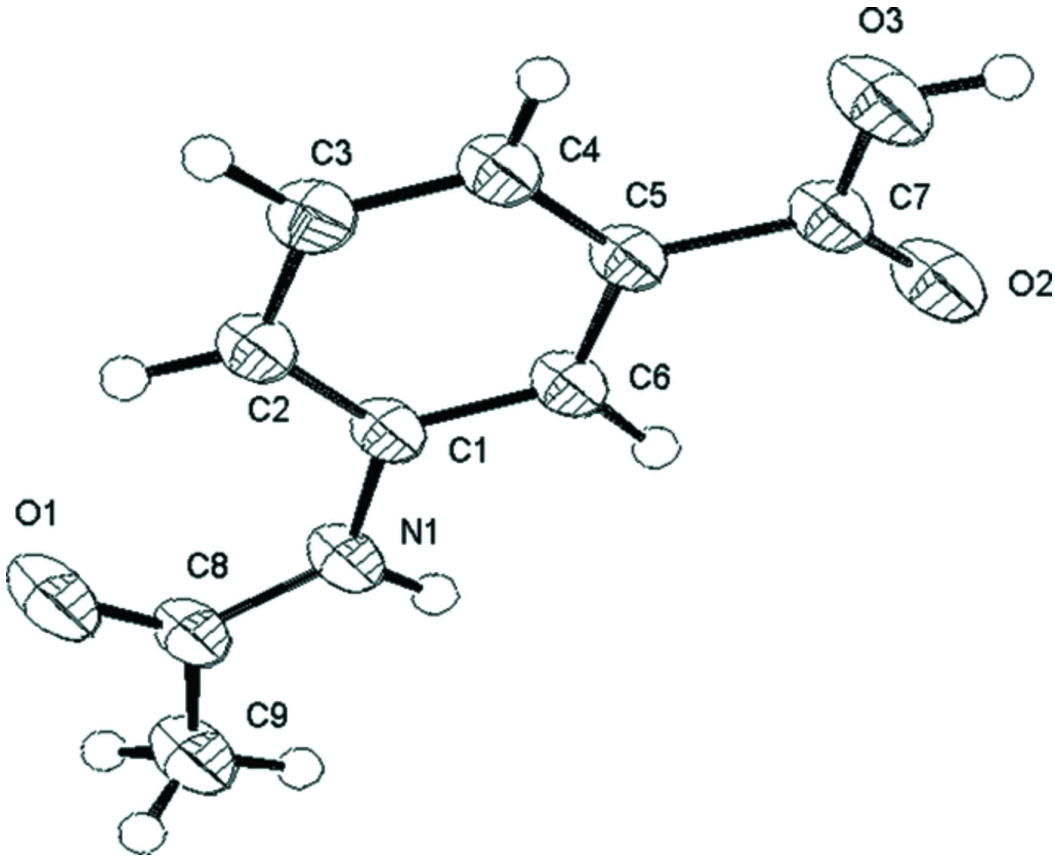


Fig. 2

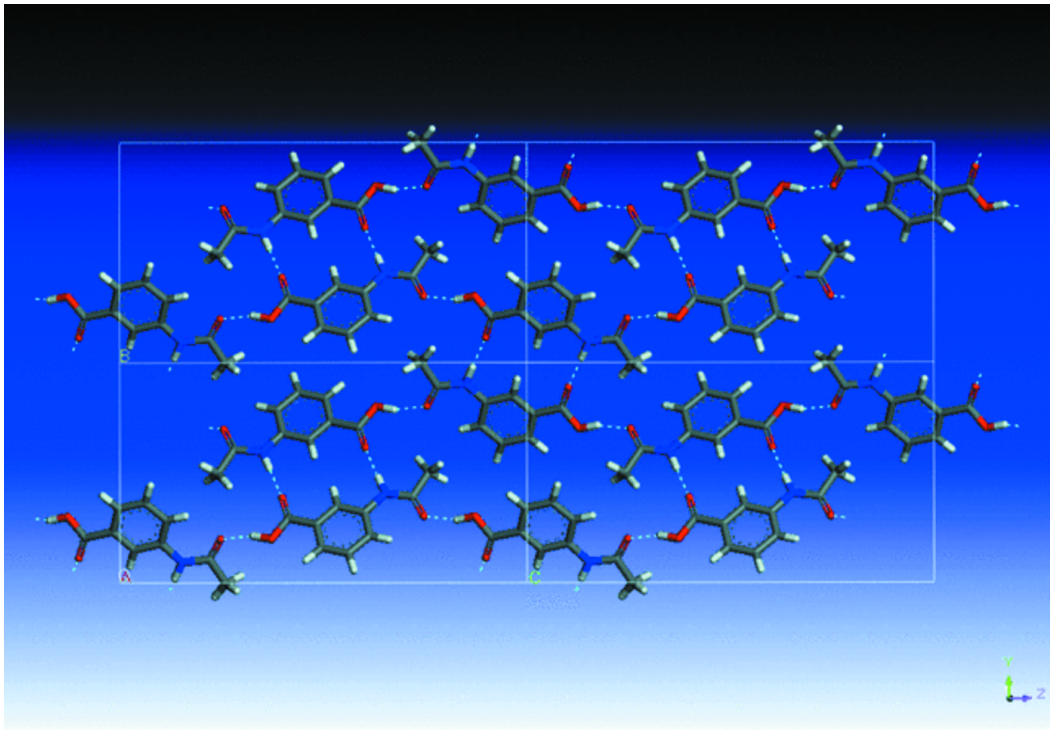


Fig. 3

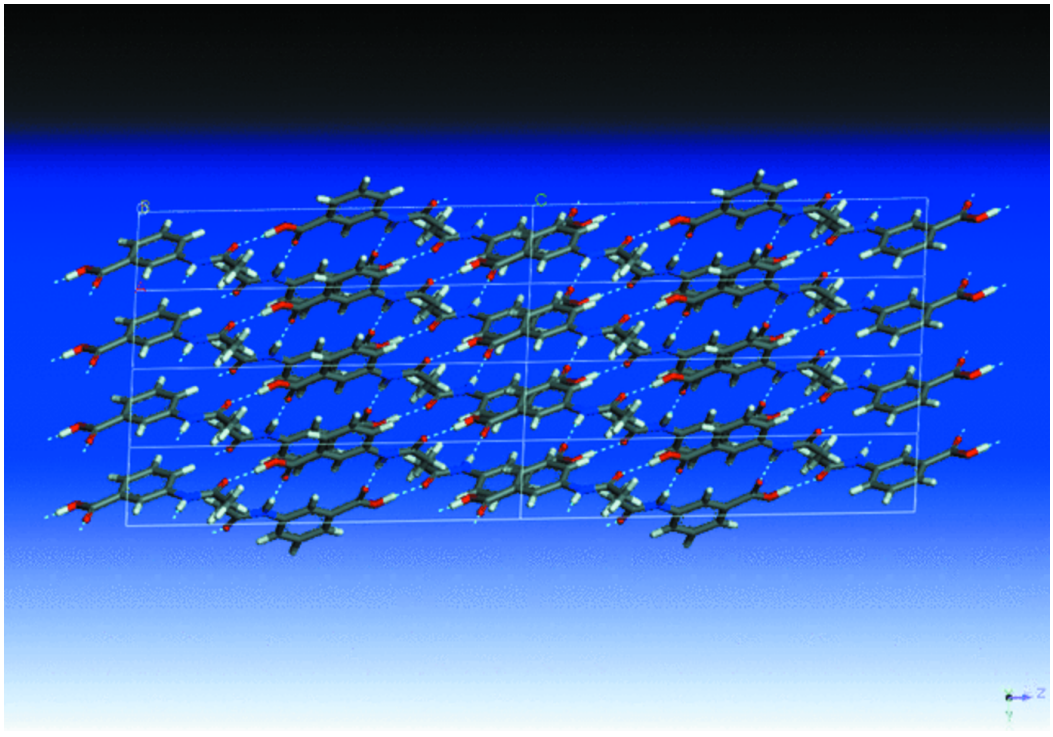
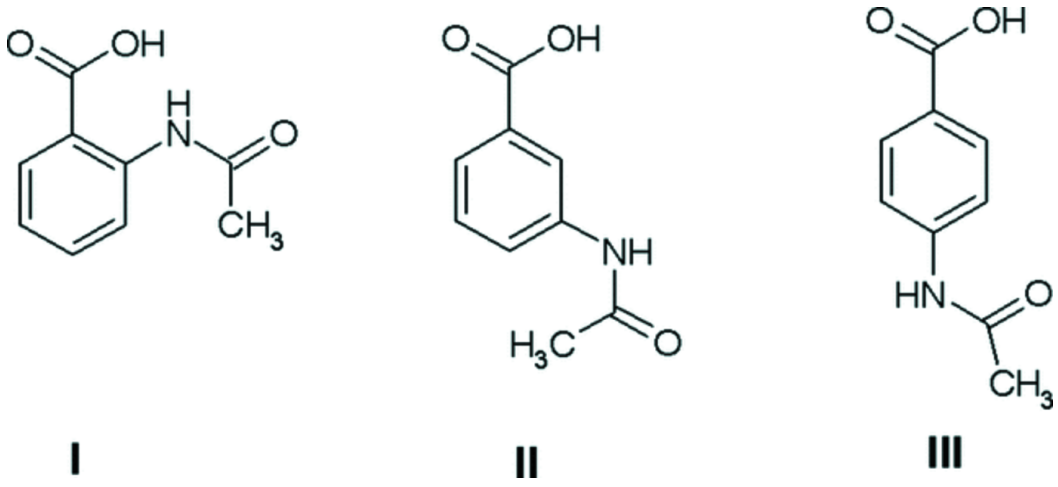


Fig. 4



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