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4-(Acetylamino)-3-hydroxy-5-nitrobenzoic Acid

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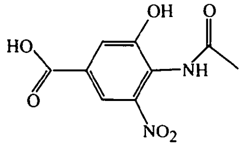
Abstract

The 4-(acetylamino)-3-hydroxy-5-nitrobenzoic acid molecule, $C_9H_8N_2O_6$, a designed inhibitor for the influenza virus neuraminidase protein, crystallizes as hydrogen-bonded dimers. The dihedral angles of the substituent groups with respect to the planar phenyl moiety are 5.0 (3)° for the carboxyl group, 45.0 (2)° for the nitro group and 37.3 (1)° for the acetylamino substituent. The crystal structure is stabilized by intermolecular hydrogen bonding.

Comment

The current X-ray diffraction study establishes the structure of the benzoic acid derivative 4-(acetylamino)-3-hydroxy-5-nitrobenzoic acid, (I). This structure and those of similar compounds are of importance both for their application in structure-based drug design and in structure–activity studies of the influenza virus neuraminidase protein (Jedrzejas *et al.*, 1995). The structure of benzoic acid has been well established by both X-ray and neutron studies (Sim, Robertson & Goodwin, 1955; Bruno & Randaccio, 1980; Feld, Lehman, Muir & Speakman, 1981). However, further details concerning the conformation of the carboxyl, *N*-acetyl and nitro substituents of the title compound were needed for the purpose of further characterization.



(I)

The benzene ring atoms are planar to within 0.026 (2) Å, with C—C bond lengths varying from 1.377 (3) to 1.408 (3) Å and endocyclic C—C—C bond angles varying from 118.7 (2) to 122.2 (2)°, which agrees with accepted values (Bruno & Randaccio, 1980; Karle, 1952*a*,*b*). The carboxyl, nitro and *N*-acetyl groups are rotated with respect to the benzene ring by 5.0 (3), 45.0 (2) and 37.3 (1)°, respectively. The acetylamino group (N4, C7, O7 and C8) is planar to within 0.0009 (2) Å, with a C—O distance of 1.209 (3) Å. The two C—O bond distances of the carboxyl group are 1.290 (3) (C9—O9) and 1.224 (3) Å (C9—O9′), and the corresponding angles are 115.6 (2) (C1—C9—O9) and 120.2 (2)° (C1—C9—O9′). The nitro group N—O distances are, as expected, almost equal: 1.231 (3) (N5—O5) and 1.198 (3) Å (N5—O5′), with corresponding angles of 117.0 (2) (C5—N5—O5) and 118.5 (2)° (C5—N5—O5′).

Fig. 1 presents an *ORTEP*II (Johnson, 1976) drawing of the title molecule together with the atomic labelling scheme. The title compound crystallizes in a dimeric form. The molecules involved in this dimer interact through hydrogen bonds between their carboxyl groups (see Fig. 2) (Benghiat & Leiserowitz, 1972). A second intermolecular hydrogen bond is present between atom O7 of the *N*-acetyl group and the HO3 atom of the hydroxyl group. Appropriate distances and angles for the

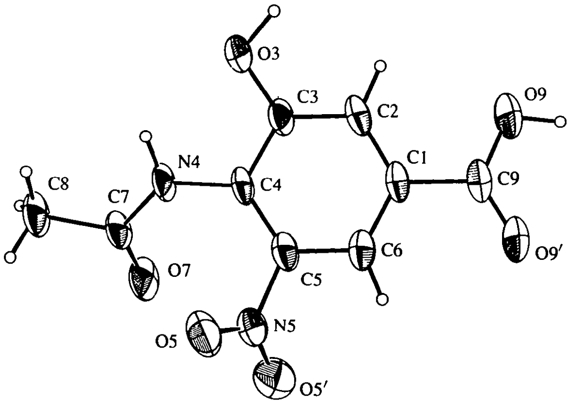


Fig. 1. An *ORTEP*II drawing (Johnson, 1976) of the title compound with the atomic labelling scheme. Ellipsoids are drawn at the 50% probability level for non-H atoms and H atoms are represented as spheres equivalent to B_{eq} = 0.5 Å^2.

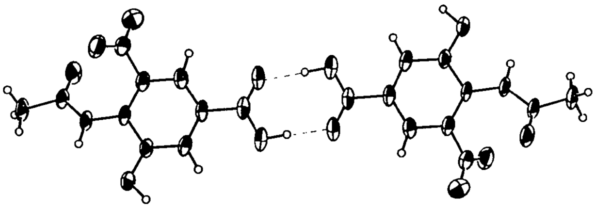


Fig. 2. Representation of the dimeric structure of the title compound in the crystalline state.

above hydrogen bonds are supplied in Table 2. Selected torsion angles describing the geometry of the phenyl substituents, *i.e.* CO_2H, NO_2, OH and C_2H_4NO, are also listed in Table 2.

The crystal structure of the title compound is similar to other phenyl-ring-based structures (Bryan, Hartley, Peckler, Fujita, Nagao & Seno, 1980; Burns & Hagaman, 1993; Carpy, Goursolle & Leger, 1980; Gozlan & Riche, 1976; Kageyama, Iwamoto, Haisa & Kashino, 1993; Metzger, Laidlaw, Torres & Panetta, 1989; Metzger, Atwood, Lee, Rao, Lal & Loo, 1993; Nielsen & Larsen, 1993; Okabe, 1993; Okabe, Nakamura & Fukuda, 1993; Soundararajan, Duesler & Hageman, 1993).

Experimental

The title compound, a yellowish powder, was prepared *via* the hydrolysis of 3-acetoxy-4-(acetylamino)-5-nitrobenzoic acid in 0.1 *M* NaOH. The mixture was acidified with concentrated HCl, diluted with water and extracted with ethyl acetate. Crystals were obtained by slow evaporation of a methanol solution at room temperature.

Crystal data

$C_9H_8N_2O_6$
M_r = 240.172
Triclinic
$P\bar{1}$
a = 7.484 (1) Å
b = 10.735 (1) Å
c = 7.1191 (9) Å
α = 98.37 (1)°
β = 109.59 (1)°
γ = 105.98 (1)°
V = 499.8 (2) Å^3
Z = 2
D_x = 1.596 Mg m^{-3}

Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 30.09–39.62°
μ = 0.114 mm^{-1}
T = 293 K
Irregular
0.45 × 0.44 × 0.15 mm
Yellowish and transparent

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
T_{min} = 0.978, T_{max} = 0.997
1619 measured reflections
1449 independent reflections

1376 observed reflections [$I > 3\sigma(I)$]
R_{int} = 0.049
θ_{max} = 65.5°
$h = -8 \rightarrow 8$
$k = -12 \rightarrow 12$
$l = 0 \rightarrow 8$
3 standard reflections
frequency: 60 min
intensity decay: 0.059% h^{-1}

Refinement

Refinement on F
R = 0.0517
wR = 0.0647
S = 2.43
1376 reflections
170 parameters
$w = 4(F_o^2)/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{max}$ = 0.00035

$\Delta\rho_{max}$ = 0.265 e Å^{-3}
$\Delta\rho_{min}$ = −0.253 e Å^{-3}
Extinction correction: $F_c = F_c/(1 + gI_c)$
Extinction coefficient: $g = 1.4657 \times 10^{-6}$
Atomic scattering factors from Cromer & Waber (1974)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
O3	0.7318 (2)	0.2525 (2)	0.3639 (2)	4.16 (3)
O5′	0.1612 (3)	0.2009 (2)	0.7616 (3)	5.26 (4)
O5	0.2263 (3)	0.0313 (2)	0.6397 (2)	4.41 (4)
O7	0.6098 (2)	0.2109 (2)	0.9485 (2)	4.16 (4)
O9	0.2464 (3)	0.4819 (2)	0.0575 (3)	5.15 (4)
O9′	0.0165 (3)	0.4299 (2)	0.1927 (3)	5.71 (4)
N4	0.6282 (2)	0.1490 (2)	0.6420 (2)	3.04 (4)
N5	0.2361 (3)	0.1493 (2)	0.6631 (3)	3.54 (4)
C1	0.2951 (3)	0.3568 (2)	0.3046 (3)	2.92 (4)
C2	0.4623 (3)	0.3427 (2)	0.2742 (3)	3.06 (4)
C3	0.5692 (3)	0.2724 (2)	0.3871 (3)	2.84 (4)
C4	0.5095 (3)	0.2166 (2)	0.5330 (3)	2.66 (4)
C5	0.3354 (3)	0.2280 (2)	0.5508 (3)	2.98 (4)
C6	0.2311 (3)	0.3011 (2)	0.4436 (3)	3.17 (4)
C7	0.6726 (3)	0.1496 (2)	0.8446 (3)	2.97 (4)
C8	0.8026 (3)	0.0691 (2)	0.9249 (3)	3.86 (5)
C9	0.1744 (3)	0.4277 (2)	0.1789 (3)	3.40 (5)

Table 2. *Selected geometric parameters* (Å, °)

O3—C3	1.349 (3)	C1—C2	1.382 (4)
O5′—N5	1.198 (3)	C1—C6	1.377 (3)
O5—N5	1.231 (3)	C1—C9	1.494 (3)
O7—C7	1.209 (3)	C2—C3	1.392 (3)
O9—C9	1.290 (3)	C3—C4	1.408 (3)
O9′—C9	1.224 (3)	C4—C5	1.387 (3)

N4—C4	1.405 (3)	C5—C6	1.386 (3)
N4—C7	1.366 (3)	C7—C8	1.502 (3)
N5—C5	1.468 (3)		
C4—N4—C7	125.4 (2)	N4—C4—C5	125.7 (2)
O5′—N5—O5	124.4 (2)	C3—C4—C5	117.7 (2)
O5′—N5—C5	118.5 (2)	N5—C5—C4	121.7 (2)
O5—N5—C5	117.0 (2)	N5—C5—C6	115.6 (2)
C2—C1—C6	121.2 (2)	C4—C5—C6	122.2 (2)
C2—C1—C9	120.1 (2)	C1—C6—C5	118.7 (2)
C6—C1—C9	118.6 (2)	O7—C7—N4	121.4 (2)
C1—C2—C3	119.6 (2)	O7—C7—C8	123.6 (2)
O3—C3—C2	123.1 (2)	N4—C7—C8	115.0 (2)
O3—C3—C4	116.4 (2)	O9—C9—O9′	124.2 (2)
C2—C3—C4	120.5 (2)	O9—C9—C1	115.6 (2)
N4—C4—C3	116.6 (2)	O9′—C9—C1	120.2 (2)
C7—N4—C4—C3	143.6 (2)	C2—C1—C9—O9	−5.4 (3)
C4—N4—C7—O7	0.1 (3)	C6—C1—C2—HC2	177 (1)
C4—N4—C7—C8	179.9 (2)	C2—C1—C6—HC6	−178 (1)
O5—N5—C5—C4	−40.6 (3)	HO9—O9—C9—C1	180.0
C9—C1—C2—C3	−177.7 (2)		

D—H···*A*	H···*A*	*D*—H···*A*
O9—HO9···O9′*	1.729	167.2
O3*—HO3*···O7	1.767	155.8
C9—O9′···HO9*		123.0
C7—O7···HO3*		125.0

* Denotes an atom of a symmetry-related molecule.

Profile analysis was performed on all reflections (Lehman & Larsen, 1974; Grant & Gabe, 1978). Intensities were corrected for Lorentz–polarization effects, linear decay and absorption. The Enraf–Nonius *MolEN* structure determination package (Fair, 1990) was used to solve and refine the structure. The H atoms attached to atoms C2, C6 and N4 were refined (positional and displacement parameters) and the H atoms attached to atoms O9 and O3 were located on a difference electron density map and were not refined, their displacement parameters being calculated based on the temperature factors of the atoms to which they were bonded ($1.45B_{eq}$). The H atoms on atoms C8, C8′ and C8″ were located or calculated and were not refined.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and root-mean-square amplitudes of anisotropic displacement have been deposited with the IUCr (Reference: PT1001). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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4-(2-Carboxybenzoyl)-2(3*H*)-benzothiazolone

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Abstract

Although 6-benzoyl-2(3*H*)-benzothiazolone derivatives are known, their 4-benzoyl analogues have not been described previously. The title compound [2-(2-oxo-3*H*-1,3-benzothiazol-4-oyl)benzoic acid, $C_{15}H_9NO_4S$] is the first of this series to be isolated and is characterized here. It was obtained in low yield in a one-step reaction from 3*H*-benzothiazolone and phthalic anhydride (in