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Crystal structures of 2-amino-2-oxoethyl 4-bromobenzoate, 2-amino-2-oxoethyl 4-nitrobenzoate and 2-amino-2-oxoethyl 4-aminobenzoate monohydrate

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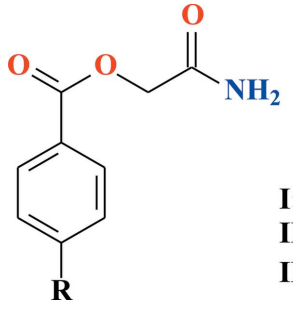
CCDC references: 2041177; 2041176; 2041175

Supporting information: this article has supporting information at journals.iucr.org/e

The title molecules were synthesized by the reaction of 4-substituted sodium benzoates with chloroacetic acid amide in the presence of dimethylformamide. The yields of 2-amino-2-oxoethyl 4-bromobenzoate, $C_9H_8BrNO_3$, **I**, 2-amino-2-oxoethyl 4-nitrobenzoate, $C_9H_8N_2O_5$, **II**, and 2-amino-2-oxoethyl 4-aminobenzoate monohydrate, $C_9H_{10}N_2O_3{\cdot}H_2O$, **III**, are 86, 78 and 88%, respectively. The low yield of **II** is explained by the reduced reactivity of the molecule in a nucleophilic exchange reaction because of the negative induction and negative mesomeric effects of the nitro group on the benzene ring. Single crystals were obtained from the products under the same (temperature and solvent) conditions. In the case of **III**, the crystals formed as a monohydrate. In all three crystal structures, the same type of intermolecular N—H···O hydrogen bonds are observed, but the molecules differ in some torsion angles as well as in the dihedral angles between the mean planes of the benzene rings and the amide groups.

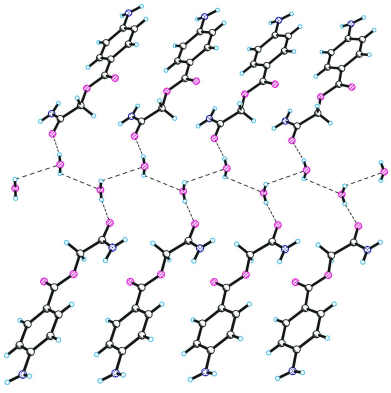
1. Chemical context

Molecules containing an aromatic ring, a carboxyl and an amino group represent an important class of organic compounds and, with several reaction centers, they are important intermediates in industry. They are often used as synthons in organic synthesis and are also widely used as ligands in the coordination chemistry of various transition metals. These ligands can form a variety of complex compounds as they possess several Lewis base sites.



I: R = Br
II: R = NO_2
III: R = NH_2 **monohydrate**

The new crystalline compounds 2-amino-2-oxoethyl 4-bromobenzoate (**I**), 2-amino-2-oxoethyl 4-nitrobenzoate (**II**) and 2-amino-2-oxoethyl)-4-aminobenzoate monohydrate (**III**) (Fig. 1) were synthesized from the reaction of 4-substituted sodium benzoates with chloroacetic acid amide in the presence of dimethylformamide. Their structures were determined by X-ray crystallographic analysis.



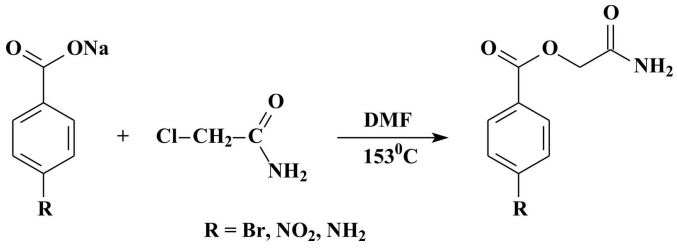


Figure 1
Reaction scheme for the synthesis of (2-amino-2-oxoethyl)benzoate derivatives.

2. Structural commentary

All of the title structures have planar benzoate (C1–C7/O1/O2) and amide (O3/C9/N1) units but the dihedral angle between these planes is different in each case because of the torsion angle about the bridging methylene group (C8; Tables 1–3). The asymmetric unit of each crystal structure is illustrated in Fig. 2. That of **I** consists of two independent molecules (*A* and *B*), which differ in the position of the amide groups relative to the benzoate (r.m.s. deviations of 0.021 Å for *A* and 0.031 Å for *B*) fragments, as indicated by the dihedral angles of 82.5 (4) and 75.9 (3)° in *A* and *B*, respectively. The asymmetric unit of **II** contains only one molecule of 2-amino-2-oxoethyl 4-nitrobenzoate. The dihedral angle between the mean planes of the amide and the benzoate (r.m.s. deviation = 0.070 Å) groups is 89.4 (2)°. The asymmetric unit of **III** contains one water molecule and one 2-amino-2-oxoethyl 4-aminobenzoate molecule (Fig. 2). The dihedral angle between the mean planes of the amide and benzoate (r.m.s. deviation = 0.027 Å) groups is 4.4 (5)°. Analysis of the bond lengths and bond angles of **I**–**III** shows slight differences, but these data are in the expected ranges (Allen *et al.*, 1987).

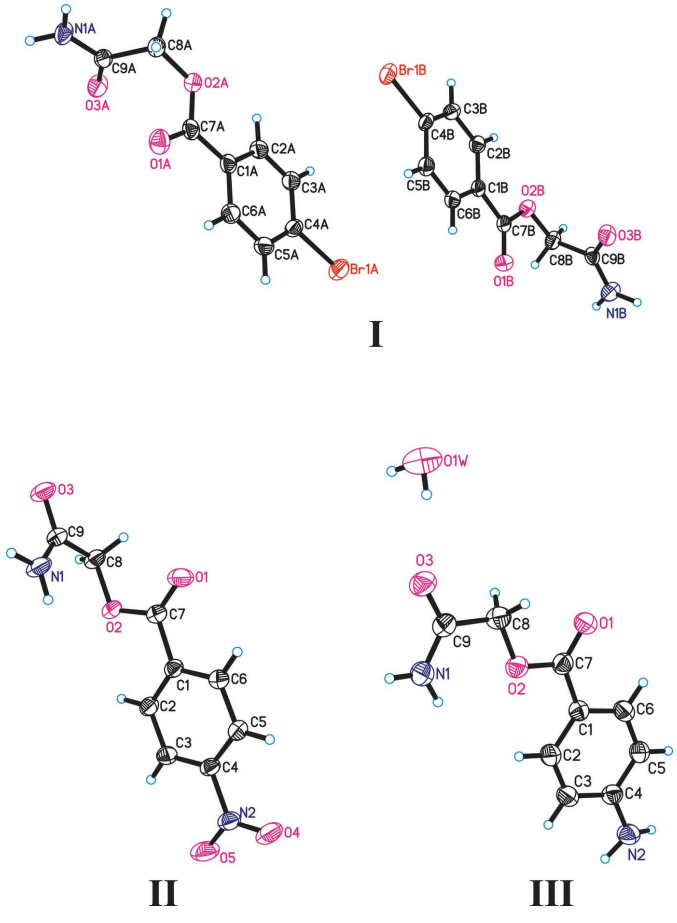


Figure 2
The asymmetric units of **I**–**III** with atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

Table 1
Selected torsion angles (°) for (I).

C8*A*—O2*A*—C7*A*—C1*A*	179.4 (3)	C8*B*—O2*B*—C7*B*—C1*B*	177.1 (3)
C6*A*—C1*A*—C7*A*—O2*A*	178.3 (3)	C6*B*—C1*B*—C7*B*—O2*B*	−176.7 (3)
C7*A*—O2*A*—C8*A*—C9*A*	−72.9 (5)	C7*B*—O2*B*—C8*B*—C9*B*	−69.1 (5)
O2*A*—C8*A*—C9*A*—O3*A*	−16.9 (6)	O2*B*—C8*B*—C9*B*—O3*B*	−17.6 (6)

Table 2
Selected torsion angles (°) for (II).

C8—O2—C7—C1	−176.52 (14)	C7—O2—C8—C9	−95.53 (19)
C6—C1—C7—O2	−170.76 (15)	O2—C8—C9—O3	175.79 (17)

Table 3
Selected torsion angles (°) for (III).

C8—O2—C7—C1	−178.9 (2)	C7—O2—C8—C9	−179.2 (2)
C6—C1—C7—O2	−177.0 (2)	O2—C8—C9—O3	177.4 (2)

3. Supramolecular features

In the crystal structures, several types of intermolecular interactions are observed but all contain intermolecular N—H···O hydrogen bonds.

In **I**, intermolecular *A*···*A* (N1*A*—H1*A*···O3*A*^{i}), *B*···*B* (N1*B*—H2*B*···O3*B*^{ii}) and *B*···*A* (N1*B*—H2*B*···O3*B*^{iii}) interactions cross-link the molecules, generating rings with an $R_3^3(12)$ graph-set motif (Fig. 3, Table 4) (Grell *et al.*, 1999). Although both the *A* and *B* molecules contain a bromine atom, a short intermolecular Br···Br interaction only occurs between *B* molecules [Br1*B*···Br1*B*($-x+\frac{3}{2}$, $y+\frac{1}{2}$, $-z+\frac{1}{2}$) = 3.519 (1) Å, 0.18 Å less than the sum of the van der Waals radii]. This interaction connects the molecules into chains extending along the *b*-axis direction (Fig. 3). A similar short Br ··· Br interaction was observed in the crystal structures of 4-chlorophenyl-4-bromobenzoate (TAYNEP; Saha & Desiraju, 2017) and 4-bromophenyl-4-bromobenzoate (VEWSIC; Saha & Desiraju, 2018).

In **II**, the angle between the mean planes of the nitro group and the aromatic ring is 4.1 (1)°. A characteristic intermolecular interaction for **II** is the formation of centrosymmetric dimers as a result of the N1—H1···O3^{i} hydrogen bonds formed between amide fragments (Table 5). Short intermolecular O5···O5($-x+1$, $-y+2$, $-z+1$) interactions [at 2.874 (4) Å these are 0.14 Å less than the sum of the van der

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

D—H···A	D—H	H···A	D···A	D—H···A
N1A—H1A···O3A^{i}	0.85 (6)	2.18 (7)	2.969 (5)	153
N1B—H1B···O3A^{ii}	0.93 (6)	2.24 (6)	3.163 (5)	170
N1B—H2B···O3B^{iii}	0.86 (9)	1.97 (9)	2.825 (5)	178

Symmetry codes: (i) $x, y+1, z$; (ii) $x-1, y-1, z$; (iii) $x, y-1, z$.

Table 5
Hydrogen-bond geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3^{i}	0.93 (2)	1.97 (2)	2.898 (2)	174 (2)

Waals radii] are observed between the nitro groups of the dimers (Fig. 4). A similar intermolecular O···O contact was observed in the crystal structure of *meta*-dinitrobenzene (DNBENZ11, DNBENZ12; Wójcik *et al.*, 2002).

In **III**, as in **II**, inversion dimers are formed by N1—H1···O3^{i} hydrogen bonds (Fig. 5, Table 6). An intermolecular hydrogen bond is also observed between the oxygen of the amide fragment and the water molecule (Fig. 6), although the

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3^{i}	0.87 (4)	2.06 (4)	2.915 (3)	168
N2—H3···O1^{ii}	0.96 (3)	1.98 (3)	2.919 (4)	163
O1W—H1W···O3	0.78 (4)	2.14 (4)	2.916 (4)	169 (4)
O1W—H2W···O1W^{iii}	0.91 (9)	2.46 (9)	2.782 (7)	101

Symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $x-1, y, z$; (iii) $-x+2, -y+1, -z+1$.

angle is only 101°, and the water molecules are further connected by hydrogen bonds to form an infinite chain along the *b*-axis direction.

4. Database survey

A search for the 2-amino-2-oxoethyl benzoate (carbamoylmethylbenzoate) scaffold in the Cambridge Structural Database (CSD Version 5.41, update of November 2019; Groom *et al.*, 2016) gave 34 hits. Of these, the structures most closely related to the title compounds are MAMJOC [2-(dimethylamino)-2-oxoethyl 5-bromo-2-hydroxybenzoate; Santra *et al.*, 2016], CEPWID (1-benzoyloxy-1-methoxy-*N*-methyl-

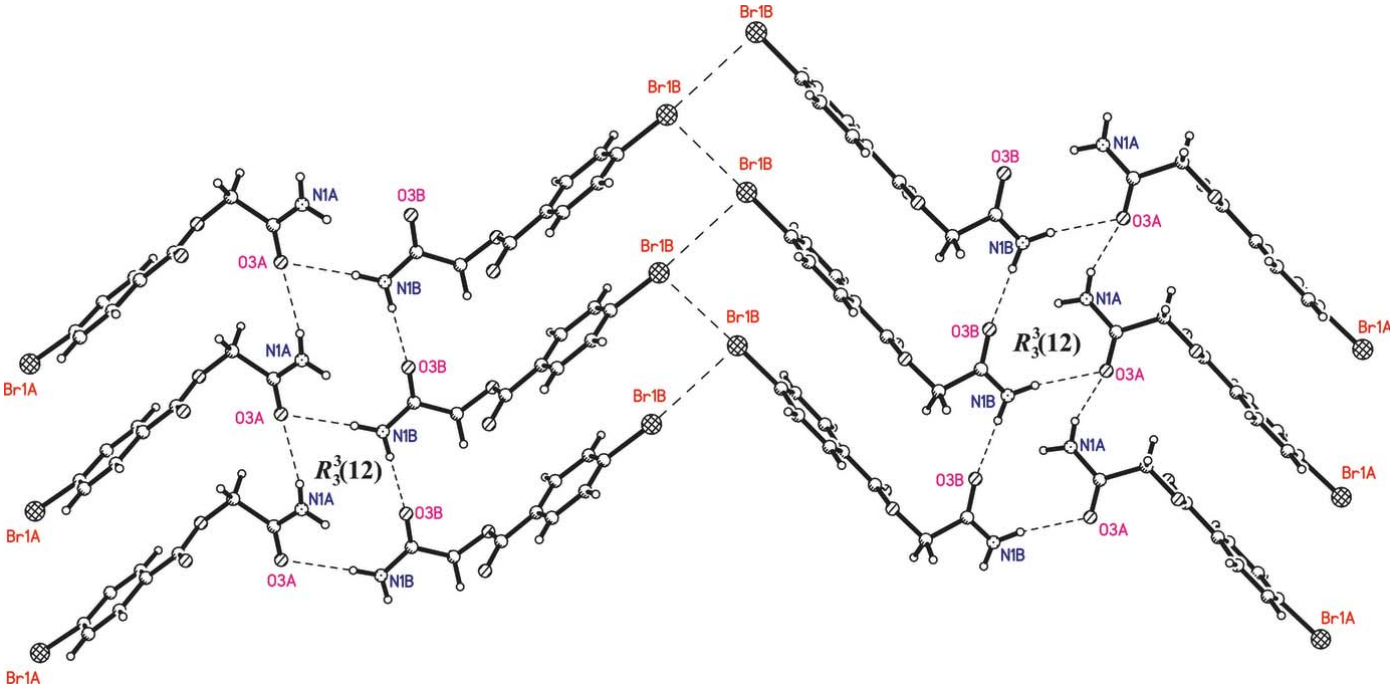


Figure 3
Hydrogen bonds (formation of rings) and intermolecular Br···Br contacts in **I**.

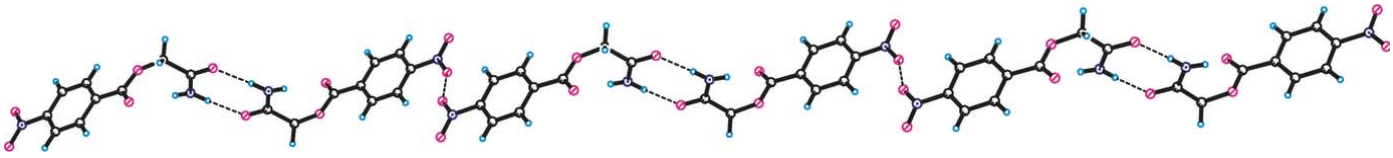


Figure 4
Hydrogen bonds and intermolecular O···O contacts in **II**.

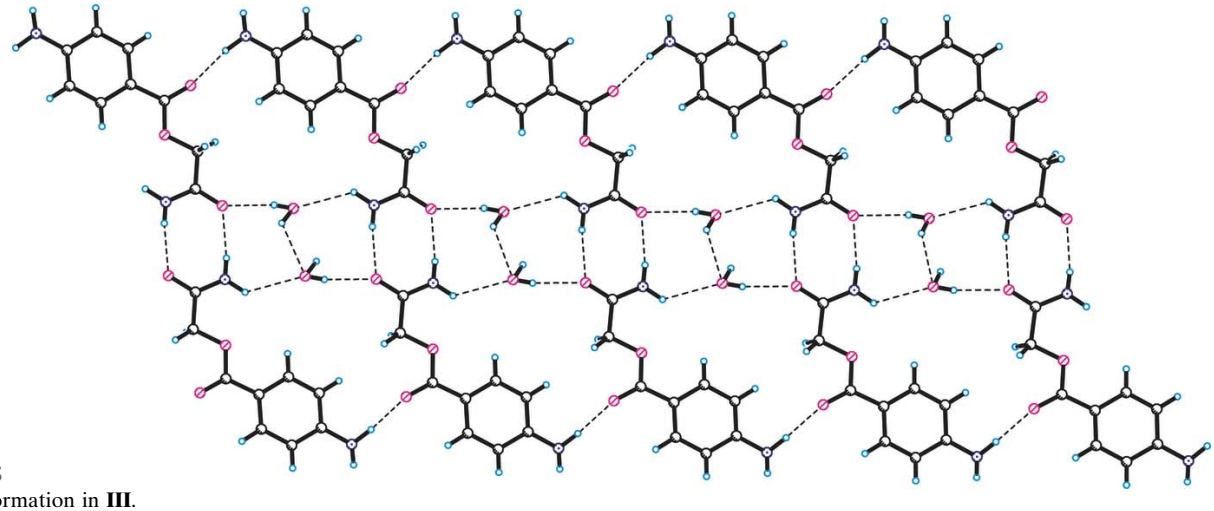


Figure 5
Dimer formation in **III**.

acetamide; Nishio *et al.*, 1984) and HUMJII (carbamoylmethyl 3,4,5-trihydroxybenzoate hydrate; Parkin *et al.*, 2002).

5. Synthesis and crystallization

Synthesis 2-amino-2-oxoethyl 4-bromobenzoate: General method. To a 25 mL round-bottom flask containing 0.27 g (1.2 mmol) of the sodium salt of *p*-bromobenzoic acid were added 6 mL of DMF. The resulting mixture was heated for 10 min (for partial dissolution of the salt) and 0.1 g (1 mmol) of chloroacetamide was added. The flask was equipped with a reflux condenser and mechanical stirrer and was heated in a sand bath with stirring at 426 K for 6 h. The DMF was removed *in vacuo* (15 mm Hg) at 328 K. After cooling, cold water was poured into the reaction mixture to completely eliminate the residual reactants and DMF. The resulting precipitate was filtered off to give 0.22 g (86% yield) of product. R_F = 0.65 [in 5:1.5:1 (*v*/*v*) $CHCl_3/C_6H_6/CH_3OH$ solvent system]; m.p. 475–477 K. ^{1}H NMR [400 MHz, CD_3OD, δ (ppm.), *J* (Hz)]: 7.95 (2H, *d*, *J* = 8.64, H3 and H5), 7.61 (2H, *d*, *J* = 8.63, H2 and H6), 4.72 (2H, *s*, CH_2).

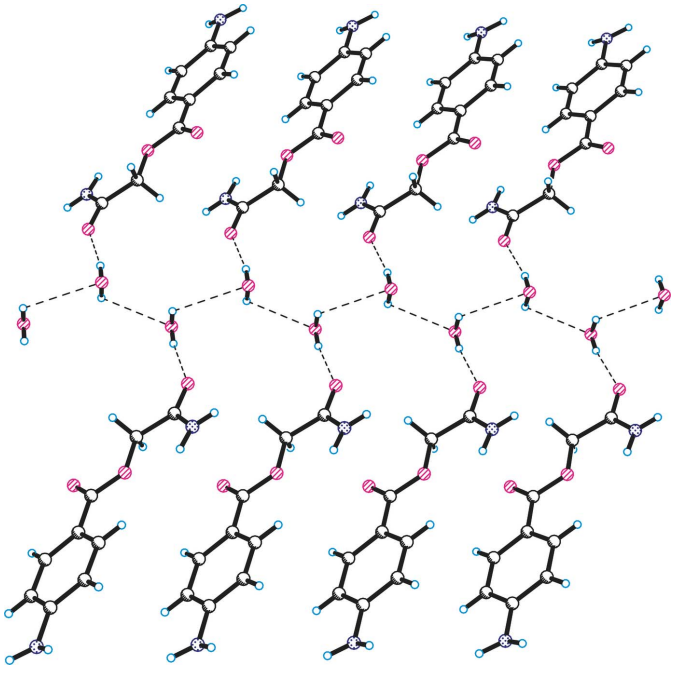


Figure 6
Intermolecular hydrogen bonds between water and 2-amino-2-oxoethyl 4-aminobenzoate molecules in **III**.

(2-Amino-2-oxoethyl)-4-nitrobenzoate. The reaction yield is 78%. R_F = 0.62 [in 5:1.5:1 (*v*/*v*) $CHCl_3/C_6H_6/CH_3OH$ solvent system]; m.p. 439–441 K. ^{1}H NMR [400 MHz, CD_3OD+$CDCl_3$+C_2D_5OD δ (ppm), *J* (Hz)]: 8.23 (2H, *d*, *J* = 9.28, H3 and H5), 8.19 (2H, *d*, *J* = 9.31, H2 and H6), 4.73 (2H, *s*, CH_2).

(2-Amino-2-oxoethyl)-4-aminobenzoate. The reaction yield is 88%. R_F = 0.53 [in 5:1.5:1 (*v*/*v*) $CHCl_3/C_6H_6/CH_3OH$ solvent system]; m.p. 435–438 K. ^{1}H NMR [400 MHz, CD_3OD, δ (ppm), *J* (Hz)]: 7.75 (2H, *d*, *J* = 8.75, H3 and H5), 6.58 (2H, *d*, *J* = 8.75, H2 and H6), 4.62 (2H, *s*, CH_2).

Each compound was dissolved in ethanol and the solvent allowed to evaporate at room temperature. Colourless crystals suitable for X-ray diffraction analysis were obtained.

The crystal of the 2-amino-2-oxoethyl 4-aminobenzoate monohydrate loses its transparency without chemical change (without becoming amorphous) in the range 344–346 K when the crystals are heated and melts in the range 435–438 K.

The yields of 2-amino-2-oxoethyl 4-bromobenzoate, $C_9H_8BrNO_3$, **I**, 2-amino-2-oxoethyl 4-nitrobenzoate, $C_9H_8N_2O_5$, **II**, and 2-amino-2-oxoethyl 4-aminobenzoate monohydrate, $C_9H_{10}N_2O_3{\cdot}H_2O$, **III**, are 86, 78 and 88%, respectively. The low yield of **II** is explained by the reduced reactivity of the molecule in a nucleophilic exchange reaction because of the negative induction and negative mesomeric effects of the nitro group on the benzene ring.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 7. C-bound H atoms were placed

Table 7
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	$C_9H_8BrNO_3$	$C_9H_8N_2O_5$	$C_9H_{10}N_2O_3{\cdot}H_2O$
M_r	258.07	224.17	212.21
Crystal system, space group	Monoclinic, $P2_1/n$	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/n$
Temperature (K)	291	291	291
a, b, c (Å)	18.623 (4), 4.8255 (10), 23.195 (5)	7.1238 (14), 7.3683 (15), 10.063 (2)	8.2431 (16), 4.8088 (10), 26.754 (5)
α, β, γ (°)	90, 112.96 (3), 90	107.82 (3), 94.95 (3), 96.32 (3)	90, 90.10 (3), 90
V (Å^3)	1919.3 (8)	495.76 (19)	1060.5 (4)
Z	8	2	4
Radiation type	Cu $K\alpha$	Cu $K\alpha$	Cu $K\alpha$
μ (mm^{-1})	5.71	1.08	0.90
Crystal size (mm)	0.60 × 0.20 × 0.15	0.40 × 0.34 × 0.21	0.28 × 0.24 × 0.17
Data collection			
Diffractometer	Oxford Diffraction Xcalibur, Ruby	Oxford Diffraction Xcalibur, Ruby	Oxford Diffraction Xcalibur, Ruby
Absorption correction	Multi-scan (*SADABS*; Bruker, 2008)	Multi-scan (*SADABS*; Bruker, 2008)	Multi-scan (*SADABS*; Bruker, 2008)
T_{min}, T_{max}	0.292, 0.425	0.681, 0.797	0.778, 0.859
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6390, 3832, 3165	2971, 1859, 1560	6444, 2165, 1129
R_{int}	0.033	0.018	0.055
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.629	0.609	0.630
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.049, 0.138, 1.06	0.050, 0.148, 1.06	0.053, 0.150, 0.99
No. of reflections	3832	1859	2165
No. of parameters	269	153	160
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.62, −0.85	0.24, −0.29	0.16, −0.24

Computer programs: *CrysAlis PRO* (Rigaku OD, 2018), *SHELXS97* (Sheldrick, 2008), *SHELXL2014/8* (Sheldrick, 2015), *XP* in *SHELXTL* (Sheldrick, 2008), *SHELXTL* (Sheldrick, 2008), *PLATON* (Spek, 2020) and *publCIF* (Westrip, 2010)'.

geometrically (with C—H distances of 0.97 Å for CH_2 and 0.93 Å for C_{ar}) and included in the refinement as riding contributions with $U_{iso}(H) = 1.2U_{eq}(C)$ [$U_{iso} = 1.5\ U_{eq}(C)$ for methyl H atoms]. The hydrogen atoms attached to N and O (water) were located in difference-Fourier maps and refined freely.

Acknowledgements

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supporting information

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Computing details

For all structures, data collection: *CrysAlis PRO* (Rigaku OD, 2018); cell refinement: *CrysAlis PRO* (Rigaku OD, 2018); data reduction: *CrysAlis PRO* (Rigaku OD, 2018); program(s) used to solve structure: *SHELXS7* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014/8* (Sheldrick, 2015); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008), *PLATON* (Spek, 2020) and *publCIF* (Westrip, 2010)′.

2-Amino-2-oxoethyl 4-bromobenzoate (I)

Crystal data

$C_9H_8BrNO_3$
M_r = 258.07
Monoclinic, $P2_1/n$
a = 18.623 (4) Å
b = 4.8255 (10) Å
c = 23.195 (5) Å
β = 112.96 (3)°
V = 1919.3 (8) Å^3
Z = 8
$F(000)$ = 1024
D_x = 1.786 Mg m^{-3}
Melting point: 475(2) K
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 2381 reflections
θ = 3.9–75.4°
μ = 5.71 mm^{-1}
T = 291 K
Prismatic, colorless
0.60 × 0.20 × 0.15 mm

Data collection

Oxford Diffraction Xcalibur, Ruby diffractometer
Radiation source: Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 10.2576 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2008)
T_{min} = 0.292, T_{max} = 0.425
6390 measured reflections
3832 independent reflections
3165 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 75.9°, θ_{min} = 3.9°
h = −20→22
k = −3→5
l = −28→28

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.138
S = 1.06
3832 reflections
269 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0772P)^2 + 0.3454P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.62$ e Å^{-3}
$\Delta\rho_{min} = -0.85$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1A	0.59795 (3)	0.16726 (12)	0.02621 (3)	0.06387 (18)
O1A	0.8638 (2)	1.0616 (8)	−0.01058 (15)	0.0605 (8)
O2A	0.89019 (17)	1.1199 (6)	0.09136 (15)	0.0503 (7)
O3A	1.03189 (18)	0.9293 (6)	0.09906 (17)	0.0565 (8)
N1A	1.0666 (2)	1.3499 (8)	0.0755 (2)	0.0562 (10)
C1A	0.7921 (2)	0.7970 (9)	0.03619 (17)	0.0415 (8)
C2A	0.7811 (2)	0.7232 (10)	0.09046 (18)	0.0480 (9)
H2AA	0.8124	0.8027	0.1285	0.058*
C3A	0.7248 (2)	0.5349 (10)	0.08845 (19)	0.0490 (9)
H3AA	0.7173	0.4880	0.1246	0.059*
C4A	0.6792 (2)	0.4160 (9)	0.03104 (19)	0.0448 (8)
C5A	0.6903 (3)	0.4759 (9)	−0.0231 (2)	0.0502 (10)
H5AA	0.6607	0.3889	−0.0606	0.060*
C6A	0.7466 (2)	0.6684 (10)	−0.02010 (18)	0.0490 (9)
H6AA	0.7544	0.7128	−0.0563	0.059*
C7A	0.8509 (2)	1.0019 (9)	0.03547 (19)	0.0446 (9)
C8A	0.9478 (2)	1.3163 (9)	0.0930 (2)	0.0513 (10)
H8AA	0.9637	1.4220	0.1316	0.062*
H8AB	0.9257	1.4444	0.0583	0.062*
C9A	1.0189 (2)	1.1759 (8)	0.08903 (19)	0.0423 (8)
Br1B	0.67568 (2)	0.97787 (9)	0.22125 (2)	0.05283 (17)
O1B	0.38919 (19)	−0.0158 (7)	0.13654 (16)	0.0566 (8)
O2B	0.40704 (17)	0.0563 (7)	0.23657 (14)	0.0499 (7)
O3B	0.2552 (2)	0.2216 (6)	0.17521 (19)	0.0599 (8)
N1B	0.2115 (2)	−0.2140 (8)	0.1638 (2)	0.0590 (10)
C1B	0.4827 (2)	0.3192 (8)	0.19559 (18)	0.0403 (8)
C2B	0.5228 (2)	0.4425 (8)	0.25363 (18)	0.0438 (8)
H2BA	0.5112	0.3927	0.2877	0.053*
C3B	0.5798 (2)	0.6380 (8)	0.26086 (18)	0.0431 (8)
H3BA	0.6070	0.7184	0.2998	0.052*
C4B	0.5961 (2)	0.7133 (8)	0.20969 (19)	0.0413 (8)
C5B	0.5564 (2)	0.5973 (9)	0.15118 (19)	0.0463 (9)
H5BA	0.5673	0.6527	0.1170	0.056*
C6B	0.5005 (2)	0.3988 (9)	0.14437 (18)	0.0451 (8)
H6BA	0.4743	0.3166	0.1055	0.054*

C7B	0.4222 (2)	0.1058 (9)	0.18490 (19)	0.0433 (8)
C8B	0.3457 (2)	−0.1403 (9)	0.2275 (2)	0.0499 (9)
H8BA	0.3449	−0.1889	0.2679	0.060*
H8BB	0.3562	−0.3078	0.2090	0.060*
C9B	0.2666 (2)	−0.0281 (8)	0.1857 (2)	0.0430 (8)
H1A	1.064 (4)	1.527 (14)	0.074 (3)	0.070 (18)*
H2A	1.107 (3)	1.286 (12)	0.067 (2)	0.058 (14)*
H1B	0.160 (4)	−0.159 (12)	0.142 (3)	0.064 (16)*
H2B	0.225 (5)	−0.385 (19)	0.167 (4)	0.11 (3)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1A	0.0482 (3)	0.0654 (3)	0.0756 (3)	−0.0109 (2)	0.0215 (2)	−0.0061 (2)
O1A	0.0569 (19)	0.074 (2)	0.0558 (17)	0.0015 (16)	0.0275 (14)	0.0065 (16)
O2A	0.0425 (15)	0.0511 (16)	0.0623 (16)	−0.0055 (12)	0.0258 (13)	−0.0114 (14)
O3A	0.0476 (16)	0.0373 (15)	0.087 (2)	0.0071 (13)	0.0286 (16)	0.0083 (15)
N1A	0.0413 (19)	0.039 (2)	0.096 (3)	0.0026 (15)	0.0345 (19)	0.0011 (19)
C1A	0.0327 (17)	0.048 (2)	0.0439 (17)	0.0086 (15)	0.0146 (14)	−0.0008 (16)
C2A	0.042 (2)	0.057 (2)	0.0425 (18)	0.0013 (18)	0.0139 (15)	−0.0077 (17)
C3A	0.043 (2)	0.059 (3)	0.0448 (19)	0.0018 (18)	0.0166 (16)	−0.0040 (18)
C4A	0.0346 (18)	0.041 (2)	0.054 (2)	−0.0005 (15)	0.0117 (15)	−0.0046 (17)
C5A	0.045 (2)	0.053 (2)	0.048 (2)	0.0029 (18)	0.0125 (17)	−0.0100 (18)
C6A	0.041 (2)	0.061 (3)	0.0414 (18)	0.0027 (18)	0.0131 (15)	−0.0046 (18)
C7A	0.0373 (19)	0.045 (2)	0.053 (2)	0.0099 (16)	0.0200 (16)	0.0010 (17)
C8A	0.039 (2)	0.043 (2)	0.077 (3)	−0.0009 (16)	0.0283 (19)	−0.010 (2)
C9A	0.0335 (17)	0.038 (2)	0.0526 (19)	0.0053 (14)	0.0141 (15)	−0.0037 (16)
Br1B	0.0354 (2)	0.0468 (3)	0.0738 (3)	−0.00022 (16)	0.01852 (19)	0.0072 (2)
O1B	0.0513 (17)	0.0560 (18)	0.0640 (18)	−0.0109 (14)	0.0240 (14)	−0.0166 (15)
O2B	0.0429 (15)	0.0532 (16)	0.0554 (15)	−0.0074 (13)	0.0211 (12)	−0.0041 (13)
O3B	0.0531 (17)	0.0311 (15)	0.097 (2)	0.0056 (13)	0.0305 (17)	0.0047 (15)
N1B	0.048 (2)	0.0341 (19)	0.080 (3)	0.0025 (15)	0.0084 (18)	0.0062 (18)
C1B	0.0310 (16)	0.0385 (19)	0.0511 (19)	0.0056 (14)	0.0155 (14)	−0.0013 (15)
C2B	0.0410 (19)	0.044 (2)	0.0453 (18)	0.0022 (16)	0.0157 (15)	0.0024 (16)
C3B	0.0401 (19)	0.040 (2)	0.0459 (18)	−0.0042 (15)	0.0136 (15)	−0.0016 (15)
C4B	0.0254 (15)	0.0373 (18)	0.056 (2)	0.0002 (13)	0.0106 (14)	0.0024 (16)
C5B	0.0371 (18)	0.054 (2)	0.0483 (19)	0.0046 (17)	0.0166 (15)	0.0018 (17)
C6B	0.0350 (17)	0.052 (2)	0.0453 (18)	0.0046 (16)	0.0124 (14)	−0.0055 (17)
C7B	0.0327 (17)	0.042 (2)	0.054 (2)	0.0067 (15)	0.0152 (15)	−0.0038 (17)
C8B	0.039 (2)	0.044 (2)	0.067 (2)	−0.0004 (16)	0.0206 (18)	0.0046 (19)
C9B	0.043 (2)	0.0319 (19)	0.061 (2)	0.0028 (15)	0.0282 (17)	0.0023 (16)

Geometric parameters (Å, º)

Br1A—C4A	1.900 (4)	Br1B—C4B	1.894 (4)
O1A—C7A	1.217 (6)	O1B—C7B	1.201 (5)
O2A—C7A	1.342 (5)	O2B—C7B	1.355 (5)
O2A—C8A	1.421 (5)	O2B—C8B	1.437 (5)

O3A—C9A	1.219 (5)	O3B—C9B	1.231 (5)
N1A—C9A	1.345 (6)	N1B—C9B	1.307 (6)
N1A—H1A	0.85 (7)	N1B—H1B	0.93 (6)
N1A—H2A	0.91 (6)	N1B—H2B	0.86 (9)
C1A—C6A	1.394 (5)	C1B—C2B	1.393 (6)
C1A—C2A	1.398 (6)	C1B—C6B	1.406 (6)
C1A—C7A	1.480 (6)	C1B—C7B	1.474 (6)
C2A—C3A	1.374 (6)	C2B—C3B	1.381 (6)
C2A—H2AA	0.9300	C2B—H2BA	0.9300
C3A—C4A	1.394 (6)	C3B—C4B	1.383 (6)
C3A—H3AA	0.9300	C3B—H3BA	0.9300
C4A—C5A	1.380 (6)	C4B—C5B	1.385 (6)
C5A—C6A	1.382 (7)	C5B—C6B	1.378 (6)
C5A—H5AA	0.9300	C5B—H5BA	0.9300
C6A—H6AA	0.9300	C6B—H6BA	0.9300
C8A—C9A	1.522 (5)	C8B—C9B	1.512 (6)
C8A—H8AA	0.9700	C8B—H8BA	0.9700
C8A—H8AB	0.9700	C8B—H8BB	0.9700

C7A—O2A—C8A	115.5 (3)	C7B—O2B—C8B	114.5 (3)
C9A—N1A—H1A	127 (4)	C9B—N1B—H1B	120 (4)
C9A—N1A—H2A	121 (4)	C9B—N1B—H2B	117 (5)
H1A—N1A—H2A	111 (6)	H1B—N1B—H2B	122 (6)
C6A—C1A—C2A	118.7 (4)	C2B—C1B—C6B	119.1 (4)
C6A—C1A—C7A	117.9 (4)	C2B—C1B—C7B	123.2 (4)
C2A—C1A—C7A	123.3 (4)	C6B—C1B—C7B	117.7 (4)
C3A—C2A—C1A	121.1 (4)	C3B—C2B—C1B	120.4 (4)
C3A—C2A—H2AA	119.5	C3B—C2B—H2BA	119.8
C1A—C2A—H2AA	119.5	C1B—C2B—H2BA	119.8
C2A—C3A—C4A	118.5 (4)	C2B—C3B—C4B	119.4 (4)
C2A—C3A—H3AA	120.7	C2B—C3B—H3BA	120.3
C4A—C3A—H3AA	120.7	C4B—C3B—H3BA	120.3
C5A—C4A—C3A	122.1 (4)	C3B—C4B—C5B	121.5 (4)
C5A—C4A—Br1A	118.5 (3)	C3B—C4B—Br1B	118.5 (3)
C3A—C4A—Br1A	119.4 (3)	C5B—C4B—Br1B	120.0 (3)
C4A—C5A—C6A	118.4 (4)	C6B—C5B—C4B	119.0 (4)
C4A—C5A—H5AA	120.8	C6B—C5B—H5BA	120.5
C6A—C5A—H5AA	120.8	C4B—C5B—H5BA	120.5
C5A—C6A—C1A	121.2 (4)	C5B—C6B—C1B	120.7 (4)
C5A—C6A—H6AA	119.4	C5B—C6B—H6BA	119.7
C1A—C6A—H6AA	119.4	C1B—C6B—H6BA	119.7
O1A—C7A—O2A	121.8 (4)	O1B—C7B—O2B	122.2 (4)
O1A—C7A—C1A	124.6 (4)	O1B—C7B—C1B	125.3 (4)
O2A—C7A—C1A	113.6 (4)	O2B—C7B—C1B	112.6 (3)
O2A—C8A—C9A	111.5 (3)	O2B—C8B—C9B	112.1 (3)
O2A—C8A—H8AA	109.3	O2B—C8B—H8BA	109.2
C9A—C8A—H8AA	109.3	C9B—C8B—H8BA	109.2
O2A—C8A—H8AB	109.3	O2B—C8B—H8BB	109.2

C9A—C8A—H8AB	109.3	C9B—C8B—H8BB	109.2
H8AA—C8A—H8AB	108.0	H8BA—C8B—H8BB	107.9
O3A—C9A—N1A	123.7 (4)	O3B—C9B—N1B	123.1 (4)
O3A—C9A—C8A	122.4 (4)	O3B—C9B—C8B	121.8 (4)
N1A—C9A—C8A	113.9 (4)	N1B—C9B—C8B	115.1 (4)
C6A—C1A—C2A—C3A	−2.3 (6)	C6B—C1B—C2B—C3B	−0.6 (6)
C7A—C1A—C2A—C3A	178.9 (4)	C7B—C1B—C2B—C3B	178.9 (4)
C1A—C2A—C3A—C4A	0.7 (7)	C1B—C2B—C3B—C4B	0.8 (6)
C2A—C3A—C4A—C5A	1.8 (7)	C2B—C3B—C4B—C5B	0.1 (6)
C2A—C3A—C4A—Br1A	−177.3 (3)	C2B—C3B—C4B—Br1B	−179.1 (3)
C3A—C4A—C5A—C6A	−2.6 (6)	C3B—C4B—C5B—C6B	−1.3 (6)
Br1A—C4A—C5A—C6A	176.5 (3)	Br1B—C4B—C5B—C6B	177.9 (3)
C4A—C5A—C6A—C1A	0.9 (6)	C4B—C5B—C6B—C1B	1.4 (6)
C2A—C1A—C6A—C5A	1.5 (6)	C2B—C1B—C6B—C5B	−0.5 (6)
C7A—C1A—C6A—C5A	−179.7 (4)	C7B—C1B—C6B—C5B	179.9 (4)
C8A—O2A—C7A—O1A	−0.4 (6)	C8B—O2B—C7B—O1B	−3.3 (6)
C8A—O2A—C7A—C1A	179.4 (3)	C8B—O2B—C7B—C1B	177.1 (3)
C6A—C1A—C7A—O1A	−1.9 (6)	C2B—C1B—C7B—O1B	−175.9 (4)
C2A—C1A—C7A—O1A	176.9 (4)	C6B—C1B—C7B—O1B	3.7 (6)
C6A—C1A—C7A—O2A	178.3 (3)	C2B—C1B—C7B—O2B	3.7 (5)
C2A—C1A—C7A—O2A	−3.0 (5)	C6B—C1B—C7B—O2B	−176.7 (3)
C7A—O2A—C8A—C9A	−72.9 (5)	C7B—O2B—C8B—C9B	−69.1 (5)
O2A—C8A—C9A—O3A	−16.9 (6)	O2B—C8B—C9B—O3B	−17.6 (6)
O2A—C8A—C9A—N1A	165.0 (4)	O2B—C8B—C9B—N1B	164.7 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H1*A*···O3*A*^{i}	0.85 (6)	2.18 (7)	2.969 (5)	153
N1*B*—H1*B*···O3*A*^{ii}	0.93 (6)	2.24 (6)	3.163 (5)	170
N1*B*—H2*B*···O3*B*^{iii}	0.86 (9)	1.97 (9)	2.825 (5)	178

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

2-Amino-2-oxoethyl 4-nitrobenzoate (II)

Crystal data

$C_9H_8N_2O_5$
M_r = 224.17
Triclinic, $P\overline{1}$
a = 7.1238 (14) Å
b = 7.3683 (15) Å
c = 10.063 (2) Å
α = 107.82 (3)°
β = 94.95 (3)°
γ = 96.32 (3)°
V = 495.76 (19) Å^3
Z = 2

F(000) = 232
D_x = 1.502 Mg m^{-3}
Melting point: 439(2) K
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 1249 reflections
θ = 4.6–75.6°
μ = 1.08 mm^{-1}
T = 291 K
Prismatic, colorless
0.40 × 0.34 × 0.21 mm

Data collection

Oxford Diffraction Xcalibur, Ruby diffractometer
Radiation source: Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 10.2576 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2008)
T_{min} = 0.681, T_{max} = 0.797
2971 measured reflections
1859 independent reflections
1560 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 70.0°, θ_{min} = 4.7°
h = −5→8
k = −8→8
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.148
S = 1.06
1859 reflections
153 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.080P)^2 + 0.1088P]$ 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}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.0727 (3)	−0.1039 (2)	0.29955 (18)	0.0820 (6)
O2	0.19162 (19)	−0.00610 (18)	0.13179 (13)	0.0492 (4)
O3	0.2532 (2)	−0.49315 (19)	−0.04910 (17)	0.0635 (5)
O4	0.3147 (3)	0.8233 (2)	0.75383 (18)	0.0866 (6)
O5	0.3833 (3)	0.9205 (2)	0.58393 (19)	0.0852 (6)
N1	0.4638 (3)	−0.2474 (3)	0.0945 (2)	0.0608 (5)
N2	0.3258 (2)	0.7948 (2)	0.62962 (19)	0.0526 (4)
C1	0.1925 (2)	0.2274 (2)	0.35306 (18)	0.0409 (4)
C2	0.2520 (3)	0.3754 (3)	0.30129 (19)	0.0443 (4)
H2A	0.2634	0.3488	0.2061	0.053*
C3	0.2945 (3)	0.5635 (3)	0.3920 (2)	0.0466 (4)
H3A	0.3342	0.6645	0.3590	0.056*
C4	0.2762 (2)	0.5962 (2)	0.53229 (19)	0.0425 (4)
C5	0.2171 (3)	0.4526 (3)	0.58712 (19)	0.0465 (4)
H5A	0.2065	0.4799	0.6825	0.056*
C6	0.1739 (3)	0.2665 (3)	0.4952 (2)	0.0466 (4)
H6A	0.1321	0.1666	0.5287	0.056*
C7	0.1433 (3)	0.0225 (3)	0.2615 (2)	0.0465 (4)
C8	0.1380 (3)	−0.1993 (3)	0.0349 (2)	0.0509 (5)

H8A	0.0999	−0.1916	−0.0579	0.061*
H8B	0.0285	−0.2604	0.0640	0.061*
C9	0.2934 (3)	−0.3240 (2)	0.02512 (19)	0.0455 (4)
H1	0.561 (3)	−0.323 (3)	0.082 (2)	0.057 (6)*
H2	0.488 (3)	−0.127 (4)	0.138 (3)	0.062 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1300 (16)	0.0426 (9)	0.0620 (10)	−0.0163 (9)	0.0267 (10)	0.0061 (7)
O2	0.0603 (8)	0.0354 (7)	0.0467 (7)	0.0097 (5)	0.0122 (6)	0.0032 (5)
O3	0.0579 (8)	0.0369 (7)	0.0759 (10)	0.0070 (6)	−0.0010 (7)	−0.0085 (7)
O4	0.1315 (16)	0.0536 (10)	0.0537 (10)	−0.0086 (10)	0.0189 (10)	−0.0075 (7)
O5	0.1262 (16)	0.0381 (8)	0.0797 (12)	−0.0066 (9)	0.0143 (11)	0.0079 (8)
N1	0.0543 (10)	0.0380 (9)	0.0710 (12)	0.0099 (8)	−0.0013 (8)	−0.0091 (8)
N2	0.0515 (9)	0.0380 (9)	0.0591 (10)	0.0061 (7)	0.0043 (7)	0.0030 (7)
C1	0.0403 (8)	0.0353 (9)	0.0443 (9)	0.0092 (7)	0.0064 (7)	0.0073 (7)
C2	0.0510 (10)	0.0394 (9)	0.0414 (9)	0.0097 (7)	0.0086 (7)	0.0094 (7)
C3	0.0522 (10)	0.0352 (9)	0.0528 (10)	0.0077 (7)	0.0077 (8)	0.0139 (8)
C4	0.0386 (8)	0.0343 (9)	0.0481 (10)	0.0078 (7)	0.0035 (7)	0.0032 (7)
C5	0.0504 (10)	0.0439 (10)	0.0406 (9)	0.0062 (7)	0.0086 (7)	0.0060 (7)
C6	0.0530 (10)	0.0385 (9)	0.0471 (10)	0.0045 (7)	0.0106 (8)	0.0116 (8)
C7	0.0527 (10)	0.0369 (10)	0.0464 (10)	0.0060 (7)	0.0078 (8)	0.0080 (8)
C8	0.0552 (11)	0.0402 (10)	0.0467 (10)	0.0088 (8)	0.0023 (8)	−0.0011 (8)
C9	0.0526 (10)	0.0347 (9)	0.0426 (9)	0.0053 (7)	0.0066 (7)	0.0027 (7)

Geometric parameters (Å, º)

O1—C7	1.190 (2)	C1—C7	1.492 (3)
O2—C7	1.339 (2)	C2—C3	1.390 (3)
O2—C8	1.445 (2)	C2—H2A	0.9300
O3—C9	1.228 (2)	C3—C4	1.378 (3)
O4—N2	1.213 (2)	C3—H3A	0.9300
O5—N2	1.203 (2)	C4—C5	1.379 (3)
N1—C9	1.320 (3)	C5—C6	1.383 (3)
N1—H1	0.93 (2)	C5—H5A	0.9300
N1—H2	0.85 (3)	C6—H6A	0.9300
N2—C4	1.474 (2)	C8—C9	1.506 (3)
C1—C2	1.387 (3)	C8—H8A	0.9700
C1—C6	1.391 (3)	C8—H8B	0.9700
C7—O2—C8	115.74 (15)	C4—C5—C6	117.62 (17)
C9—N1—H1	118.2 (14)	C4—C5—H5A	121.2
C9—N1—H2	120.2 (16)	C6—C5—H5A	121.2
H1—N1—H2	121 (2)	C5—C6—C1	120.57 (17)
O5—N2—O4	122.58 (18)	C5—C6—H6A	119.7
O5—N2—C4	118.83 (17)	C1—C6—H6A	119.7
O4—N2—C4	118.52 (17)	O1—C7—O2	123.05 (17)

C2—C1—C6	120.33 (17)	O1—C7—C1	124.14 (17)
C2—C1—C7	122.69 (16)	O2—C7—C1	112.80 (16)
C6—C1—C7	116.98 (16)	O2—C8—C9	114.12 (15)
C1—C2—C3	119.85 (17)	O2—C8—H8A	108.7
C1—C2—H2A	120.1	C9—C8—H8A	108.7
C3—C2—H2A	120.1	O2—C8—H8B	108.7
C4—C3—C2	118.12 (17)	C9—C8—H8B	108.7
C4—C3—H3A	120.9	H8A—C8—H8B	107.6
C2—C3—H3A	120.9	O3—C9—N1	123.64 (18)
C3—C4—C5	123.50 (16)	O3—C9—C8	117.23 (17)
C3—C4—N2	118.35 (17)	N1—C9—C8	119.14 (16)
C5—C4—N2	118.14 (17)		

C6—C1—C2—C3	0.4 (3)	C2—C1—C6—C5	−0.9 (3)
C7—C1—C2—C3	179.61 (16)	C7—C1—C6—C5	179.83 (16)
C1—C2—C3—C4	0.2 (3)	C8—O2—C7—O1	5.0 (3)
C2—C3—C4—C5	−0.4 (3)	C8—O2—C7—C1	−176.52 (14)
C2—C3—C4—N2	178.47 (15)	C2—C1—C7—O1	−171.5 (2)
O5—N2—C4—C3	−1.2 (3)	C6—C1—C7—O1	7.7 (3)
O4—N2—C4—C3	−178.06 (18)	C2—C1—C7—O2	10.0 (3)
O5—N2—C4—C5	177.72 (18)	C6—C1—C7—O2	−170.76 (15)
O4—N2—C4—C5	0.8 (3)	C7—O2—C8—C9	−95.53 (19)
C3—C4—C5—C6	−0.1 (3)	O2—C8—C9—O3	175.79 (17)
N2—C4—C5—C6	−178.96 (15)	O2—C8—C9—N1	−4.7 (3)
C4—C5—C6—C1	0.8 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3^{i}	0.93 (2)	1.97 (2)	2.898 (2)	174 (2)

Symmetry code: (i) −*x*+1, −*y*−1, −*z*.

2-Amino-2-oxoethyl 4-aminobenzoate monohydrate (III)

Crystal data

$C_9H_{10}N_2O_3·H_2O$
M_r = 212.21
Monoclinic, $P2_1/n$
a = 8.2431 (16) Å
b = 4.8088 (10) Å
c = 26.754 (5) Å
β = 90.10 (3)°
V = 1060.5 (4) Å^3
Z = 4
$F(000)$ = 448
D_x = 1.329 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 927 reflections
θ = 5.6–71.4°
μ = 0.90 mm^{-1}
T = 291 K
Prismatic, colorless
0.28 × 0.24 × 0.17 mm

Data collection

Oxford Diffraction Xcalibur, Ruby diffractometer
Radiation source: Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 10.2576 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2008)

T_{min} = 0.778, T_{max} = 0.859
6444 measured reflections
2165 independent reflections
1129 reflections with $I > 2\sigma(I)$
R_{int} = 0.055

θ_{max} = 76.1°, θ_{min} = 3.3°
h = −10→10
k = −4→5
l = −32→33

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.150
S = 0.99
2165 reflections
160 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.055P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.4903 (2)	0.0764 (5)	0.65718 (8)	0.0717 (6)
O2	0.3607 (2)	0.3784 (4)	0.60830 (7)	0.0589 (5)
O3	0.6302 (2)	0.8021 (4)	0.53839 (9)	0.0729 (6)
N1	0.3578 (3)	0.7857 (6)	0.54142 (11)	0.0633 (7)
N2	−0.2461 (3)	−0.2041 (6)	0.70993 (11)	0.0691 (8)
C1	0.2029 (3)	0.0794 (5)	0.65846 (10)	0.0496 (6)
C2	0.0592 (3)	0.1846 (6)	0.63861 (11)	0.0626 (8)
H2A	0.0631	0.3213	0.6141	0.075*
C3	−0.0884 (3)	0.0879 (6)	0.65503 (11)	0.0641 (8)
H3A	−0.1833	0.1587	0.6411	0.077*
C4	−0.0982 (3)	−0.1143 (6)	0.69216 (10)	0.0540 (7)
C5	0.0452 (3)	−0.2176 (6)	0.71203 (11)	0.0592 (7)
H5A	0.0413	−0.3527	0.7369	0.071*
C6	0.1923 (3)	−0.1237 (6)	0.69558 (11)	0.0583 (7)
H6A	0.2869	−0.1963	0.7094	0.070*
C7	0.3628 (3)	0.1724 (6)	0.64262 (10)	0.0531 (7)
C8	0.5179 (3)	0.4682 (6)	0.59167 (11)	0.0598 (7)
H8A	0.5748	0.3129	0.5766	0.072*
H8B	0.5809	0.5319	0.6201	0.072*
C9	0.5026 (3)	0.6994 (6)	0.55425 (10)	0.0549 (7)
O1W	0.9804 (4)	0.7452 (7)	0.52690 (16)	0.1083 (11)
H1	0.348 (4)	0.917 (8)	0.5195 (15)	0.107 (14)*
H2	0.264 (4)	0.697 (7)	0.5524 (12)	0.088 (11)*
H3	−0.337 (4)	−0.147 (6)	0.6897 (12)	0.078 (10)*

H4	−0.253 (4)	−0.365 (8)	0.7275 (15)	0.108 (14)*
H1W	0.887 (5)	0.743 (8)	0.5326 (16)	0.100 (16)*
H2W	0.939 (13)	0.731 (19)	0.495 (3)	0.35 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0530 (12)	0.0881 (16)	0.0740 (14)	0.0039 (11)	0.0000 (10)	0.0249 (12)
O2	0.0543 (11)	0.0566 (11)	0.0659 (12)	0.0019 (9)	0.0084 (9)	0.0131 (9)
O3	0.0613 (13)	0.0704 (14)	0.0872 (15)	−0.0025 (11)	0.0146 (11)	0.0193 (12)
N1	0.0587 (16)	0.0643 (17)	0.0669 (17)	−0.0031 (13)	0.0020 (13)	0.0124 (13)
N2	0.0517 (15)	0.0773 (19)	0.0784 (19)	−0.0002 (14)	0.0039 (13)	0.0163 (15)
C1	0.0502 (15)	0.0501 (15)	0.0487 (14)	0.0018 (12)	0.0004 (11)	0.0015 (12)
C2	0.0592 (17)	0.0656 (19)	0.0632 (18)	0.0069 (14)	0.0038 (14)	0.0195 (15)
C3	0.0530 (17)	0.069 (2)	0.0699 (19)	0.0072 (14)	−0.0018 (14)	0.0149 (15)
C4	0.0531 (16)	0.0515 (15)	0.0572 (16)	0.0022 (13)	0.0046 (13)	0.0000 (13)
C5	0.0612 (18)	0.0587 (17)	0.0577 (17)	0.0028 (14)	0.0018 (14)	0.0136 (13)
C6	0.0532 (16)	0.0612 (17)	0.0603 (17)	0.0052 (14)	−0.0049 (13)	0.0077 (14)
C7	0.0569 (16)	0.0541 (16)	0.0483 (14)	0.0035 (13)	0.0044 (12)	−0.0002 (12)
C8	0.0555 (17)	0.0547 (17)	0.0694 (19)	0.0013 (13)	0.0076 (14)	0.0014 (14)
C9	0.0591 (17)	0.0485 (15)	0.0571 (16)	−0.0005 (13)	0.0087 (13)	−0.0012 (13)
O1W	0.0720 (19)	0.112 (2)	0.141 (3)	−0.0013 (17)	0.0223 (19)	−0.017 (2)

Geometric parameters (Å, º)

O1—C7	1.212 (3)	C2—C3	1.376 (4)
O2—C7	1.351 (3)	C2—H2A	0.9300
O2—C8	1.437 (3)	C3—C4	1.393 (4)
O3—O3	0.000 (7)	C3—H3A	0.9300
O3—C9	1.238 (3)	C4—C5	1.388 (4)
N1—C9	1.309 (4)	C5—C6	1.368 (4)
N1—H1	0.87 (4)	C5—H5A	0.9300
N1—H2	0.93 (4)	C6—H6A	0.9300
N2—C4	1.378 (4)	C8—C9	1.501 (4)
N2—H3	0.96 (3)	C8—H8A	0.9700
N2—H4	0.91 (4)	C8—H8B	0.9700
C1—C2	1.393 (4)	C9—O3	1.238 (3)
C1—C6	1.396 (4)	O1W—H1W	0.78 (4)
C1—C7	1.455 (4)	O1W—H2W	0.91 (9)
C7—O2—C8	114.9 (2)	C6—C5—H5A	119.5
C9—N1—H1	120 (2)	C4—C5—H5A	119.5
C9—N1—H2	122 (2)	C5—C6—C1	121.1 (3)
H1—N1—H2	118 (3)	C5—C6—H6A	119.4
C4—N2—H3	113.9 (19)	C1—C6—H6A	119.4
C4—N2—H4	120 (2)	O1—C7—O2	120.5 (3)
H3—N2—H4	119 (3)	O1—C7—C1	125.1 (3)
C2—C1—C6	118.1 (2)	O2—C7—C1	114.4 (2)

C2—C1—C7	123.2 (2)	O2—C8—C9	110.8 (2)
C6—C1—C7	118.7 (2)	O2—C8—H8A	109.5
C3—C2—C1	120.5 (3)	C9—C8—H8A	109.5
C3—C2—H2A	119.7	O2—C8—H8B	109.5
C1—C2—H2A	119.7	C9—C8—H8B	109.5
C2—C3—C4	121.1 (3)	H8A—C8—H8B	108.1
C2—C3—H3A	119.5	O3—C9—N1	124.0 (3)
C4—C3—H3A	119.5	O3—C9—N1	124.0 (3)
N2—C4—C5	120.6 (3)	O3—C9—C8	117.0 (3)
N2—C4—C3	121.1 (3)	O3—C9—C8	117.0 (3)
C5—C4—C3	118.2 (3)	N1—C9—C8	119.0 (3)
C6—C5—C4	120.9 (3)	H1W—O1W—H2W	79 (7)
C6—C1—C2—C3	−0.6 (5)	C8—O2—C7—O1	−0.1 (4)
C7—C1—C2—C3	179.9 (3)	C8—O2—C7—C1	−178.9 (2)
C1—C2—C3—C4	0.8 (5)	C2—C1—C7—O1	−176.2 (3)
C2—C3—C4—N2	177.6 (3)	C6—C1—C7—O1	4.3 (4)
C2—C3—C4—C5	−0.4 (5)	C2—C1—C7—O2	2.5 (4)
N2—C4—C5—C6	−178.1 (3)	C6—C1—C7—O2	−177.0 (2)
C3—C4—C5—C6	0.0 (4)	C7—O2—C8—C9	−179.2 (2)
C4—C5—C6—C1	0.2 (5)	O2—C8—C9—O3	177.4 (2)
C2—C1—C6—C5	0.1 (4)	O2—C8—C9—N1	−0.6 (4)
C7—C1—C6—C5	179.7 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3^{i}	0.87 (4)	2.06 (4)	2.915 (3)	168
N2—H3···O1^{ii}	0.96 (3)	1.98 (3)	2.919 (4)	163
O1*W*—H1*W*···O3	0.78 (4)	2.14 (4)	2.916 (4)	169 (4)
O1*W*—H2*W*···O1*W*^{iii}	0.91 (9)	2.46 (9)	2.782 (7)	101

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