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3-Acetamido-5-nitrobenzyl acetate

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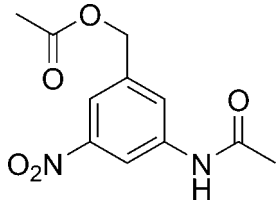
Key indicators: single-crystal X-ray study; T = 89 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.110; data-to-parameter ratio = 13.8.

The title compound, $C_{11}H_{12}N_2O_5$, was prepared by the reaction of 3-amino-5-nitrobenzyl alcohol with acetic anhydride. The asymmetric unit contains three independent molecules which differ in geometry only by their rotation about the single bonds external to the benzene ring. The title compound is an intermediate in the synthesis of DNA minor-groove-binding polybenzamide agents.

Related literature

For related literature on the biological activity of polybenzamide DNA binding agents, see: Storl *et al.* (1993). For related literature on natural and synthetic minor-groove-binding agents, see: Arcamone *et al.* (1964); Atwell *et al.* (1995); Baraldi *et al.* (1999, 2004, 2007); Turner *et al.* (1999); Wemmer (2000); Yan *et al.* (1997).



Experimental

Crystal data

$C_{11}H_{12}N_2O_5$
M_r = 252.23
Monoclinic, $P2_1/n$
a = 10.5303 (2) Å
b = 21.2894 (2) Å
c = 15.5410 (2) Å
β = 105.508 (1)°
V = 3357.20 (8) Å^3
Z = 12
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 89 (2) K
0.42 × 0.28 × 0.22 mm

Data collection

Siemens SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.846, T_{max} = 0.979
19573 measured reflections
6805 independent reflections
5399 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.110
S = 1.04
6805 reflections
493 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1A—H1A···O3A^{i}	0.86	2.35	3.1897 (17)	166
N1B—H1B···O3B^{ii}	0.86	2.32	3.1506 (16)	164
N1C—H1C···O3C^{i}	0.86	2.31	3.1458 (17)	164
C2A—H2A···O2A^{i}	0.93	2.54	3.4345 (18)	161
C2B—H2B···O2B^{ii}	0.93	2.49	3.4046 (18)	169
C2C—H2C···O2C^{i}	0.93	2.56	3.4546 (18)	161

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

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXTL* (Siemens, 1995).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2042).

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supplementary materials

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3-Acetamido-5-nitrobenzyl acetate

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Comment

The naturally occurring antibiotic oligopeptides distamycin A, isolated from *Streptomyces Distallicus*, and netropsin, from *Streptomyces netropsis*, are powerful DNA minor groove-binding agents but their cytotoxity precludes their use as medicines (Arcamone *et al.*,1964, Baraldi *et al.*, 2004, Wemmer, 2000, Storl *et al.*, 1993). In order to increase the DNA binding affinity and sequence specifity along with minimizing the unwanted physiological activities associated with these natural DNA binders, many synthetic oligopeptides have been prepared (Baraldi *et al.*, 2007). The title compound is a key intermediate required in the synthesis of a novel polybenzamide DNA minor groove-binding agent. For background information on polybenzamide DNA binding agents see (Atwell *et al.*, 1995, Turner *et al.*, 1999, Yan *et al.*, 1997).

Experimental

To a solution of 3-amino-5-nitrobenzyl alcohol (1 g, 5.95 mmol) in DMF (25 ml) was added acetic anhydride (1.69 ml, 17.84 mmol) and triethylamine (3.34 ml, 23.79 mmol), and the resulting mixture was stirred at room temperature for 24 h. Water (75 ml) was added, and the mixture stirred for 10 minutes. The resultant precipitate was filtered, and dried *in vacuo*, to afford a yellow solid, which was recrystallized from ethyl acetate to give the title compound (1.34 g, 90%), as yellow crystals suitable for X-ray crystallography (m.p. 430–431 K). Spectroscopic analysis. IR (ν_{max}, thin film, cm^{-1}) 3367, 1742, 1547, 1242. ^{1}H NMR (400 MHz, $CDCl_3$, δ, p.p.m.) 2.15 (3*H*, s, OCOC*H*$_3$), 2.19 (3*H*, s, NHCOCH_3, 5.15 (2*H*, s, CH_2OAc), 7.89 (1*H*, s, Ar—H), 8.15 (1*H*, s, Ar—H), 8.34 (1*H*, s, Ar—H) and 9.44 (NH). ^{13}C NMR (100 MHz, $CDCl_3$, δ, p.p.m.) 20.8 (CH_3, OCO*C*H_3), 24.3 (CH_3, NHCO*C*H_3), 64.9 (CH_2, *C*H_2OAc), 113.7 (CH, Ar—C) 117.1 (CH, Ar—C), 124.4 (CH, Ar—C), 138.4 (quat. Ar—C), 140.2 (quat. Ar—C), 148.4 (quat. Ar—C), 169.4 (C=O, NHAc) and 170.5 (C=O, OAc). MS *m/z* (EI) 252 (M^+, 12%), 210 (M^+—C_2H_2O, 33), 168 (M^+—$C_4H_4O_2$, 52), 43 ($COCH_3$, 100). HRMS (EI), found: M^+ 252.07469. $C_{11}H_{12}N_2O_5$ requires: 252.07462.

Refinement

Hydrogen atoms were placed in calculated positions and refined using the riding model [C—H 0.93–0.97, N—H 0.86 Å), with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(C_{methyl}).

Figures

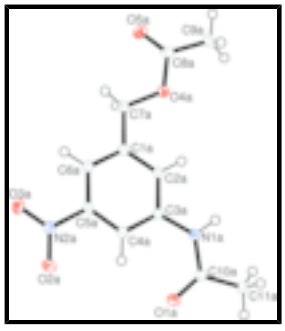


Fig. 1. Structure of molecule A showing 50% probability displacement ellipsoids for non-hydrogen atoms and hydrogen atoms as arbitary spheres (Burnett & Johnson, 1996).

3-Acetamido-5-nitrobenzyl acetate

Crystal data

$C_{11}H_{12}N_2O_5$	F_{000} = 1584
M_r = 252.23	D_x = 1.497 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 5612 reflections
a = 10.5303 (2) Å	θ = 1.9–26.3º
b = 21.2894 (2) Å	μ = 0.12 mm^{-1}
c = 15.5410 (2) Å	T = 89 (2) K
β = 105.508 (1)º	Triangular plate, yellow
V = 3357.20 (8) Å^3	0.42 × 0.28 × 0.22 mm
Z = 12	

Data collection

Siemens SMART CCD diffractometer	6805 independent reflections
Radiation source: fine-focus sealed tube	5399 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.019
T = 89(2) K	θ_{max} = 26.3º
Area detector ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (SADABS; Sheldrick, 1997)	h = −13→12
T_{min} = 0.846, T_{max} = 0.979	k = 0→26
19573 measured reflections	l = 0→19

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.041	H-atom parameters constrained

$wR(F^2) = 0.110$	$w = 1/[\sigma^2(F_o^2) + (0.0494P)^2 + 1.614P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} = 0.006$
6805 reflections	$\Delta\rho_{max} = 0.32$ e Å^{-3}
493 parameters	$\Delta\rho_{min} = -0.35$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. After primary data collection, a portion of the first block of data was re-measured to check for crystal decay. No decay was detected.

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

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1A	0.20242 (12)	0.40432 (5)	1.03528 (7)	0.0256 (3)
O2A	0.03071 (11)	0.27053 (5)	0.82048 (7)	0.0229 (3)
O3A	0.02592 (11)	0.16851 (5)	0.81733 (7)	0.0232 (3)
O4A	0.38742 (10)	0.10197 (5)	1.20058 (7)	0.0195 (2)
O5A	0.37340 (11)	−0.00115 (5)	1.22967 (8)	0.0251 (3)
N1A	0.31059 (12)	0.32713 (6)	1.12891 (8)	0.0167 (3)
H1A	0.3680	0.3210	1.1791	0.020*
N2A	0.06020 (12)	0.21898 (6)	0.85658 (8)	0.0175 (3)
C1A	0.24095 (14)	0.15840 (7)	1.08144 (10)	0.0167 (3)
C2A	0.28688 (14)	0.21476 (7)	1.12436 (10)	0.0160 (3)
H2A	0.3376	0.2140	1.1834	0.019*
C3A	0.25812 (14)	0.27261 (7)	1.08026 (10)	0.0153 (3)
C4A	0.18188 (14)	0.27437 (7)	0.99157 (10)	0.0161 (3)
H4A	0.1606	0.3122	0.9611	0.019*
C5A	0.13906 (14)	0.21703 (7)	0.95063 (10)	0.0159 (3)
C6A	0.16598 (14)	0.15904 (7)	0.99234 (10)	0.0174 (3)
H6A	0.1353	0.1220	0.9622	0.021*
C7A	0.26692 (15)	0.09636 (7)	1.13020 (10)	0.0209 (3)
H7A1	0.1944	0.0860	1.1552	0.025*
H7A2	0.2752	0.0632	1.0892	0.025*
C8A	0.42784 (15)	0.04897 (7)	1.24824 (10)	0.0189 (3)
C9A	0.54698 (16)	0.06175 (8)	1.32327 (11)	0.0256 (4)
H9A1	0.5725	0.0241	1.3575	0.038*

H9A2	0.5273	0.0940	1.3609	0.038*
H9A3	0.6178	0.0753	1.2997	0.038*
C10A	0.28073 (15)	0.38884 (7)	1.10552 (10)	0.0174 (3)
C11A	0.35432 (16)	0.43580 (7)	1.17357 (10)	0.0201 (3)
H11A	0.4441	0.4384	1.1707	0.030*
H11B	0.3523	0.4225	1.2322	0.030*
H11C	0.3133	0.4763	1.1611	0.030*
O1B	0.57431 (11)	0.40304 (5)	1.05607 (7)	0.0213 (2)
O2B	0.77968 (11)	0.27015 (5)	1.26096 (7)	0.0213 (2)
O3B	0.78042 (11)	0.16832 (5)	1.26736 (7)	0.0223 (3)
O4B	0.40024 (11)	0.10339 (5)	0.88349 (7)	0.0215 (3)
O5B	0.37726 (11)	−0.00115 (5)	0.86389 (7)	0.0246 (3)
N1B	0.47811 (12)	0.32491 (6)	0.95923 (8)	0.0153 (3)
H1B	0.4269	0.3184	0.9068	0.018*
N2B	0.74344 (12)	0.21833 (6)	1.22784 (8)	0.0164 (3)
C1B	0.52801 (14)	0.15630 (7)	1.01409 (10)	0.0152 (3)
C2B	0.48614 (14)	0.21223 (7)	0.96936 (10)	0.0144 (3)
H2B	0.4298	0.2110	0.9120	0.017*
C3B	0.52750 (14)	0.27076 (7)	1.00929 (10)	0.0135 (3)
C4B	0.61325 (14)	0.27305 (7)	1.09487 (10)	0.0148 (3)
H4B	0.6427	0.3111	1.1225	0.018*
C5B	0.65302 (14)	0.21562 (7)	1.13730 (9)	0.0146 (3)
C6B	0.61306 (14)	0.15735 (7)	1.10019 (10)	0.0151 (3)
H6B	0.6417	0.1204	1.1313	0.018*
C7B	0.48392 (15)	0.09296 (7)	0.97218 (10)	0.0178 (3)
H7B1	0.5599	0.0681	0.9694	0.021*
H7B2	0.4360	0.0704	1.0077	0.021*
C8B	0.35221 (15)	0.05156 (7)	0.83557 (10)	0.0182 (3)
C9B	0.26650 (17)	0.06949 (8)	0.74595 (11)	0.0265 (4)
H9B1	0.2311	0.0323	0.7134	0.040*
H9B2	0.3177	0.0922	0.7136	0.040*
H9B3	0.1956	0.0955	0.7532	0.040*
C10B	0.50159 (14)	0.38687 (7)	0.98385 (10)	0.0155 (3)
C11B	0.43046 (16)	0.43349 (7)	0.91460 (10)	0.0212 (3)
H11D	0.4490	0.4753	0.9375	0.032*
H11E	0.3373	0.4259	0.9007	0.032*
H11F	0.4598	0.4289	0.8615	0.032*
O1C	0.71916 (12)	0.40226 (5)	0.85142 (7)	0.0252 (3)
O2C	0.48246 (11)	0.27074 (5)	0.65827 (7)	0.0223 (3)
O3C	0.48780 (11)	0.16908 (5)	0.64814 (7)	0.0251 (3)
O4C	0.86545 (11)	0.09864 (5)	1.02619 (7)	0.0200 (2)
O5C	0.87540 (15)	−0.00643 (6)	1.04009 (8)	0.0389 (3)
N1C	0.81090 (12)	0.32433 (6)	0.95011 (8)	0.0168 (3)
H1C	0.8617	0.3180	1.0027	0.020*
N2C	0.52430 (12)	0.21883 (6)	0.68831 (8)	0.0176 (3)
C1C	0.76316 (14)	0.15560 (7)	0.89269 (10)	0.0156 (3)
C2C	0.80760 (14)	0.21158 (7)	0.93740 (10)	0.0152 (3)
H2C	0.8694	0.2101	0.9927	0.018*
C3C	0.76084 (14)	0.27013 (7)	0.90059 (10)	0.0149 (3)

C4C	0.66657 (14)	0.27263 (7)	0.81786 (10)	0.0160 (3)
H4C	0.6339	0.3108	0.7920	0.019*
C5C	0.62360 (14)	0.21569 (7)	0.77576 (10)	0.0154 (3)
C6C	0.66830 (14)	0.15707 (7)	0.81015 (10)	0.0156 (3)
H6C	0.6365	0.1203	0.7796	0.019*
C7C	0.81812 (16)	0.09256 (7)	0.93031 (10)	0.0185 (3)
H7C1	0.7499	0.0607	0.9150	0.022*
H7C2	0.8897	0.0802	0.9055	0.022*
C8C	0.89136 (15)	0.04494 (7)	1.07379 (11)	0.0186 (3)
C9C	0.93755 (16)	0.05777 (8)	1.17183 (10)	0.0222 (3)
H9C1	0.8657	0.0521	1.1982	0.033*
H9C2	0.9692	0.1002	1.1813	0.033*
H9C3	1.0075	0.0293	1.1989	0.033*
C10C	0.78867 (15)	0.38631 (7)	0.92479 (10)	0.0175 (3)
C11C	0.85702 (16)	0.43276 (7)	0.99532 (11)	0.0216 (3)
H11G	0.8471	0.4743	0.9705	0.032*
H11H	0.9490	0.4225	1.0156	0.032*
H11I	0.8185	0.4310	1.0447	0.032*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.0318 (6)	0.0168 (6)	0.0220 (6)	0.0032 (5)	−0.0036 (5)	0.0006 (5)
O2A	0.0258 (6)	0.0205 (6)	0.0187 (6)	0.0017 (5)	−0.0006 (5)	0.0038 (5)
O3A	0.0266 (6)	0.0209 (6)	0.0196 (6)	−0.0049 (5)	0.0015 (5)	−0.0050 (5)
O4A	0.0205 (5)	0.0151 (5)	0.0190 (6)	−0.0015 (4)	−0.0015 (4)	0.0024 (4)
O5A	0.0314 (6)	0.0150 (6)	0.0262 (6)	−0.0032 (5)	0.0030 (5)	0.0013 (5)
N1A	0.0180 (6)	0.0154 (6)	0.0141 (6)	0.0010 (5)	−0.0002 (5)	−0.0011 (5)
N2A	0.0165 (6)	0.0194 (7)	0.0161 (6)	−0.0010 (5)	0.0036 (5)	0.0000 (5)
C1A	0.0145 (7)	0.0165 (8)	0.0192 (8)	0.0001 (6)	0.0046 (6)	0.0015 (6)
C2A	0.0154 (7)	0.0185 (8)	0.0129 (7)	0.0004 (6)	0.0017 (6)	0.0007 (6)
C3A	0.0138 (7)	0.0139 (7)	0.0182 (7)	−0.0001 (6)	0.0044 (6)	−0.0012 (6)
C4A	0.0159 (7)	0.0163 (8)	0.0159 (7)	0.0011 (6)	0.0037 (6)	0.0004 (6)
C5A	0.0145 (7)	0.0179 (8)	0.0144 (7)	0.0003 (6)	0.0023 (6)	0.0006 (6)
C6A	0.0163 (7)	0.0156 (8)	0.0194 (8)	−0.0015 (6)	0.0035 (6)	−0.0015 (6)
C7A	0.0210 (8)	0.0166 (8)	0.0213 (8)	−0.0037 (6)	−0.0011 (6)	0.0021 (6)
C8A	0.0223 (8)	0.0163 (8)	0.0189 (8)	0.0013 (6)	0.0071 (6)	0.0014 (6)
C9A	0.0287 (9)	0.0210 (8)	0.0231 (8)	0.0018 (7)	0.0002 (7)	0.0047 (7)
C10A	0.0191 (7)	0.0158 (7)	0.0179 (7)	0.0018 (6)	0.0059 (6)	0.0004 (6)
C11A	0.0239 (8)	0.0151 (8)	0.0203 (8)	0.0008 (6)	0.0044 (6)	0.0000 (6)
O1B	0.0267 (6)	0.0151 (5)	0.0176 (6)	−0.0004 (5)	−0.0020 (5)	−0.0014 (4)
O2B	0.0263 (6)	0.0157 (6)	0.0178 (6)	−0.0016 (5)	−0.0014 (5)	−0.0030 (4)
O3B	0.0276 (6)	0.0166 (6)	0.0177 (6)	0.0030 (5)	−0.0023 (5)	0.0036 (4)
O4B	0.0290 (6)	0.0126 (5)	0.0162 (5)	−0.0017 (5)	−0.0053 (5)	−0.0006 (4)
O5B	0.0326 (6)	0.0136 (6)	0.0225 (6)	−0.0016 (5)	−0.0014 (5)	0.0002 (5)
N1B	0.0178 (6)	0.0127 (6)	0.0122 (6)	0.0002 (5)	−0.0017 (5)	0.0004 (5)
N2B	0.0184 (6)	0.0150 (7)	0.0147 (6)	0.0010 (5)	0.0027 (5)	0.0002 (5)
C1B	0.0152 (7)	0.0145 (7)	0.0157 (7)	0.0003 (6)	0.0038 (6)	−0.0012 (6)

C2B	0.0133 (7)	0.0165 (7)	0.0125 (7)	−0.0004 (6)	0.0020 (6)	0.0000 (6)
C3B	0.0139 (7)	0.0126 (7)	0.0140 (7)	0.0008 (5)	0.0039 (6)	0.0007 (6)
C4B	0.0156 (7)	0.0128 (7)	0.0152 (7)	−0.0004 (6)	0.0029 (6)	−0.0015 (6)
C5B	0.0143 (7)	0.0170 (8)	0.0115 (7)	0.0002 (6)	0.0018 (6)	0.0002 (6)
C6B	0.0169 (7)	0.0128 (7)	0.0148 (7)	0.0010 (6)	0.0028 (6)	0.0021 (6)
C7B	0.0202 (8)	0.0144 (8)	0.0153 (7)	0.0005 (6)	−0.0012 (6)	0.0016 (6)
C8B	0.0192 (7)	0.0156 (8)	0.0191 (8)	−0.0024 (6)	0.0041 (6)	−0.0036 (6)
C9B	0.0337 (9)	0.0176 (8)	0.0215 (8)	−0.0020 (7)	−0.0041 (7)	−0.0013 (7)
C10B	0.0156 (7)	0.0150 (7)	0.0161 (7)	0.0004 (6)	0.0045 (6)	0.0007 (6)
C11B	0.0262 (8)	0.0148 (8)	0.0193 (8)	0.0005 (6)	0.0000 (7)	0.0012 (6)
O1C	0.0326 (6)	0.0166 (6)	0.0211 (6)	−0.0017 (5)	−0.0021 (5)	0.0021 (5)
O2C	0.0235 (6)	0.0194 (6)	0.0204 (6)	0.0034 (5)	−0.0006 (5)	0.0036 (5)
O3C	0.0288 (6)	0.0198 (6)	0.0213 (6)	−0.0012 (5)	−0.0029 (5)	−0.0052 (5)
O4C	0.0281 (6)	0.0138 (5)	0.0148 (5)	0.0006 (4)	−0.0003 (5)	0.0004 (4)
O5C	0.0691 (10)	0.0154 (6)	0.0245 (7)	0.0014 (6)	−0.0007 (7)	0.0011 (5)
N1C	0.0180 (6)	0.0152 (6)	0.0141 (6)	0.0000 (5)	−0.0010 (5)	−0.0007 (5)
N2C	0.0171 (6)	0.0192 (7)	0.0155 (6)	−0.0006 (5)	0.0028 (5)	−0.0012 (5)
C1C	0.0168 (7)	0.0153 (7)	0.0156 (7)	0.0008 (6)	0.0059 (6)	0.0008 (6)
C2C	0.0135 (7)	0.0177 (8)	0.0130 (7)	0.0005 (6)	0.0010 (6)	0.0007 (6)
C3C	0.0159 (7)	0.0145 (7)	0.0152 (7)	−0.0005 (6)	0.0057 (6)	−0.0006 (6)
C4C	0.0175 (7)	0.0150 (7)	0.0156 (7)	0.0002 (6)	0.0044 (6)	0.0010 (6)
C5C	0.0150 (7)	0.0173 (8)	0.0136 (7)	0.0004 (6)	0.0033 (6)	0.0001 (6)
C6C	0.0172 (7)	0.0150 (7)	0.0152 (7)	−0.0017 (6)	0.0052 (6)	−0.0018 (6)
C7C	0.0232 (8)	0.0152 (8)	0.0150 (7)	0.0007 (6)	0.0011 (6)	−0.0015 (6)
C8C	0.0207 (8)	0.0131 (8)	0.0209 (8)	0.0013 (6)	0.0037 (6)	0.0029 (6)
C9C	0.0245 (8)	0.0199 (8)	0.0204 (8)	0.0001 (7)	0.0029 (7)	0.0024 (6)
C10C	0.0167 (7)	0.0153 (8)	0.0200 (8)	−0.0012 (6)	0.0042 (6)	0.0009 (6)
C11C	0.0250 (8)	0.0136 (8)	0.0232 (8)	−0.0017 (6)	0.0013 (7)	0.0002 (6)

Geometric parameters (Å, °)

O1A—C10A	1.2243 (19)	C3B—C4B	1.394 (2)
O2A—N2A	1.2339 (17)	C4B—C5B	1.399 (2)
O3A—N2A	1.2405 (17)	C4B—H4B	0.9300
O4A—C8A	1.3546 (18)	C5B—C6B	1.385 (2)
O4A—C7A	1.4417 (18)	C6B—H6B	0.9300
O5A—C8A	1.2091 (19)	C7B—H7B1	0.9700
N1A—C10A	1.377 (2)	C7B—H7B2	0.9700
N1A—C3A	1.4145 (19)	C8B—C9B	1.493 (2)
N1A—H1A	0.8600	C9B—H9B1	0.9600
N2A—C5A	1.4761 (19)	C9B—H9B2	0.9600
C1A—C2A	1.395 (2)	C9B—H9B3	0.9600
C1A—C6A	1.399 (2)	C10B—C11B	1.508 (2)
C1A—C7A	1.511 (2)	C11B—H11D	0.9600
C2A—C3A	1.403 (2)	C11B—H11E	0.9600
C2A—H2A	0.9300	C11B—H11F	0.9600
C3A—C4A	1.398 (2)	O1C—C10C	1.2267 (18)
C4A—C5A	1.395 (2)	O2C—N2C	1.2344 (17)
C4A—H4A	0.9300	O3C—N2C	1.2372 (17)

C5A—C6A	1.388 (2)	O4C—C8C	1.3493 (18)
C6A—H6A	0.9300	O4C—C7C	1.4451 (17)
C7A—H7A1	0.9700	O5C—C8C	1.205 (2)
C7A—H7A2	0.9700	N1C—C10C	1.379 (2)
C8A—C9A	1.492 (2)	N1C—C3C	1.4088 (19)
C9A—H9A1	0.9600	N1C—H1C	0.8600
C9A—H9A2	0.9600	N2C—C5C	1.4781 (19)
C9A—H9A3	0.9600	C1C—C2C	1.396 (2)
C10A—C11A	1.510 (2)	C1C—C6C	1.400 (2)
C11A—H11A	0.9600	C1C—C7C	1.516 (2)
C11A—H11B	0.9600	C2C—C3C	1.404 (2)
C11A—H11C	0.9600	C2C—H2C	0.9300
O1B—C10B	1.2268 (18)	C3C—C4C	1.400 (2)
O2B—N2B	1.2339 (16)	C4C—C5C	1.394 (2)
O3B—N2B	1.2388 (16)	C4C—H4C	0.9300
O4B—C8B	1.3515 (18)	C5C—C6C	1.389 (2)
O4B—C7B	1.4409 (17)	C6C—H6C	0.9300
O5B—C8B	1.2085 (19)	C7C—H7C1	0.9700
N1B—C10B	1.3773 (19)	C7C—H7C2	0.9700
N1B—C3B	1.4101 (19)	C8C—C9C	1.495 (2)
N1B—H1B	0.8600	C9C—H9C1	0.9600
N2B—C5B	1.4743 (18)	C9C—H9C2	0.9600
C1B—C2B	1.390 (2)	C9C—H9C3	0.9600
C1B—C6B	1.397 (2)	C10C—C11C	1.509 (2)
C1B—C7B	1.515 (2)	C11C—H11G	0.9600
C2B—C3B	1.408 (2)	C11C—H11H	0.9600
C2B—H2B	0.9300	C11C—H11I	0.9600
C8A—O4A—C7A	115.47 (12)	O4B—C7B—C1B	108.23 (12)
C10A—N1A—C3A	127.79 (13)	O4B—C7B—H7B1	110.1
C10A—N1A—H1A	116.1	C1B—C7B—H7B1	110.1
C3A—N1A—H1A	116.1	O4B—C7B—H7B2	110.1
O2A—N2A—O3A	122.82 (13)	C1B—C7B—H7B2	110.1
O2A—N2A—C5A	118.81 (12)	H7B1—C7B—H7B2	108.4
O3A—N2A—C5A	118.37 (12)	O5B—C8B—O4B	122.98 (14)
C2A—C1A—C6A	119.79 (14)	O5B—C8B—C9B	126.58 (14)
C2A—C1A—C7A	121.27 (13)	O4B—C8B—C9B	110.44 (13)
C6A—C1A—C7A	118.92 (13)	C8B—C9B—H9B1	109.5
C1A—C2A—C3A	121.31 (14)	C8B—C9B—H9B2	109.5
C1A—C2A—H2A	119.3	H9B1—C9B—H9B2	109.5
C3A—C2A—H2A	119.3	C8B—C9B—H9B3	109.5
C4A—C3A—C2A	119.82 (14)	H9B1—C9B—H9B3	109.5
C4A—C3A—N1A	122.98 (13)	H9B2—C9B—H9B3	109.5
C2A—C3A—N1A	117.19 (13)	O1B—C10B—N1B	123.01 (14)
C5A—C4A—C3A	117.18 (14)	O1B—C10B—C11B	122.52 (14)
C5A—C4A—H4A	121.4	N1B—C10B—C11B	114.47 (13)
C3A—C4A—H4A	121.4	C10B—C11B—H11D	109.5
C6A—C5A—C4A	124.39 (14)	C10B—C11B—H11E	109.5
C6A—C5A—N2A	118.56 (13)	H11D—C11B—H11E	109.5
C4A—C5A—N2A	117.05 (13)	C10B—C11B—H11F	109.5

C5A—C6A—C1A	117.50 (14)	H11D—C11B—H11F	109.5
C5A—C6A—H6A	121.3	H11E—C11B—H11F	109.5
C1A—C6A—H6A	121.3	C8C—O4C—C7C	116.93 (12)
O4A—C7A—C1A	108.22 (12)	C10C—N1C—C3C	128.15 (13)
O4A—C7A—H7A1	110.1	C10C—N1C—H1C	115.9
C1A—C7A—H7A1	110.1	C3C—N1C—H1C	115.9
O4A—C7A—H7A2	110.1	O2C—N2C—O3C	122.98 (13)
C1A—C7A—H7A2	110.1	O2C—N2C—C5C	118.77 (12)
H7A1—C7A—H7A2	108.4	O3C—N2C—C5C	118.24 (12)
O5A—C8A—O4A	123.03 (14)	C2C—C1C—C6C	119.95 (14)
O5A—C8A—C9A	126.41 (15)	C2C—C1C—C7C	121.43 (13)
O4A—C8A—C9A	110.56 (13)	C6C—C1C—C7C	118.60 (13)
C8A—C9A—H9A1	109.5	C1C—C2C—C3C	121.37 (13)
C8A—C9A—H9A2	109.5	C1C—C2C—H2C	119.3
H9A1—C9A—H9A2	109.5	C3C—C2C—H2C	119.3
C8A—C9A—H9A3	109.5	C4C—C3C—C2C	119.56 (14)
H9A1—C9A—H9A3	109.5	C4C—C3C—N1C	122.72 (13)
H9A2—C9A—H9A3	109.5	C2C—C3C—N1C	117.72 (13)
O1A—C10A—N1A	122.96 (14)	C5C—C4C—C3C	117.36 (14)
O1A—C10A—C11A	122.92 (14)	C5C—C4C—H4C	121.3
N1A—C10A—C11A	114.11 (13)	C3C—C4C—H4C	121.3
C10A—C11A—H11A	109.5	C6C—C5C—C4C	124.50 (14)
C10A—C11A—H11B	109.5	C6C—C5C—N2C	118.54 (13)
H11A—C11A—H11B	109.5	C4C—C5C—N2C	116.96 (13)
C10A—C11A—H11C	109.5	C5C—C6C—C1C	117.26 (14)
H11A—C11A—H11C	109.5	C5C—C6C—H6C	121.4
H11B—C11A—H11C	109.5	C1C—C6C—H6C	121.4
C8B—O4B—C7B	116.39 (12)	O4C—C7C—C1C	108.01 (12)
C10B—N1B—C3B	128.13 (12)	O4C—C7C—H7C1	110.1
C10B—N1B—H1B	115.9	C1C—C7C—H7C1	110.1
C3B—N1B—H1B	115.9	O4C—C7C—H7C2	110.1
O2B—N2B—O3B	122.71 (12)	C1C—C7C—H7C2	110.1
O2B—N2B—C5B	118.81 (12)	H7C1—C7C—H7C2	108.4
O3B—N2B—C5B	118.48 (12)	O5C—C8C—O4C	123.12 (15)
C2B—C1B—C6B	120.10 (13)	O5C—C8C—C9C	125.33 (15)
C2B—C1B—C7B	121.85 (13)	O4C—C8C—C9C	111.52 (13)
C6B—C1B—C7B	118.05 (13)	C8C—C9C—H9C1	109.5
C1B—C2B—C3B	121.22 (13)	C8C—C9C—H9C2	109.5
C1B—C2B—H2B	119.4	H9C1—C9C—H9C2	109.5
C3B—C2B—H2B	119.4	C8C—C9C—H9C3	109.5
C4B—C3B—C2B	119.77 (13)	H9C1—C9C—H9C3	109.5
C4B—C3B—N1B	123.15 (13)	H9C2—C9C—H9C3	109.5
C2B—C3B—N1B	117.08 (12)	O1C—C10C—N1C	122.94 (14)
C3B—C4B—C5B	117.05 (13)	O1C—C10C—C11C	122.96 (14)
C3B—C4B—H4B	121.5	N1C—C10C—C11C	114.10 (13)
C5B—C4B—H4B	121.5	C10C—C11C—H11G	109.5
C6B—C5B—C4B	124.55 (13)	C10C—C11C—H11H	109.5
C6B—C5B—N2B	118.63 (13)	H11G—C11C—H11H	109.5
C4B—C5B—N2B	116.82 (13)	C10C—C11C—H11I	109.5

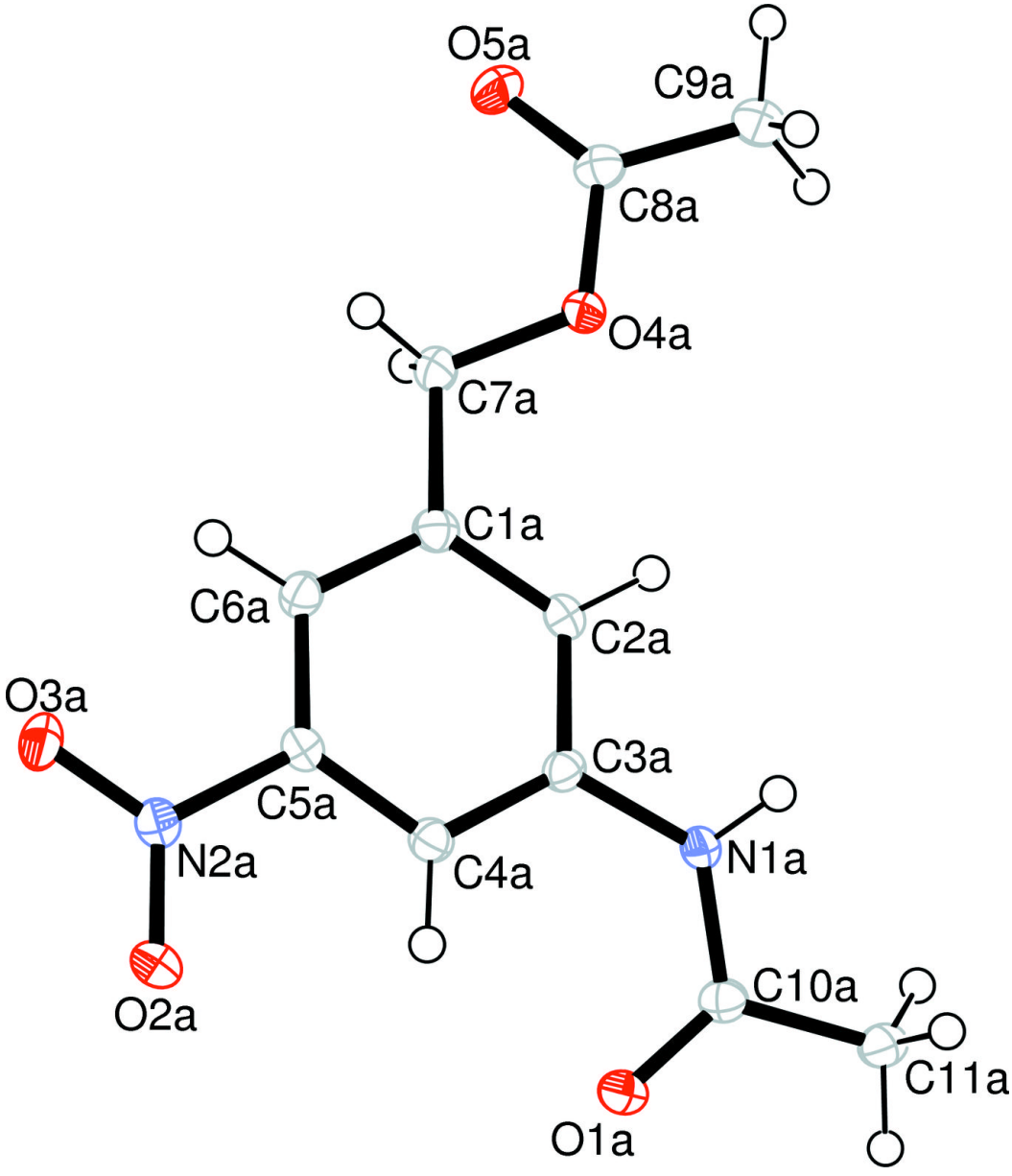
C5B—C6B—C1B	117.30 (13)	H11G—C11C—H11I	109.5
C5B—C6B—H6B	121.3	H11H—C11C—H11I	109.5
C1B—C6B—H6B	121.3		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1A—H1A···O3A^{i}	0.86	2.35	3.1897 (17)	166
N1B—H1B···O3B^{ii}	0.86	2.32	3.1506 (16)	164
N1C—H1C···O3C^{i}	0.86	2.31	3.1458 (17)	164
C2A—H2A···O2A^{i}	0.93	2.54	3.4345 (18)	161
C2B—H2B···O2B^{ii}	0.93	2.49	3.4046 (18)	169
C2C—H2C···O2C^{i}	0.93	2.56	3.4546 (18)	161

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

Fig. 1


O5a
C9a
C8a
O4a
C7a
C1a
C6a
C2a
O3a
C3a
C5a
N2a
C4a
N1a
O2a
C10a
C11a
O1a