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Methyl 2-(3,5-dinitrobenzamido)-3-methylbutanoate

Xiaokun Li[a]* and Yuqing Zhao[b]

[a]College of Pharmacy, Henan University of Traditional Chinese Medicine, Zhengzhou, 450008, People's Republic of China, and [b]School of Civil Engineering and Communication, North China University of Water Source and Electric Power, Zhengzhou 450011, People's Republic of China
Correspondence e-mail: li96052122@126.com

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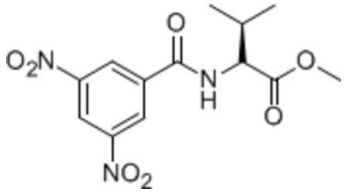
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.131; data-to-parameter ratio = 17.2.

In the title compound, $C_{13}H_{15}N_3O_7$, the dihedral angle between the amide plane (r.m.s. deviation = 0.008 Å) and the benzene ring is 33.2 (2)°. In the crystal, molecules are connected by N–H···O=C hydrogen bonds, forming a chain along the b-axis direction.

Related literature

For the biological activity of related compounds, see: Sykes *et al.* (1999).



Experimental

Crystal data

$C_{13}H_{15}N_3O_7$ M_r = 325.28
Orthorhombic, $P2_12_12_1$
a = 7.060 (2) Å
b = 9.412 (3) Å
c = 23.321 (6) Å
V = 1549.8 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 296 K
0.43 × 0.32 × 0.30 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.660, T_{max} = 0.746
9197 measured reflections
3580 independent reflections
3003 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.131
S = 1.03
3580 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N5—H5A···O5^{i}	0.86	2.12	2.949 (2)	161

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *publCIF* (Westrip, 2010).

The author thanks North China University of Water Source and Electric Power for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2434).

References

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Sykes, B. M., Atwell, G. J., Hogg, A., Wilson, W. R., O'Connor, C. J. & Denny, W. A. (1999). *J. Med. Chem.* **42**, 346–355.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2012). E**68**, o3098 [doi:10.1107/S1600536812040895]

Methyl 2-(3,5-dinitrobenzamido)-3-methylbutanoate

Xiaokun Li and Yuqing Zhao

S1. Comment

Nitro and ester groups widely exist in variety of biologically active compounds that could be used as prodrugs (Sykes *et al.*, 1999). We synthesized the title compound and determined its crystal structure (Fig. 1) whereas its biological activily is planned to be examined as well. In the crystal of the title compound, the carbonyl group acts as an acceptor and the amide group is a proton donor in the intermolecular N–H···O=C hydrogen bond, forming a chain along the *b* axis (Table 1, Fig. 2).

S2. Experimental

To a solution of methyl 2-amino-3-methylbutanoate hydrochloride (0.8 g, 5 mmol) and triethylamine (0.5 mL) in dry methylene chloride (100 mL) was added to 3,5-dinitrobenzoyl chloride (1.1 g, 5 mmol)in dry methylene chloride (50 mL) at 273 K. The mixture was allowed to warm to room temperature for 0.5 h. After concentrating, the residue was subjected to chromatography(petroleum ether/ acetone, 4:1) to provide the product as a white crystal (1.2 g, 71.1%).

S3. Refinement

The C-bound H-atoms were included in calculated positions and treated as riding atoms: C—H = 0.93, 0.96 and 0.98 Å for CH(aromatic), CH_3 and CH(methine) H-atoms, respectively,and N—H =0.86 Å, with U_{iso}(H)= k *r*imes U_{eq}(parent C-atom, N), where k = 1.5 for CH_3 H-atoms and k = 1.2 for all other H-atoms. The absolute configuration can be assigned to be R according to the known chirality of the precursor, only.

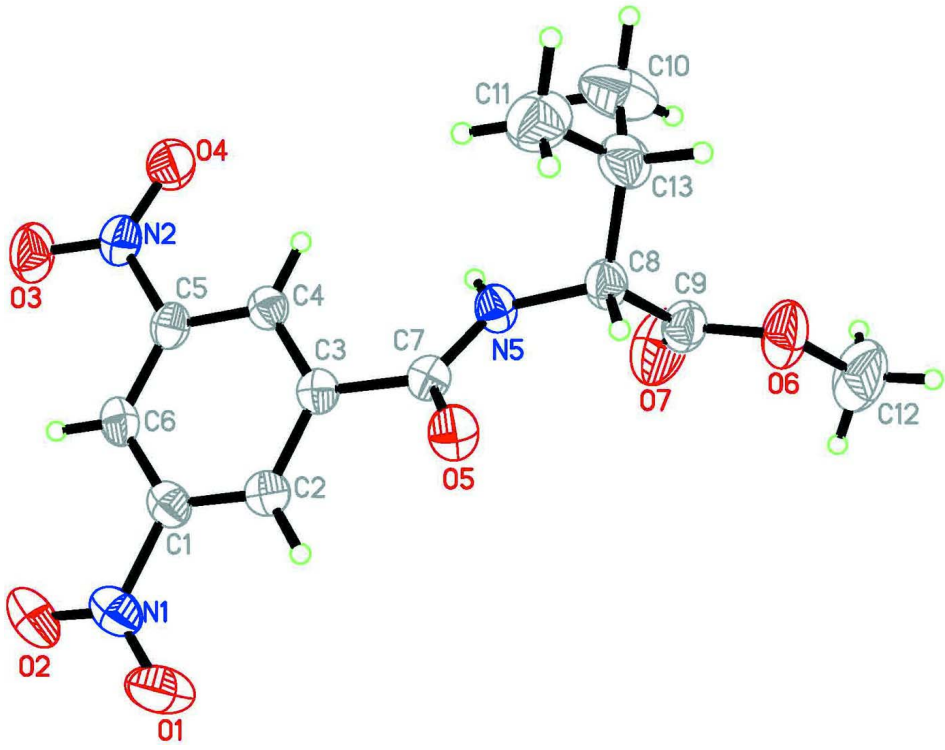

Figure 1

A view of the molecular strcuture of the title compound; the displacement ellipsoids drawn at the 30% probability level.

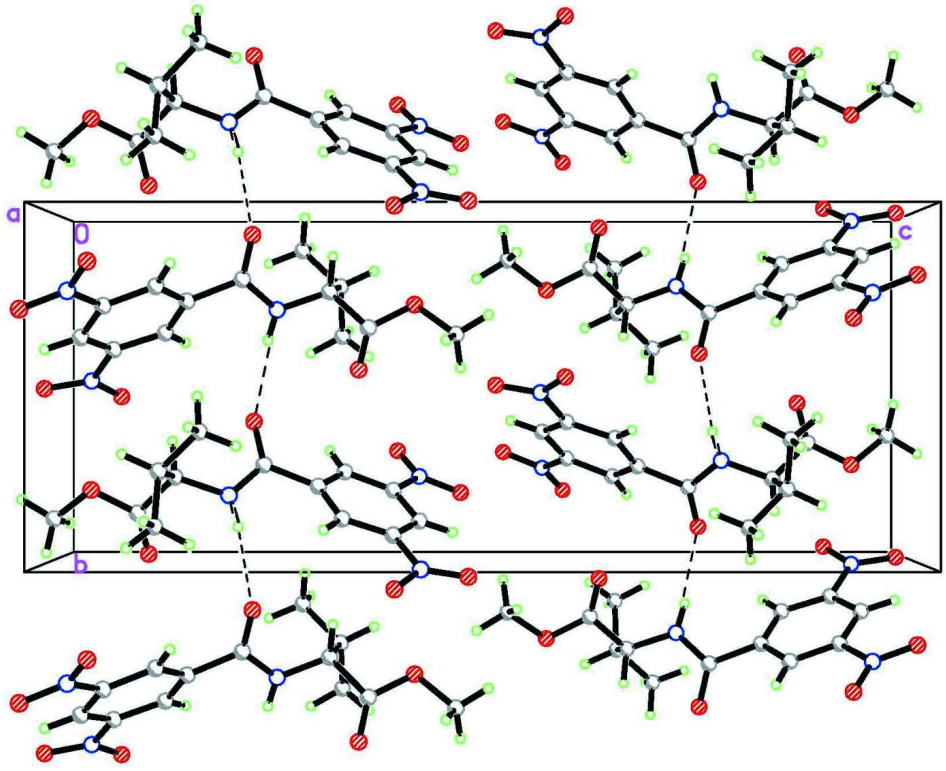

Figure 2

A view of the hydrogen bonded chain running in the [100] direction. The hydrogen bonds are shown as dashed lines.

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Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 7.060$ (2) Å
$b = 9.412$ (3) Å
$c = 23.321$ (6) Å
$V = 1549.8$ (7) Å^3
$Z = 4$
$F(000) = 680$
$D_x = 1.394$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9233 reflections
$\theta = 1.0$–$22.7°$
$\mu = 0.12$ mm^{-1}
$T = 296$ K
Block, colourless
0.43 × 0.32 × 0.30 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.660$, $T_{max} = 0.746$
9197 measured reflections
3580 independent reflections
3003 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 27.7°$, $\theta_{min} = 1.8°$
$h = -7 \rightarrow 9$
$k = -12 \rightarrow 12$
$l = -26 \rightarrow 30$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.131$
$S = 1.03$
3580 reflections
208 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0802P)^2 + 0.1123P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Special details

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 $F^2 > \sigma(F^2)$ 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. C10, C11, C13 and C12 belong to terminal alkyl chains which show signs of disorder and have higher thermal parameters.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	1.2857 (3)	0.28856 (19)	0.10037 (8)	0.0526 (4)
C2	1.2504 (3)	0.23672 (19)	0.15455 (8)	0.0503 (4)
H2A	1.3390	0.1801	0.1732	0.060*
C3	1.0795 (2)	0.27084 (17)	0.18071 (7)	0.0453 (4)
C4	0.9487 (3)	0.35376 (18)	0.15216 (8)	0.0469 (4)
H4A	0.8350	0.3785	0.1696	0.056*
C5	0.9895 (3)	0.39948 (18)	0.09717 (7)	0.0495 (4)
C6	1.1576 (3)	0.3692 (2)	0.07014 (8)	0.0521 (4)
H6A	1.1834	0.4015	0.0333	0.062*
C7	1.0425 (2)	0.21140 (17)	0.23943 (7)	0.0460 (4)
C8	0.8869 (3)	0.2398 (2)	0.33103 (8)	0.0557 (4)
H8A	0.9465	0.1466	0.3358	0.067*
C9	0.9790 (4)	0.3399 (3)	0.37526 (9)	0.0671 (6)
C10	0.5694 (5)	0.3567 (4)	0.3394 (2)	0.1160 (12)
H15A	0.4365	0.3390	0.3442	0.174*
H15B	0.5902	0.4049	0.3036	0.174*
H15C	0.6142	0.4149	0.3703	0.174*
C11	0.5992 (4)	0.1150 (4)	0.29434 (14)	0.0947 (9)
H14A	0.4656	0.1019	0.2998	0.142*
H14B	0.6627	0.0253	0.2983	0.142*
H14C	0.6221	0.1526	0.2567	0.142*
C12	1.0720 (6)	0.3594 (4)	0.47134 (13)	0.1166 (13)
H12A	1.0668	0.3069	0.5066	0.175*
H12B	1.0072	0.4483	0.4760	0.175*

H12C	1.2018	0.3769	0.4613	0.175*
C13	0.6744 (4)	0.2186 (3)	0.33924 (11)	0.0735 (6)
H13A	0.6560	0.1746	0.3769	0.088*
N1	1.4675 (3)	0.2555 (2)	0.07277 (9)	0.0671 (5)
N2	0.8475 (3)	0.48375 (18)	0.06597 (7)	0.0613 (4)
N5	0.9379 (2)	0.29050 (15)	0.27435 (6)	0.0506 (4)
H5A	0.9000	0.3728	0.2632	0.061*
O1	1.5804 (3)	0.1847 (3)	0.09906 (10)	0.0990 (6)
O2	1.4962 (3)	0.3026 (2)	0.02520 (8)	0.0877 (6)
O3	0.8739 (3)	0.5050 (2)	0.01510 (7)	0.0856 (5)
O4	0.7125 (3)	0.5274 (2)	0.09269 (7)	0.0781 (5)
O5	1.1072 (2)	0.09485 (13)	0.25244 (6)	0.0596 (4)
O6	0.9809 (3)	0.2774 (2)	0.42582 (6)	0.0897 (6)
O7	1.0395 (5)	0.4534 (3)	0.36573 (10)	0.1163 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0540 (10)	0.0489 (9)	0.0547 (10)	−0.0103 (8)	0.0075 (8)	−0.0126 (8)
C2	0.0526 (9)	0.0426 (8)	0.0556 (10)	−0.0011 (7)	−0.0013 (8)	−0.0040 (7)
C3	0.0525 (9)	0.0375 (7)	0.0460 (8)	−0.0054 (7)	−0.0006 (7)	−0.0030 (7)
C4	0.0503 (9)	0.0439 (8)	0.0464 (8)	−0.0035 (7)	−0.0001 (7)	−0.0047 (7)
C5	0.0614 (10)	0.0427 (8)	0.0445 (8)	−0.0027 (8)	−0.0047 (8)	−0.0028 (7)
C6	0.0639 (11)	0.0497 (9)	0.0426 (8)	−0.0102 (8)	0.0046 (8)	−0.0043 (7)
C7	0.0500 (9)	0.0402 (8)	0.0477 (8)	−0.0039 (7)	−0.0024 (7)	0.0004 (7)
C8	0.0658 (11)	0.0549 (10)	0.0463 (9)	0.0091 (9)	0.0023 (8)	0.0045 (8)
C9	0.0770 (14)	0.0720 (13)	0.0523 (11)	0.0169 (12)	−0.0047 (10)	−0.0061 (9)
C10	0.0821 (19)	0.094 (2)	0.172 (4)	0.0177 (17)	0.034 (2)	0.017 (2)
C11	0.0834 (18)	0.099 (2)	0.102 (2)	−0.0206 (17)	−0.0107 (15)	0.0137 (17)
C12	0.152 (3)	0.130 (3)	0.0680 (16)	0.019 (3)	−0.0295 (19)	−0.0348 (17)
C13	0.0736 (14)	0.0762 (14)	0.0707 (13)	−0.0033 (12)	0.0113 (11)	0.0167 (11)
N1	0.0613 (10)	0.0632 (10)	0.0769 (12)	−0.0070 (9)	0.0174 (9)	−0.0107 (9)
N2	0.0743 (11)	0.0594 (9)	0.0504 (9)	0.0028 (9)	−0.0069 (8)	0.0018 (7)
N5	0.0629 (9)	0.0432 (7)	0.0456 (7)	0.0071 (7)	0.0047 (6)	0.0055 (6)
O1	0.0641 (10)	0.1110 (14)	0.1218 (16)	0.0183 (11)	0.0206 (10)	0.0107 (13)
O2	0.0948 (13)	0.0910 (12)	0.0773 (11)	−0.0046 (11)	0.0372 (10)	−0.0117 (9)
O3	0.1103 (15)	0.0954 (12)	0.0511 (8)	0.0146 (12)	−0.0049 (9)	0.0146 (8)
O4	0.0800 (11)	0.0881 (11)	0.0661 (9)	0.0258 (9)	−0.0040 (8)	0.0041 (8)
O5	0.0776 (9)	0.0431 (6)	0.0580 (7)	0.0098 (6)	0.0016 (7)	0.0035 (6)
O6	0.1274 (15)	0.0904 (11)	0.0512 (8)	0.0167 (12)	−0.0109 (9)	−0.0112 (8)
O7	0.162 (2)	0.0923 (13)	0.0946 (14)	−0.0369 (15)	−0.0343 (14)	−0.0015 (12)

Geometric parameters (Å, º)

C1—C6	1.375 (3)	C9—O6	1.318 (3)
C1—C2	1.377 (3)	C10—C13	1.497 (4)
C1—N1	1.469 (3)	C10—H15A	0.9600
C2—C3	1.390 (3)	C10—H15B	0.9600

C2—H2A	0.9300	C10—H15C	0.9600
C3—C4	1.380 (3)	C11—C13	1.526 (4)
C3—C7	1.502 (2)	C11—H14A	0.9600
C4—C5	1.383 (3)	C11—H14B	0.9600
C4—H4A	0.9300	C11—H14C	0.9600
C5—C6	1.373 (3)	C12—O6	1.462 (3)
C5—N2	1.471 (3)	C12—H12A	0.9600
C6—H6A	0.9300	C12—H12B	0.9600
C7—O5	1.226 (2)	C12—H12C	0.9600
C7—N5	1.328 (2)	C13—H13A	0.9800
C8—N5	1.451 (2)	N1—O1	1.206 (3)
C8—C13	1.525 (3)	N1—O2	1.212 (3)
C8—C9	1.541 (3)	N2—O4	1.211 (2)
C8—H8A	0.9800	N2—O3	1.217 (2)
C9—O7	1.171 (3)	N5—H5A	0.8600

C6—C1—C2	123.15 (18)	H15A—C10—H15B	109.5
C6—C1—N1	117.83 (18)	C13—C10—H15C	109.5
C2—C1—N1	119.02 (19)	H15A—C10—H15C	109.5
C1—C2—C3	118.55 (18)	H15B—C10—H15C	109.5
C1—C2—H2A	120.7	C13—C11—H14A	109.5
C3—C2—H2A	120.7	C13—C11—H14B	109.5
C4—C3—C2	119.99 (17)	H14A—C11—H14B	109.5
C4—C3—C7	122.29 (16)	C13—C11—H14C	109.5
C2—C3—C7	117.70 (16)	H14A—C11—H14C	109.5
C3—C4—C5	118.94 (17)	H14B—C11—H14C	109.5
C3—C4—H4A	120.5	O6—C12—H12A	109.5
C5—C4—H4A	120.5	O6—C12—H12B	109.5
C6—C5—C4	122.75 (17)	H12A—C12—H12B	109.5
C6—C5—N2	118.28 (16)	O6—C12—H12C	109.5
C4—C5—N2	118.97 (17)	H12A—C12—H12C	109.5
C5—C6—C1	116.59 (17)	H12B—C12—H12C	109.5
C5—C6—H6A	121.7	C10—C13—C8	111.9 (2)
C1—C6—H6A	121.7	C10—C13—C11	112.6 (3)
O5—C7—N5	123.84 (16)	C8—C13—C11	109.9 (2)
O5—C7—C3	119.61 (16)	C10—C13—H13A	107.4
N5—C7—C3	116.54 (14)	C8—C13—H13A	107.4
N5—C8—C13	113.70 (18)	C11—C13—H13A	107.4
N5—C8—C9	107.70 (17)	O1—N1—O2	123.8 (2)
C13—C8—C9	114.29 (18)	O1—N1—C1	118.12 (19)
N5—C8—H8A	106.9	O2—N1—C1	118.0 (2)
C13—C8—H8A	106.9	O4—N2—O3	124.5 (2)
C9—C8—H8A	106.9	O4—N2—C5	117.71 (17)
O7—C9—O6	125.0 (2)	O3—N2—C5	117.81 (19)
O7—C9—C8	125.7 (2)	C7—N5—C8	120.85 (15)
O6—C9—C8	109.3 (2)	C7—N5—H5A	119.6
C13—C10—H15A	109.5	C8—N5—H5A	119.6
C13—C10—H15B	109.5	C9—O6—C12	114.7 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5*A*···O5^{i}	0.86	2.12	2.949 (2)	161

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