Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-(2-Methyl-3,6-dinitrophenyl)acetamide

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Received 17 October 2005
Accepted 24 October 2005
Online 27 October 2005

Key indicators

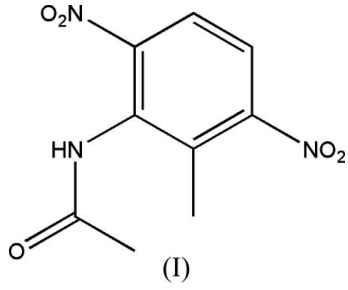
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.024
wR factor = 0.062
Data-to-parameter ratio = 7.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The structure of the title compound, $C_9H_9N_3O_5$, was determined as one of a group of five related compounds in order to assess its suitability as a test material for the 2004 Cambridge Crystallographic Data Centre 'Blind Structure Prediction Test'. The structure consists of hydrogen-bonded ribbons of molecules stacked along the *a* axis with the benzene rings parallel by unit-cell translations.

Comment

The structure of the title material, (I), was determined as part of the preparations for the 2004 Cambridge Crystallographic Data Centre 'Blind Structure Prediction Tests' (Watkin *et al.*, 2004), though (I) was not used in the test. The material was from a collection of nitrotoluene derivatives synthesized by Wilhelm Koerner about a century ago and retrieved from a depository at the University of Milan.



(I)

The sample consisted of large, striated, pale-cream laths. Attempts were made to obtain a roughly isometric sample, but the specimens inevitably splintered freely if any attempt was

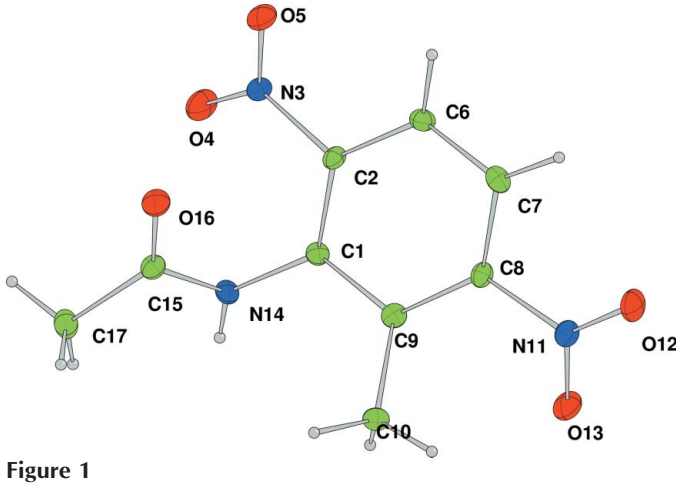


Figure 1
The title compound, with atomic displacement parameters drawn at the 50% probability level and the H atoms with arbitary radii.

made to cut them into shorter lengths. One was selected on the basis of its sharp diffraction pattern and relative thickness. Changes in illuminated volume were kept to a minimum by the data collection strategy, and were taken into account (Görbitz, 1999) by the multi-scan inter-frame scaling (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997).

The two nitro groups are twisted by almost the same angle from the plane of the benzene ring [C1—C2—N3—O5 = 143.3 (3)° and C9—C8—C11—O12 = −147.9 (3)°]. The almost planar acetamide group is rotated out of the ring plane [C9—C1—N14—C15 = 129.3 (3)°] (Fig. 1).

The structure consists of ribbons of molecules stacked with the benzene rings parallel by unit-cell translations along the *a* axis, giving an interplanar separation of 3.618 (3) Å (Fig. 2). Molecules in these ribbons are linked together by hydrogen bonds (Fig. 3 and Table 1). Other intermolecular contacts are unexceptional.

Experimental

The material was from a collection of nitrotoluene derivatives synthesized by Wilhelm Koerner about a century ago and retrieved from a depository at the University of Milan (Demartin *et al.*, 2004). Details of the preparation and crystallization are unknown.

Crystal data

$C_9H_9N_3O_5$
M_r = 239.19
Monoclinic, $P2_1$
a = 4.9309 (2) Å
b = 11.7571 (4) Å
c = 8.7944 (3) Å
β = 99.8608 (14)°
V = 502.31 (3) Å^3
Z = 2
D_x = 1.581 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 939 reflections
θ = 5–27°
μ = 0.13 mm^{-1}
T = 120 K
Lath, pale yellow
0.76 × 0.20 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.80, T_{max} = 0.99
3452 measured reflections
1188 independent reflections
1188 reflections with $I > -10\sigma(I)$
R_{int} = 0.021
θ_{max} = 27.5°
$h = -6 \rightarrow 6$
$k = -15 \rightarrow 12$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.062
S = 1.09
1188 reflections
154 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F^2) + (0.02P)^2 + 0.13P]$ where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 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
N14—H7···O16^{i}	0.84	2.13	2.963 (2)	168

Symmetry code: (i) $x-1, y, z$.

In the absence of significant anomalous scattering, Friedel pairs were merged. The H atoms were all located in a difference map, but those attached to C atoms were repositioned geometrically. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H in the target range 0.93–98 Å and N—H 0.86 Å) and isotropic displacement parameters [U_{iso}(H) in the range 1.2–1.5 times U_{eq} of the parent atom], after which they were refined with riding constraints.

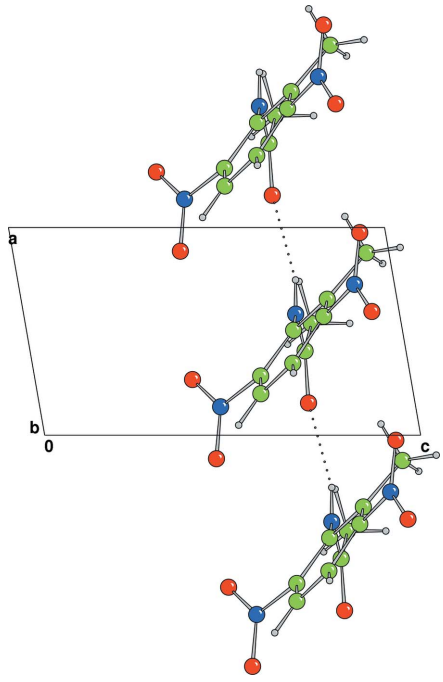


Figure 2
Projection along the *b* axis, showing the hydrogen bonding (dotted lines) and the aromatic ring stacking.

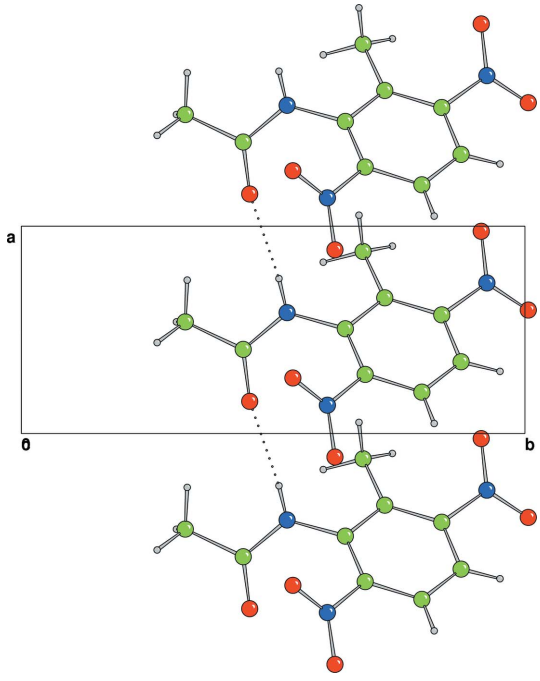


Figure 3
Projection along the *a* axis, showing the hydrogen-bonded (dotted lines) chain, with the benzene rings all on the same side of the *c* axis.

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al*., 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al*., 2003); molecular graphics: *CAMERON* (Watkin *et al*., 1996); software used to prepare material for publication: *CRYSTALS*.

We thank Professor Angelo Gavezzotti for obtaining the samples, Professor Lucio Merlini, Director of the Dipartmento di Scienze Molecolari Agroalimentari of the University of Milan, for generously donating them, and Professor Anna Arnoldi for help in their retrieval.

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supporting information

Acta Cryst. (2005). E**61**, o3888–o3890 [https://doi.org/10.1107/S1600536805034355]

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Cell parameters from 939 reflections
$\theta = 5$–27°
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1188 independent reflections
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$\theta_{max} = 27.5°$, $\theta_{min} = 5.2°$
$h = -6 \rightarrow 6$
$k = -15 \rightarrow 12$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.062$
$S = 1.09$
1188 reflections
154 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F^2) + (0.02P)^2 + 0.13P]$ where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max} = 0.000205$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.4948 (3)	0.14319 (14)	0.28928 (18)	0.0111
C2	0.7148 (3)	0.18283 (14)	0.39815 (18)	0.0114
N3	0.8638 (3)	0.10778 (13)	0.51881 (15)	0.0140
O4	0.7326 (3)	0.03921 (12)	0.58113 (14)	0.0219

O5	1.1140 (2)	0.12260 (12)	0.55405 (15)	0.0219
C6	0.8003 (3)	0.29526 (15)	0.40571 (18)	0.0134
C7	0.6540 (3)	0.37335 (15)	0.30692 (19)	0.0139
C8	0.4280 (3)	0.33503 (15)	0.20287 (18)	0.0116
C9	0.3458 (3)	0.22171 (15)	0.18546 (18)	0.0118
C10	0.1222 (3)	0.17863 (15)	0.06026 (19)	0.0159
N11	0.2738 (3)	0.42462 (13)	0.10628 (16)	0.0138
O12	0.4046 (3)	0.50665 (11)	0.07238 (15)	0.0211
O13	0.0249 (2)	0.41328 (11)	0.06852 (15)	0.0196
N14	0.4172 (3)	0.02758 (12)	0.27649 (17)	0.0130
C15	0.5946 (3)	−0.05962 (14)	0.26833 (18)	0.0129
O16	0.8450 (2)	−0.04647 (11)	0.28353 (14)	0.0166
C17	0.4615 (3)	−0.17467 (14)	0.2390 (2)	0.0169
H61	0.9522	0.3189	0.4777	0.0171*
H71	0.7032	0.4511	0.3102	0.0169*
H73	0.2672	−0.1711	0.2518	0.0263*
H72	0.4683	−0.1941	0.1353	0.0281*
H4	0.5585	−0.2301	0.3078	0.0284*
H7	0.2481	0.0118	0.2655	0.0178*
H1	0.1742	0.1034	0.0229	0.0241*
H2	−0.0511	0.1712	0.1004	0.0240*
H3	0.0949	0.2334	−0.0280	0.0238*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0084 (6)	0.0117 (8)	0.0136 (7)	−0.0006 (6)	0.0031 (5)	−0.0001 (6)
C2	0.0089 (7)	0.0124 (8)	0.0129 (7)	0.0016 (6)	0.0016 (5)	0.0019 (6)
N3	0.0133 (6)	0.0144 (7)	0.0136 (6)	0.0012 (6)	−0.0001 (5)	0.0007 (6)
O4	0.0207 (6)	0.0245 (7)	0.0203 (6)	−0.0025 (6)	0.0027 (5)	0.0099 (6)
O5	0.0124 (5)	0.0206 (7)	0.0300 (7)	0.0002 (5)	−0.0045 (5)	0.0049 (6)
C6	0.0111 (7)	0.0141 (8)	0.0144 (7)	−0.0009 (6)	0.0004 (5)	−0.0018 (7)
C7	0.0141 (7)	0.0122 (7)	0.0159 (8)	−0.0014 (6)	0.0040 (6)	−0.0024 (6)
C8	0.0114 (7)	0.0114 (7)	0.0127 (7)	0.0016 (6)	0.0035 (6)	0.0016 (6)
C9	0.0093 (6)	0.0142 (8)	0.0127 (7)	0.0003 (6)	0.0040 (6)	0.0002 (6)
C10	0.0134 (7)	0.0150 (8)	0.0178 (8)	−0.0005 (6)	−0.0014 (6)	−0.0006 (7)
N11	0.0140 (6)	0.0135 (7)	0.0141 (6)	0.0017 (6)	0.0032 (5)	0.0011 (6)
O12	0.0209 (6)	0.0149 (6)	0.0270 (7)	−0.0034 (5)	0.0026 (5)	0.0083 (5)
O13	0.0124 (5)	0.0192 (7)	0.0262 (6)	0.0027 (5)	0.0009 (5)	0.0042 (6)
N14	0.0080 (5)	0.0115 (7)	0.0194 (7)	−0.0012 (5)	0.0020 (5)	−0.0001 (6)
C15	0.0132 (7)	0.0138 (8)	0.0120 (7)	0.0008 (6)	0.0024 (5)	0.0014 (6)
O16	0.0099 (5)	0.0176 (6)	0.0225 (6)	0.0010 (5)	0.0027 (4)	0.0009 (5)
C17	0.0156 (7)	0.0121 (8)	0.0230 (8)	−0.0002 (7)	0.0032 (6)	0.0003 (7)

Geometric parameters (Å, º)

C1—C2	1.398 (2)	C9—C10	1.506 (2)
C1—C9	1.413 (2)	C10—H1	0.992

C1—N14	1.411 (2)	C10—H2	0.982
C2—N3	1.476 (2)	C10—H3	1.000
C2—C6	1.386 (2)	N11—O12	1.2250 (19)
N3—O4	1.221 (2)	N11—O13	1.2228 (17)
N3—O5	1.2318 (17)	N14—C15	1.358 (2)
C6—C7	1.380 (2)	N14—H7	0.843
C6—H61	0.936	C15—O16	1.2287 (18)
C7—C8	1.390 (2)	C15—C17	1.506 (2)
C7—H71	0.945	C17—H73	0.984
C8—C9	1.393 (2)	C17—H72	0.947
C8—N11	1.479 (2)	C17—H4	0.959
C2—C1—C9	118.77 (15)	C9—C10—H1	110.1
C2—C1—N14	122.93 (15)	C9—C10—H2	109.9
C9—C1—N14	118.29 (14)	H1—C10—H2	109.2
C1—C2—N3	121.71 (15)	C9—C10—H3	109.6
C1—C2—C6	123.02 (15)	H1—C10—H3	109.0
N3—C2—C6	115.20 (14)	H2—C10—H3	109.1
C2—N3—O4	118.72 (13)	C8—N11—O12	117.40 (13)
C2—N3—O5	116.77 (14)	C8—N11—O13	118.15 (14)
O4—N3—O5	124.44 (14)	O12—N11—O13	124.44 (15)
C2—C6—C7	118.84 (15)	C1—N14—C15	124.26 (14)
C2—C6—H61	121.1	C1—N14—H7	118.0
C7—C6—H61	120.0	C15—N14—H7	117.5
C6—C7—C8	118.21 (16)	N14—C15—O16	122.96 (16)
C6—C7—H71	121.4	N14—C15—C17	114.91 (13)
C8—C7—H71	120.4	O16—C15—C17	122.14 (15)
C7—C8—C9	124.57 (15)	C15—C17—H73	110.3
C7—C8—N11	114.99 (15)	C15—C17—H72	107.2
C9—C8—N11	120.43 (13)	H73—C17—H72	108.5
C1—C9—C8	116.39 (14)	C15—C17—H4	110.2
C1—C9—C10	119.16 (15)	H73—C17—H4	110.3
C8—C9—C10	124.37 (15)	H72—C17—H4	110.4

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N14—H7···O16^{i}	0.84	2.13	2.963 (2)	168

Symmetry code: (i) *x*−1, *y*, *z*.