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2-Acetamido-4-nitrotoluene

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.047
wR factor = 0.098
Data-to-parameter ratio = 16.6

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, $C_9H_{10}N_2O_3$, 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 molecules are almost planar except for the acetamide group, which is involved in hydrogen bonding. The structure consists of columns of molecules hudrogen bonded into chains parallel to the c axis.

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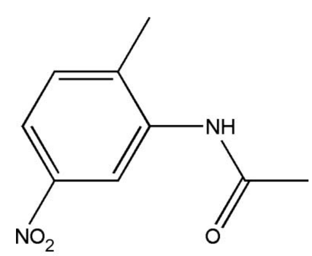
Comment

The Cambridge Crystallographic Data Centre (CCDC) 'Blind Structure Prediction Tests' are carried out periodically by a number of participating groups in order to evaluate developments in structure prediction techniques. As part of the preparations for the 2004 test, five well crystalline samples whose crystal structure was previously unknown were provided by Gavezzotti. The materials were from a collection of nitrotoluene derivatives synthesized by Wilhelm Koerner about a century ago and retrieved from a depository at the University of Milan. The structures and analyses of several other materials from this collection have recently been discussed (Demartin *et al.*, 2004).



(I)

The sample consisted of a mixture of crushed and broken fragments and some striated pale-cream lath-shaped crystals. These were always long and generally very thin. Attempts were made to obtain a roughly isometric sample, but the specimens inevitably cleaved freely parallel to their long axis if any attempt was made to cut them into shorter segments. A crystal 0.12 × 0.63 × 1.22 mm (Fig. 1) was selected on the basis of its sharp diffraction pattern. By mounting the crystal approximately parallel to the φ axis, the changes in illuminated volume were kept to a minimum, and were taken into account (Görbitz, 1999) by the multi-scan interframe scaling (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997).

The nitro group is almost complanar with the benzene ring [O8—N7—C5—C6 = −177.3 (3)°]. The acetamide group is itself planar [C14—C12—N11—C1 = 178.3 (3)°], but is rotated

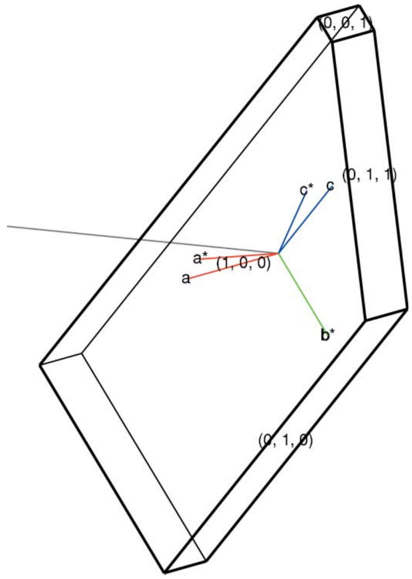


Figure 1
Perspective view of the crystal used for data collection showing the indices of the principal faces and their relationship to the diffractometer axes.

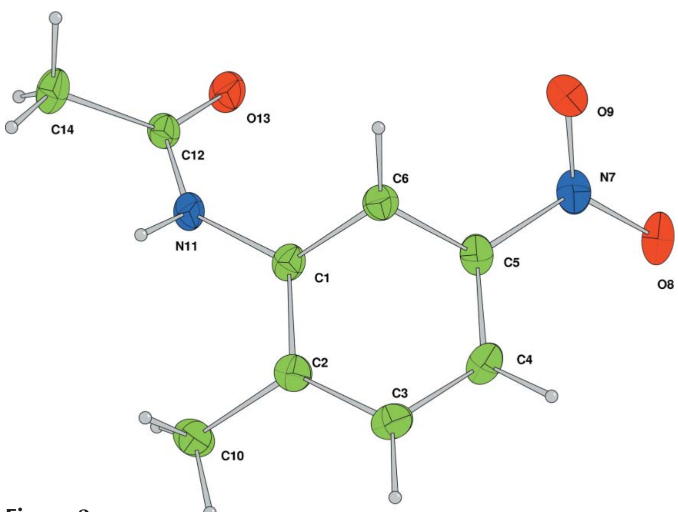


Figure 2
The molecular structure with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitary radius.

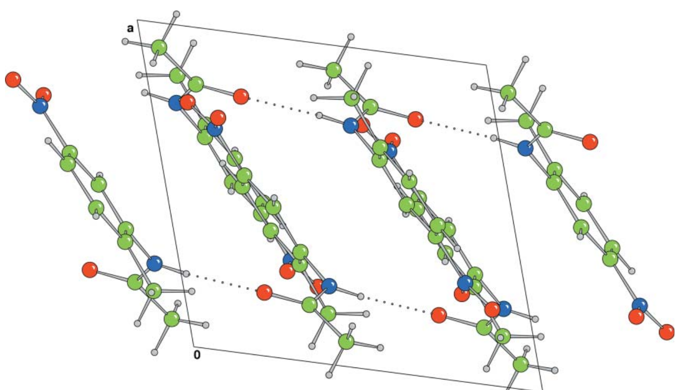


Figure 3
Packing diagram, showing the hydrogen-bonded chains parallel to the *c* axis. Hydrogen bonds are indicated as dotted lines.

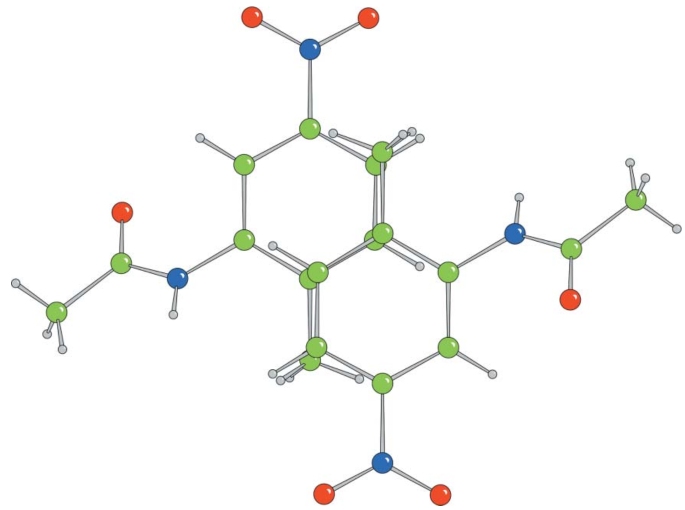


Figure 4
Projection of molecules from two adjacent chains on to the plane of one benzene ring.

out of the plane of the benzene ring [C12—N11—C1—C2 = −133.3 (3)°] (Fig. 2).

Hydrogen bonding between atom H5 of one molecule and O13 of an adjacent molecule causes the structure to consist of chains parallel to the *c* axis (Fig. 3). The benzene rings in adjacent chains lie parallel to each other, with a perpendicular separation of 3.58 Å, but do not overlap in projection (Fig. 4). Other intermolecular contacts are unexceptional.

Experimental

Details of the synthesis are unknown; the 100-year-old sample was provided from the depository at the University of Milan.

Crystal data

$C_9H_{10}N_2O_3$
M_r = 194.19
Monoclinic, $P2_1/c$
a = 8.2167 (2) Å
b = 13.6406 (3) Å
c = 8.7203 (2) Å
β = 107.2307 (9)°
V = 933.51 (4) Å^3
Z = 4
D_x = 1.382 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2141 reflections
θ = 5–27°
μ = 0.11 mm^{-1}
T = 150 K
Lath, pale yellow
1.22 × 0.63 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.77, T_{max} = 0.99
10098 measured reflections
2110 independent reflections
2110 reflections with $I > -10.0\sigma(I)$
R_{int} = 0.027
θ_{max} = 27.5°
$h = -10 \rightarrow 10$
$k = -17 \rightarrow 17$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.098
S = 1.00
2110 reflections
127 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F^2) + (0.04P)^2 + 0.32P]$
where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

$D-\mathrm{H}\cdots A$	$D-\mathrm{H}$	$\mathrm{H}\cdots A$	$D\cdots A$	$D-\mathrm{H}\cdots A$
$\mathrm{N11-H5\cdots O13^{i}}$	0.86	2.06	2.911 (1)	175

Symmetry code: (i) $x, -y+\frac{3}{2}, z-\frac{1}{2}$.

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 = 0.93–0.98 Å, N—H = 0.86–0.89 Å and O—H = 0.82 Å) and displacement parameters [$U_{\rm iso}$(H) = 1.2–1.5$U_{\rm eq}$(parent atom)], after which they were refined with riding constraints.

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO*/*SCALEPACK*; data reduction: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); 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*.

The authors thank Professor Angelo Gavezzotti for obtaining the samples, Professor Lucio Merlini, Director of the Dipartmento di Scienze Molecolari Agroalimentari of the University of Milano, for generously donationg the samples, and Professor Anna Arnoldi for help in the retrieval of the crystals.

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$\mu = 0.11$ mm^{-1}
$T = 150$ K
Lath, pale yellow
1.22 × 0.63 × 0.12 mm

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Nonius KappaCCD diffractometer
Graphite monochromator
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$R_{int} = 0.027$
$\theta_{max} = 27.5°$, $\theta_{min} = 5.1°$
$h = -10 \rightarrow 10$
$k = -17 \rightarrow 17$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.098$
$S = 1.00$
2110 reflections
127 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F^2) + (0.04P)^2 + 0.32P]$ where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max} = 0.000416$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.66060 (14)	0.65335 (8)	0.13949 (12)	0.0205
C2	0.69944 (14)	0.55296 (8)	0.14755 (13)	0.0222
C3	0.60557 (15)	0.49005 (9)	0.21537 (14)	0.0266
C4	0.47870 (15)	0.52447 (9)	0.27626 (14)	0.0272

C5	0.44408 (15)	0.62402 (9)	0.26521 (14)	0.0241
C6	0.53092 (14)	0.68921 (8)	0.19623 (13)	0.0225
N7	0.30928 (13)	0.66177 (8)	0.32777 (13)	0.0311
O8	0.23776 (16)	0.60433 (8)	0.39389 (17)	0.0584
O9	0.27408 (14)	0.74913 (7)	0.31277 (15)	0.0501
C10	0.84177 (16)	0.51420 (9)	0.08932 (15)	0.0289
N11	0.75561 (12)	0.71876 (7)	0.07257 (11)	0.0228
C12	0.82179 (14)	0.80492 (8)	0.13955 (13)	0.0204
O13	0.80172 (11)	0.83621 (6)	0.26552 (10)	0.0276
C14	0.92198 (16)	0.86113 (9)	0.05038 (14)	0.0267
H31	0.6311	0.4211	0.2200	0.0312*
H41	0.4149	0.4821	0.3221	0.0342*
H61	0.5018	0.7567	0.1887	0.0272*
H101	0.8475	0.4431	0.0964	0.0447*
H102	0.9500	0.5403	0.1533	0.0452*
H103	0.8276	0.5340	−0.0221	0.0431*
H5	0.7748	0.7013	−0.0149	0.0311*
H8	1.0336	0.8715	0.1182	0.0457*
H9	0.9273	0.8294	−0.0462	0.0438*
H10	0.8710	0.9239	0.0250	0.0461*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0235 (5)	0.0222 (5)	0.0164 (5)	−0.0024 (4)	0.0070 (4)	−0.0003 (4)
C2	0.0248 (6)	0.0239 (6)	0.0171 (5)	0.0011 (4)	0.0051 (4)	−0.0005 (4)
C3	0.0320 (6)	0.0212 (5)	0.0262 (6)	−0.0002 (5)	0.0080 (5)	0.0016 (4)
C4	0.0291 (6)	0.0261 (6)	0.0279 (6)	−0.0061 (5)	0.0106 (5)	0.0024 (5)
C5	0.0224 (5)	0.0280 (6)	0.0239 (6)	−0.0023 (5)	0.0099 (5)	−0.0012 (5)
C6	0.0252 (6)	0.0215 (5)	0.0220 (5)	−0.0002 (4)	0.0087 (4)	−0.0001 (4)
N7	0.0290 (5)	0.0327 (6)	0.0369 (6)	−0.0024 (4)	0.0181 (5)	0.0000 (5)
O8	0.0616 (7)	0.0468 (6)	0.0910 (9)	0.0008 (5)	0.0600 (7)	0.0134 (6)
O9	0.0536 (7)	0.0333 (5)	0.0804 (8)	0.0089 (5)	0.0462 (6)	0.0061 (5)
C10	0.0329 (6)	0.0285 (6)	0.0276 (6)	0.0080 (5)	0.0124 (5)	0.0019 (5)
N11	0.0304 (5)	0.0236 (5)	0.0194 (5)	−0.0022 (4)	0.0153 (4)	−0.0022 (4)
C12	0.0228 (5)	0.0222 (5)	0.0180 (5)	0.0021 (4)	0.0088 (4)	0.0022 (4)
O13	0.0400 (5)	0.0258 (4)	0.0225 (4)	−0.0042 (4)	0.0178 (4)	−0.0031 (3)
C14	0.0300 (6)	0.0317 (6)	0.0211 (6)	−0.0069 (5)	0.0119 (5)	0.0009 (5)

Geometric parameters (Å, º)

C1—C2	1.4030 (16)	N7—O8	1.2216 (14)
C1—C6	1.3899 (15)	N7—O9	1.2241 (14)
C1—N11	1.4198 (14)	C10—H101	0.973
C2—C3	1.3969 (16)	C10—H102	0.967
C2—C10	1.5023 (16)	C10—H103	0.982
C3—C4	1.3846 (17)	N11—C12	1.3528 (15)
C3—H31	0.962	N11—H5	0.857

C4—C5	1.3849 (17)	C12—O13	1.2342 (13)
C4—H41	0.945	C12—C14	1.4998 (15)
C5—C6	1.3842 (16)	C14—H8	0.943
C5—N7	1.4655 (15)	C14—H9	0.960
C6—H61	0.949	C14—H10	0.949
C2—C1—C6	120.84 (10)	O8—N7—O9	122.91 (11)
C2—C1—N11	119.30 (10)	C2—C10—H101	111.0
C6—C1—N11	119.86 (10)	C2—C10—H102	110.5
C1—C2—C3	118.35 (10)	H101—C10—H102	108.2
C1—C2—C10	120.99 (10)	C2—C10—H103	111.2
C3—C2—C10	120.64 (10)	H101—C10—H103	109.1
C2—C3—C4	121.79 (11)	H102—C10—H103	106.8
C2—C3—H31	118.6	C1—N11—C12	124.63 (9)
C4—C3—H31	119.6	C1—N11—H5	117.4
C3—C4—C5	117.93 (11)	C12—N11—H5	117.9
C3—C4—H41	122.0	N11—C12—O13	122.82 (10)
C5—C4—H41	120.1	N11—C12—C14	115.63 (9)
C4—C5—C6	122.58 (11)	O13—C12—C14	121.55 (10)
C4—C5—N7	118.75 (10)	C12—C14—H8	109.4
C6—C5—N7	118.67 (10)	C12—C14—H9	113.5
C1—C6—C5	118.48 (11)	H8—C14—H9	109.2
C1—C6—H61	121.4	C12—C14—H10	108.1
C5—C6—H61	120.1	H8—C14—H10	106.9
C5—N7—O8	118.20 (11)	H9—C14—H10	109.5
C5—N7—O9	118.88 (10)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H5···O13^{i}	0.86	2.06	2.911 (1)	175

Symmetry code: (i) x, $-y+3/2$, $z-1/2$.