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2-Acetamido-4,5-dinitrotoluene: a test molecule for the CCDC 'Blind Structure Prediction Test, 2004'

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.051
wR factor = 0.095
Data-to-parameter ratio = 12.9

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

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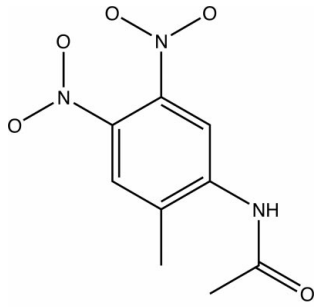
The structure of the title compound, $C_9H_9N_3O_5$, was determined as one of a group of five related compounds in order to assess its suitability as a test material for the 2004 Cambridge Crystallographic Data Centre 'Blind Structure Prediction Test'.

Comment

The Cambridge Crystallographic Data Centre '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 Professor Angelo 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).



(3)

The sample consisted of a mixture of crushed and broken fragments and some glass-clear pale yellow 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

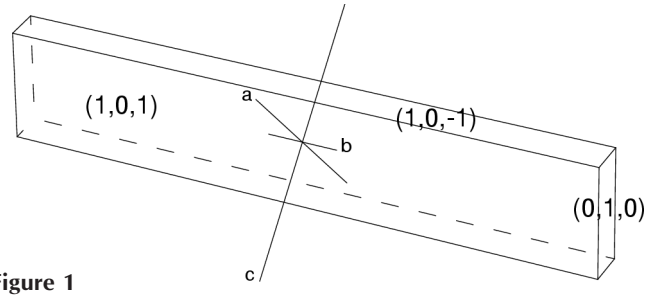


Figure 1
Perspective views of the crystal of (3), showing indices of principal faces and their relationship to the diffractometer axes.

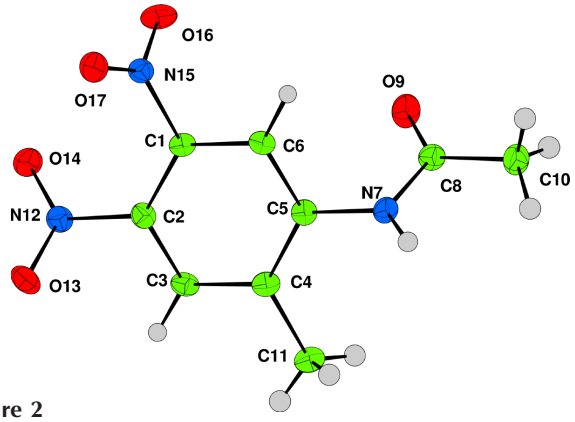


Figure 2
The molecule of the title compound, with displacement ellipsoids drawn at the 50% probability level. H-atom radii are proportional to U_{iso}.

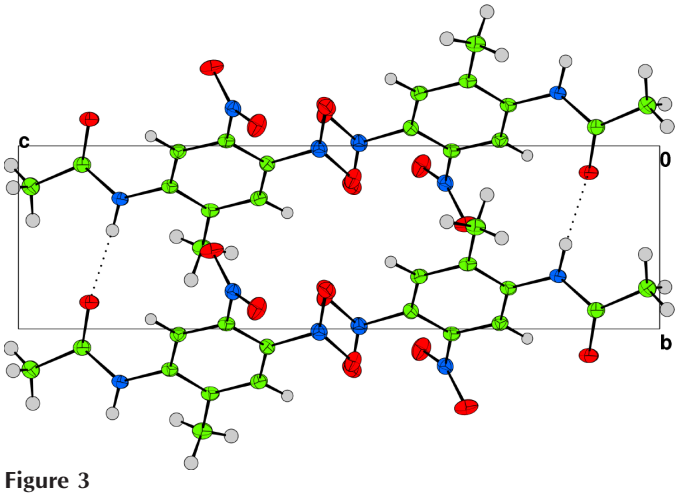


Figure 3
Packing diagram of the title compound, viewed parallel to the *a* axis, showing the hydrogen bonding as dashed lines.

length if any attempt was made to cut them into shorter lengths. Full data sets were collected for three samples [(1) 0.02 × 0.22 × 0.48 mm, 0.0021 mm^3; (2) 0.04 × 0.06 × 2.0 mm, 0.0048 mm^3; and (3) 0.04 × 0.15 × 0.83 mm, 0.0049 mm^3]. The first two samples were collected at 190 K and refined to *R*(all data) of 7.99 and 7.44%. The third sample was measured at 150 K and refined to 4.25%. Comparison of the atomic parameters for the first (smallest crystal) and third (most isometric crystal; Fig. 1) refinements had a mean atomic discrepancy of 0.0018 Å and an r.m.s. atomic discrepancy of 0.008 Å for the non-H atoms. The discrepancies between the U_{eq} values were larger, but probably not strictly comparable because of the temperature differences. The computed absorption corrections for the third sample perpendicular to the long axis are insignificant; it is presumed that the minimum and maximum scale factors reported by the multiscan calculation (*SCALEPACK*) are due to changes in illuminated volume (Görbitz, 1999). The results reported here are for the third sample only.

As reported by Demartin *et al.* (2004), the nitro groups are not coplanar with the benzene ring (Fig. 2). Those authors found that the torsion angles for a nitro group adjacent to another nitro group on one side and an H atom on the other

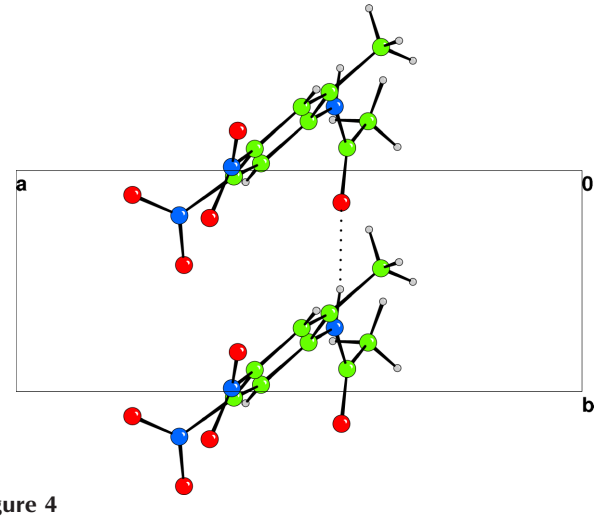


Figure 4
Part of the packing of the title compound, showing the parallel stacking of the benzene rings. The dashed line indicates a hydrogen bond.

fall in the interval 27–41°. In this case, the torsion angles are C1—C2—N12—O14 = 22.87 (19)° and C2—C1—N15—O17 = 52.08 (18)°. The acetamide group is itself almost planar [C10—C8—N7—C5 = 173.10 (12)°], but also inclined to the benzene ring [C6—C5—N7—C8 = −41.46 (19)°]. Hydrogen bonding between atom H71 of one molecule and O9 of an adjacent molecule causes the structure to consist of chains parallel to the *b* axis (Fig. 3). The benzene rings lie parallel to each other other with a perpendicular separation of 3.58 Å (Fig. 4). Other intermolecular contacts are unexceptional.

Experimental

Crystals were obtained by slow evaporation of an ethanol solution

Crystal data

$C_9H_9N_3O_5$
M_r = 239.19
Monoclinic, $P2_1/n$
a = 12.5693 (4) Å
b = 4.8531 (1) Å
c = 17.2663 (5) Å
β = 99.1624 (15)°
V = 1039.81 (5) Å^3
Z = 4
D_x = 1.528 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2364 reflections
θ = 5–27°
μ = 0.13 mm^{-1}
T = 150 K
Lath, pale yellow
0.83 × 0.15 × 0.04 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997)
T_{min} = 0.61, T_{max} = 0.99
4326 measured reflections
2357 independent reflections
2356 reflections with $I > 3\sigma(I)$
R_{int} = 0.035
θ_{max} = 27.5°
$h = -16 \rightarrow 16$
$k = -6 \rightarrow 5$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$= 0.051
$wR(F^2)$ = 0.095
S = 1.00
2356 reflections
182 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F^2) + 0.04 + 0.35p]$ where $p = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: 140 (20)

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.3958 (18)	C5—N7	1.4075 (17)
C1—C6	1.3723 (19)	N7—C8	1.3638 (18)
C1—N15	1.4716 (17)	C8—O9	1.2222 (18)
C2—C3	1.3812 (19)	C8—C10	1.500 (2)
C2—N12	1.4597 (17)	N12—O13	1.2313 (15)
C3—C4	1.3961 (19)	N12—O14	1.2273 (16)
C4—C5	1.4008 (18)	N15—O16	1.2216 (15)
C4—C11	1.5029 (19)	N15—O17	1.2202 (15)
C5—C6	1.3976 (19)		
C2—C1—C6	120.39 (12)	C6—C5—N7	119.43 (11)
C2—C1—N15	122.09 (12)	C5—C6—C1	119.70 (12)
C6—C1—N15	117.28 (11)	C5—N7—C8	124.40 (12)
C1—C2—C3	119.86 (12)	N7—C8—O9	121.95 (13)
C1—C2—N12	121.60 (12)	N7—C8—C10	115.49 (12)
C3—C2—N12	118.32 (11)	O9—C8—C10	122.56 (13)
C2—C3—C4	120.96 (12)	C2—N12—O13	118.02 (11)
C3—C4—C5	118.30 (12)	C2—N12—O14	117.94 (11)
C3—C4—C11	119.68 (12)	O13—N12—O14	124.00 (12)
C5—C4—C11	122.01 (12)	C1—N15—O16	117.18 (11)
C4—C5—C6	120.78 (12)	C1—N15—O17	117.65 (11)
C4—C5—N7	119.76 (12)	O16—N15—O17	125.09 (12)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N7—H71···O9^{i}	0.868 (19)	2.002 (19)	2.8632 (17)	171.2 (15)

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

All H atoms were seen in the difference electron-density map. Their positions and isotropic displacement parameters were regularized by several cycles of refinement using slack restraints, after which the refinement was completed using riding constraints. Reflection $\overline{1}3,3,10$ was omitted from the final refinement.

Data collection: *COLLECT* (Nonius, 1997–2001); cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK*; program(s) used to solve structure: *SIR*92 (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*.

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