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

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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.

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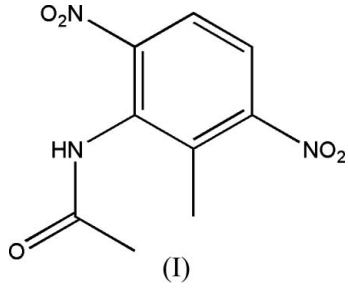
The structure of the title compound, $C_9H_9N_3O_5$, was determined as one of a group of five related compounds in order to assess its suitability as a test material for the 2004 Cambridge Crystallographic Data Centre 'Blind Structure Prediction Test'. The structure consists of hydrogen-bonded ribbons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

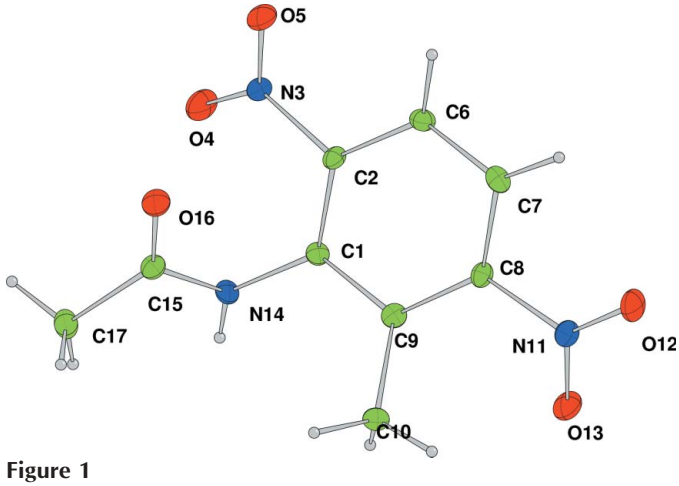


Figure 1
The title compound, with atomic displacement parameters drawn at the 50% probability level and the H atoms with arbitary radii.

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.

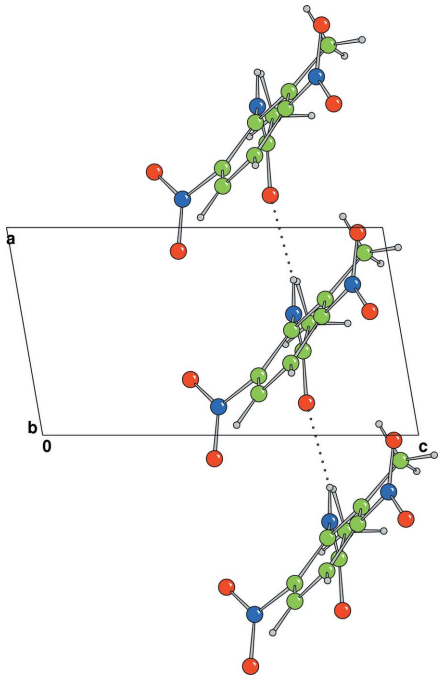


Figure 2
Projection along the *b* axis, showing the hydrogen bonding (dotted lines) and the aromatic ring stacking.

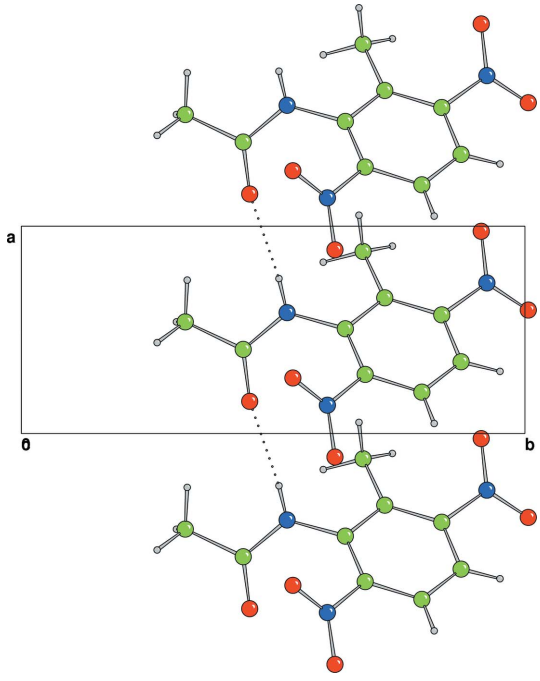


Figure 3
Projection along the *a* axis, showing the hydrogen-bonded (dotted lines) chain, with the benzene rings all on the same side of the *c* axis.

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*.

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