Acta Crystallographica Section E
Structure Reports Online

ISSN 1600-5368

3-Iodo-2,6-dinitrotoluene

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Received 29 July 2005
Accepted 3 August 2005
Online 12 August 2005

Key indicators

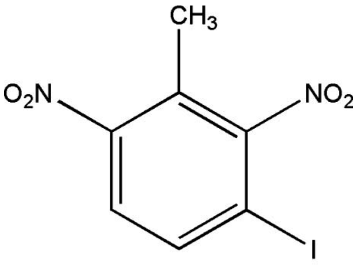
Single-crystal X-ray study
T = 110 K
Mean σ(C–C) = 0.005 Å
R factor = 0.051
wR factor = 0.067
Data-to-parameter ratio = 15.1

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, $C_7H_5IN_2O_4$, 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 crystal structure consists of columns of nearly planar molecules stacked parallel to the *a* axis, with an interplanar spacing of 3.478 (3) Å.

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), although it was not used in the test.



(I)

The sample consisted of chunky opaque pale-cream flakes. Attempts were made to obtain a roughly isometric sample, but the specimens had a tendency to crush. A suitable fragment was chosen on the basis of its sharp diffraction pattern and

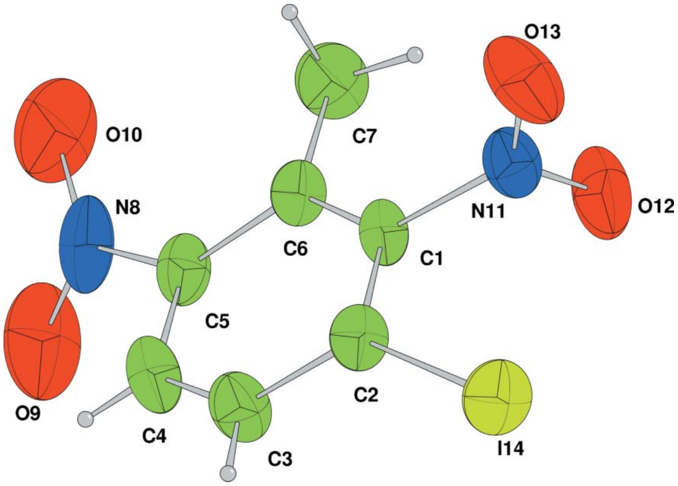


Figure 1
A view of the molecule of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitary radii.

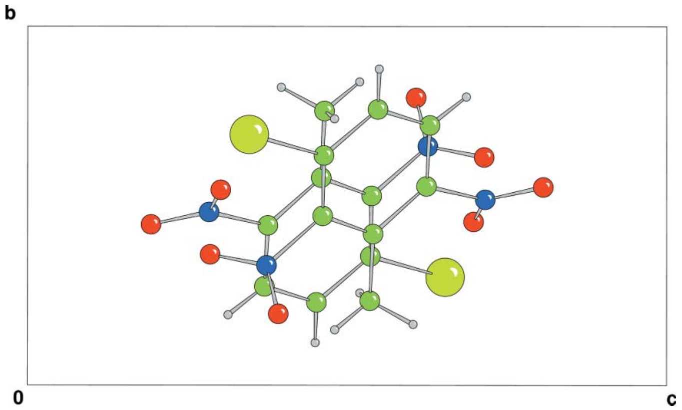


Figure 2
Diagram showing a column of molecules viewed end-on, parallel to the *a* axis.

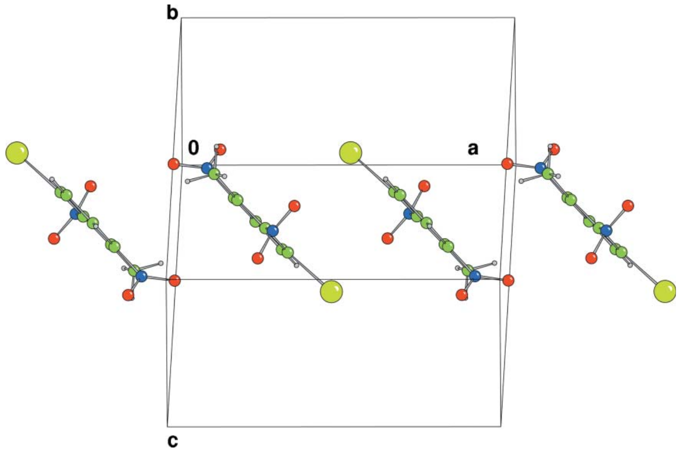


Figure 3
Diagram showing a column of molecules viewed approximately perpendicular to the column axis and parallel to the plane of the benzene group.

data were initially collected at 263 K, because of the fragililty of the material. A further data set was then collected on the same crystal at 110 K without any problems, and which gave essentially the same structure.

The methyl atom H71 is almost coplanar with the benzene group [H71—C7—C6—C5 = −165 (1)°], as are the I and N atoms (deviations of 0.02, 0.06 and 0.07 Å, respectively). The two nitro groups are rotated out of the plane of the benzene group [O12—N11—C1—C2 = 98.0 (3)° and O9—N8—C5—C4 = −42.9 (3)°] (Fig. 1). Except for the O atoms, the atomic displacement parameters conform to a rigid group ($R_{\rm TLS}$ = 0.09), with the principal axis of libration at 80 (1)° to the normal to the plane through the C atoms.

The structure of (I) consists of columns of molecules stacked along the *a* axis, with an interplanar separation of 3.780 (3) Å (Fig. 2). There are no hydrogen bonds (Fig. 3) and the only exceptionally short intermolecular contacts between the columns are from atom I14 to atoms O12 and O13 in an adjacent molecule [3.368 (3) and 3.481 (3) Å, respectively].

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

$C_7H_5IN_2O_4$
M_r = 308.03
Monoclinic, $P2_1/c$
a = 11.0997 (5) Å
b = 6.9493 (3) Å
c = 12.3296 (5) Å
β = 92.084 (2)°
V = 950.42 (7) Å^3
Z = 4

D_x = 2.153 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3027 reflections
θ = 5–27°
μ = 3.36 mm^{-1}
T = 110 K
Block, pale yellow
0.15 × 0.10 × 0.10 mm

Data collection

Nonius KappaCCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*DENZO/SCALEPACK*; Otwinowski & Minor, 1997)
$T_{\rm min}$ = 0.65, $T_{\rm max}$ = 0.71
9545 measured reflections

2140 independent reflections
2140 reflections with $I > 10\sigma(I)$
$R_{\rm int}$ = 0.033
$\theta_{\rm max}$ = 27.5°
$h = -13 \rightarrow 14$
$k = -7 \rightarrow 9$
$l = -16 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.067
S = 0.89
2140 reflections
142 parameters

Only H-atom coordinates refined
$w = 1/[\sigma^2(F^2) + (0.01P)^2]$
where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max}$ = 0.001
$\Delta\rho_{\rm max}$ = 0.89 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.95 e Å^{-3}

Table 1
Selected contact distances (Å).

I14···O12$^{\rm i}$	3.368 (3)	I14···O13$^{\rm i}$	3.481 (3)

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

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 distances in the range 0.93–0.98 Å) and displacement parameters [$U_{\rm iso}$(H) in the range 1.2–1.5 times $U_{\rm eq}$ of the parent atom], after which they were refined freely.

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*.

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 Milan, for generously donating the samples, and Professor Anna Arnoldi for help in the retrieval of the crystals.

Acta Cryst. (2005). E**61**, o2836–o2838 Watkin *et al.* • $C_7H_5IN_2O_4$ **o2837**

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.

Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, C. K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.

Demartin, F., Filippini, G., Gavezzotti, A. & Rizzato, S. (2004). *Acta Cryst.* B**60**, 609–620.

Nonius (2001). *COLLECT*. Nonius BV, Delft, The Netherlands.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.

Watkin, D. J., Motherwell, W. D. S., Cooper, R. I. & Pantos, S. (2004). *Acta Cryst.* E**60**, o2295–o2297.

Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). *CAMERON*. Chemical Crystallography Laboratory, University of Oxford, England.

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