Crystal structure of 1,3,5-trimethyl-2,4-dinitrobenzene

Ouarda Brihi,[a]* Noudjoud Hamdouni,[a] Ali Boudjada[a] and Jean Meinnel[b]

[a]Laboratoire de Cristallographie, Département de Physique, Université Mentouri-Constantine, 25000 Constantine, Algeria, and [b]UMR 6226 CNRS–Université Rennes 1 'Sciences Chimiques de Rennes', Equipe 'Matière Condensée et Systèmes Electroactifs', 263 Avenue du Général Leclerc, F-35042 Rennes, France. *Correspondence e-mail: ouardabrihi@yahoo.fr

In the title compound, $C_9H_{10}N_2O_4$, the planes of the nitro groups subtend dihedral angles of 55.04 (15) and 63.23 (15)° with that of the aromatic ring. These tilts are in opposite senses and the molecule possesses approximate mirror symmetry about a plane normal to the molecule. In the crystal, molecules form stacks in the [100] direction with adjacent molecules related by translation, although the centroid–centroid separation of 4.136 (5) Å is probably too long to regard as a significant aromatic π–π stacking interaction. An extremely weak C—H···O interaction is also present.

1. Related literature

For the structures and properties of related compounds, see: Tazi *et al.* (1995); Hernandez *et al.* (2003).
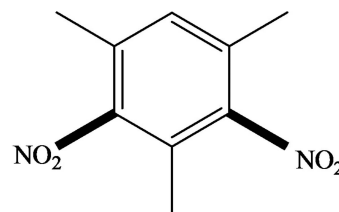
2. Experimental

2.1. Crystal data

$C_9H_{10}N_2O_4$
$M_r = 210.19$
Orthorhombic, $P2_12_12_1$
$a = 4.136$ (5) Å
$b = 13.916$ (5) Å
$c = 17.194$ (5) Å

$V = 989.6$ (13) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.11$ mm^{-1}
$T = 293$ K
$0.1 \times 0.08 \times 0.08$ mm

2.2. Data collection

Oxford Diffraction Xcalibur diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
$T_{min} = 0.618$, $T_{max} = 1.000$

3941 measured reflections
2730 independent reflections
1302 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
Standard reflections: ?

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.113$
$S = 0.93$
2730 reflections
139 parameters

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.12$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7C···O1^i	0.96	2.60	3.232 (4)	124

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

Data collection: *CrysAlis RED* (Oxford Diffraction, 2002); cell refinement: *CrysAlis RED*; data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

References

Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. & Spagna, R. (2005). *J. Appl. Cryst.* **38**, 381–388.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Hernandez, O., Cousson, A., Plazanet, M., Nierlich, M. & Meinnel, J. (2003). *Acta Cryst.* **C59**, o445–o450.

Oxford Diffraction (2002). *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, England.

Oxford Diffraction (2010). *CrysAlis PRO*. Oxford Diffraction Ltd, Abingdon, England.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Tazi, M., Meinnel, J., Sanquer, M., Nusimovici, M., Tonnard, F. & Carrie, R. (1995). *Acta Cryst.* B**51**, 838–847.

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

supporting information

Crystal structure of 1,3,5-trimethyl-2,4-dinitrobenzene

Ouarda Brihi, Noudjoud Hamdouni, Ali Boudjada and Jean Meinnel

S1. Experimental

The commercially available compound (Sigma-Aldrich) Was recrystallized from ethanol solution.

S2. Refinement

All non-H atoms were refined with anisotropic atomic displacement parameters. All H atoms were localized in a Fourier maps but introduced in calculated positions and treated as riding on their parent C atoms with C_{aryl}—H_{aryl}=0.93 Å; C_{methyl}—H_{methyl}=0.96 Å and $U_{iso}(H_{methyl})$=1.5$U_{eq}(C_{methyl})$ or $U_{iso}(H_{aryl})$=1.2$U_{eq}(C_{aryl})$. The atoms of benzene cycle present parameters of atomic displacements weaker than those of the substituent atoms.
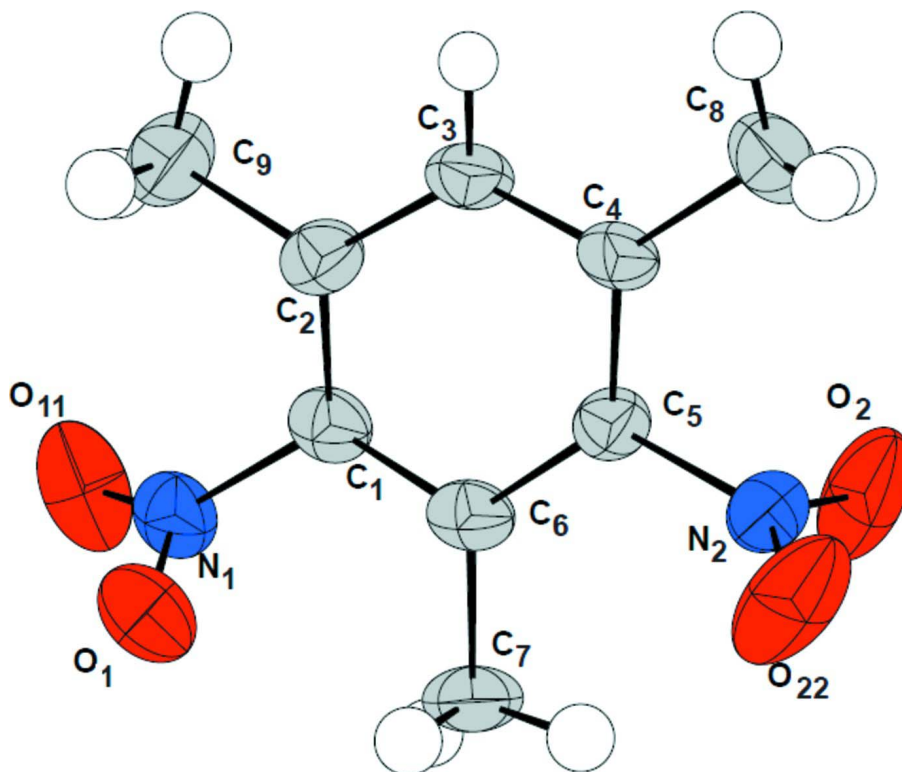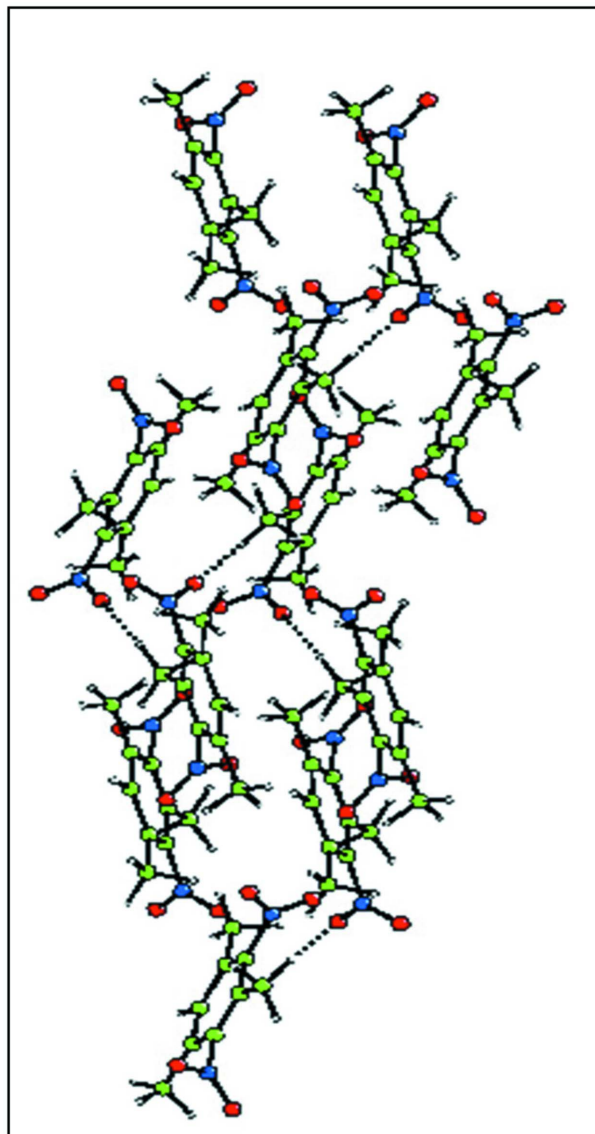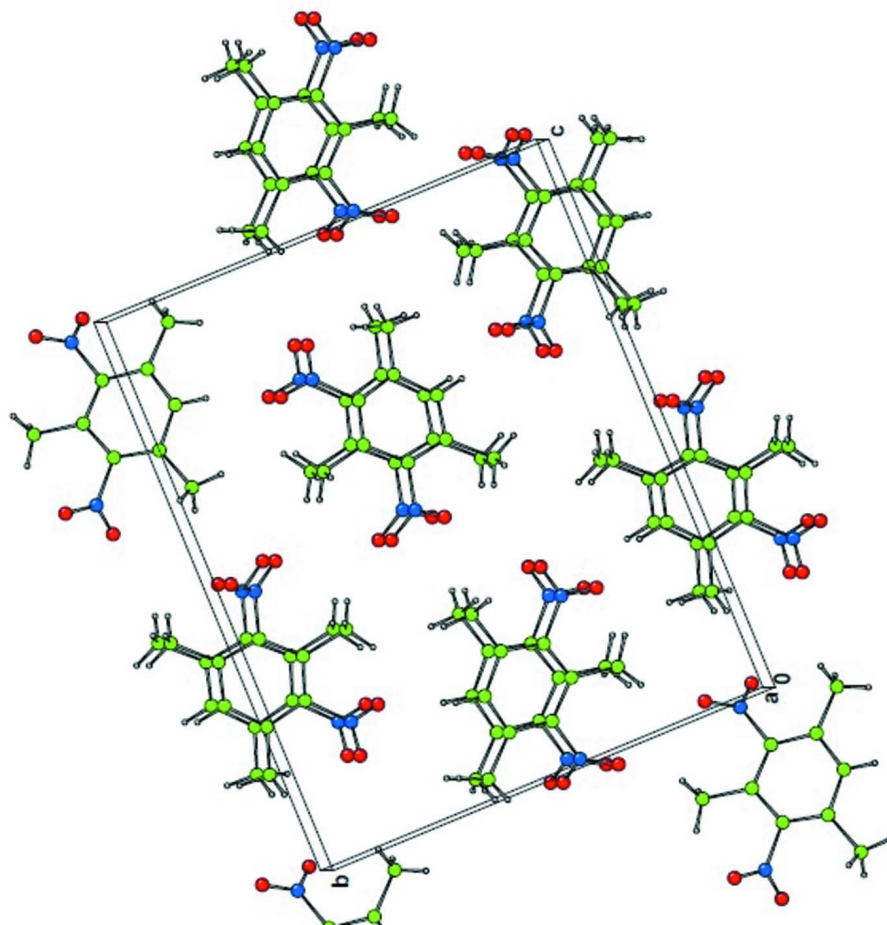

Figure 1

The molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level.

Figure 2
The crystal packing of (I) at 293 K, along the *b* axis.

Figure 3

The crystal packing of (I) at 293 K, according to the direction [100].

1,3,5-Trimethyl-2,4-dinitrobenzene

Crystal data

$C_9H_{10}N_2O_4$	$F(000) = 440$
$M_r = 210.19$	$D_x = 1.411$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 1062 reflections
$a = 4.136$ (5) Å	$\theta = 3.8–25.0°$
$b = 13.916$ (5) Å	$\mu = 0.11$ mm^{-1}
$c = 17.194$ (5) Å	$T = 293$ K
$V = 989.6$ (13) Å^3	Needle, colourless
$Z = 4$	$0.1 \times 0.08 \times 0.08$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer	Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
Radiation source: Enhance (Mo) X-ray Source	$T_{min} = 0.618$, $T_{max} = 1.000$
Graphite monochromator	3941 measured reflections
CCD scans	2730 independent reflections
	1302 reflections with $I > 2\sigma(I)$

$R_{int} = 0.032$

$\theta_{max} = 32.2°$, $\theta_{min} = 3.2°$

$h = -5 \rightarrow 3$

$k = -19 \rightarrow 17$

$l = -25 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.053$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.113$	
$S = 0.93$	H atoms treated by a mixture of independent and constrained refinement
2730 reflections	
139 parameters	$w = 1/[\sigma^2(F_o^2) + (0.0312P)^2]$
0 restraints	where $P = (F_o^2 + 2F_c^2)/3$
Primary atom site location: structure-invariant direct methods	$(\Delta/\sigma)_{max} < 0.001$
	$\Delta\rho_{max} = 0.12$ e Å^{-3}
	$\Delta\rho_{min} = -0.15$ e Å^{-3}

Special details

Experimental. Absorption correction: CrysAlisPro, Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	$U_{iso}*/U_{eq}$
O1	0.5095 (6)	0.13682 (16)	0.51948 (12)	0.0830 (9)
O2	0.4780 (6)	0.14379 (17)	0.16111 (14)	0.0975 (10)
O11	0.1589 (6)	0.0227 (2)	0.51482 (12)	0.0930 (9)
O22	0.8082 (7)	0.22717 (18)	0.22671 (15)	0.1057 (11)
N1	0.3785 (6)	0.06911 (19)	0.48712 (13)	0.0612 (9)
N2	0.6374 (6)	0.15688 (17)	0.21810 (14)	0.0576 (8)
C1	0.4961 (5)	0.04214 (19)	0.40898 (13)	0.0446 (8)
C2	0.5978 (6)	−0.05263 (18)	0.39734 (15)	0.0482 (8)
C3	0.7215 (6)	−0.07338 (17)	0.32487 (16)	0.0501 (9)
C4	0.7380 (6)	−0.00732 (19)	0.26478 (14)	0.0466 (8)
C5	0.6263 (6)	0.08533 (17)	0.28156 (15)	0.0439 (8)
C6	0.5084 (5)	0.11344 (17)	0.35299 (14)	0.0435 (8)
C7	0.3848 (8)	0.21448 (18)	0.36780 (16)	0.0664 (11)
C8	0.8759 (7)	−0.0350 (2)	0.18697 (15)	0.0632 (10)
C9	0.5834 (7)	−0.1295 (2)	0.45897 (17)	0.0701 (11)
H3	0.79817	−0.13520	0.31580	0.0601*
H7A	0.30028	0.24079	0.32036	0.0995*
H7B	0.55903	0.25404	0.38611	0.0995*
H7C	0.21682	0.21253	0.40631	0.0995*
H8A	1.00516	0.01682	0.16721	0.0947*

H8B	0.70269	−0.04818	0.15135	0.0947*
H8C	1.00773	−0.09134	0.19269	0.0947*
H9A	0.66925	−0.10482	0.50682	0.1050*
H9B	0.70885	−0.18397	0.44265	0.1050*
H9C	0.36286	−0.14884	0.46668	0.1050*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1139 (17)	0.0722 (16)	0.0629 (13)	−0.0037 (15)	−0.0125 (13)	−0.0276 (12)
O2	0.130 (2)	0.0873 (18)	0.0751 (14)	−0.0364 (15)	−0.0386 (16)	0.0295 (14)
O11	0.0756 (14)	0.129 (2)	0.0743 (14)	−0.0118 (16)	0.0166 (12)	−0.0084 (15)
O22	0.126 (2)	0.0670 (16)	0.124 (2)	−0.0504 (15)	−0.0339 (17)	0.0323 (15)
N1	0.0600 (14)	0.0690 (18)	0.0547 (14)	0.0088 (14)	−0.0066 (13)	−0.0055 (14)
N2	0.0685 (15)	0.0394 (13)	0.0648 (15)	−0.0027 (12)	−0.0067 (14)	0.0032 (13)
C1	0.0449 (13)	0.0466 (14)	0.0424 (13)	−0.0009 (12)	−0.0070 (12)	−0.0089 (12)
C2	0.0508 (14)	0.0394 (14)	0.0545 (15)	−0.0036 (12)	−0.0098 (13)	−0.0029 (13)
C3	0.0570 (16)	0.0294 (12)	0.0639 (17)	0.0000 (11)	−0.0025 (14)	−0.0069 (13)
C4	0.0507 (14)	0.0362 (14)	0.0529 (14)	−0.0032 (11)	−0.0020 (12)	−0.0082 (13)
C5	0.0477 (13)	0.0327 (12)	0.0513 (15)	−0.0047 (11)	−0.0106 (13)	0.0033 (12)
C6	0.0437 (13)	0.0353 (12)	0.0515 (15)	0.0042 (11)	−0.0110 (12)	−0.0090 (12)
C7	0.083 (2)	0.0434 (16)	0.0727 (19)	0.0206 (15)	−0.0153 (16)	−0.0123 (15)
C8	0.0709 (19)	0.0503 (16)	0.0683 (17)	−0.0045 (15)	0.0121 (15)	−0.0125 (15)
C9	0.082 (2)	0.0544 (19)	0.074 (2)	−0.0040 (18)	−0.0028 (16)	0.0144 (16)

Geometric parameters (Å, º)

O1—N1	1.221 (3)	C5—C6	1.378 (3)
O2—N2	1.195 (3)	C6—C7	1.518 (4)
O11—N1	1.212 (4)	C3—H3	0.9300
O22—N2	1.216 (4)	C7—H7A	0.9600
N1—C1	1.477 (3)	C7—H7B	0.9600
N2—C5	1.478 (3)	C7—H7C	0.9600
C1—C2	1.399 (4)	C8—H8A	0.9600
C1—C6	1.383 (3)	C8—H8B	0.9600
C2—C3	1.378 (4)	C8—H8C	0.9600
C2—C9	1.507 (4)	C9—H9A	0.9600
C3—C4	1.385 (4)	C9—H9B	0.9600
C4—C5	1.400 (4)	C9—H9C	0.9600
C4—C8	1.505 (4)		
O1—N1—O11	124.4 (2)	C5—C6—C7	122.1 (2)
O1—N1—C1	117.7 (2)	C2—C3—H3	118.00
O11—N1—C1	118.0 (2)	C4—C3—H3	118.00
O2—N2—O22	122.9 (3)	C6—C7—H7A	109.00
O2—N2—C5	119.1 (2)	C6—C7—H7B	109.00
O22—N2—C5	118.1 (2)	C6—C7—H7C	109.00
N1—C1—C2	118.0 (2)	H7A—C7—H7B	109.00

N1—C1—C6	117.6 (2)	H7A—C7—H7C	109.00
C2—C1—C6	124.5 (2)	H7B—C7—H7C	109.00
C1—C2—C3	116.0 (2)	C4—C8—H8A	109.00
C1—C2—C9	123.8 (2)	C4—C8—H8B	109.00
C3—C2—C9	120.1 (2)	C4—C8—H8C	109.00
C2—C3—C4	123.7 (2)	H8A—C8—H8B	109.00
C3—C4—C5	116.2 (2)	H8A—C8—H8C	109.00
C3—C4—C8	120.8 (2)	H8B—C8—H8C	109.00
C5—C4—C8	123.0 (2)	C2—C9—H9A	110.00
N2—C5—C4	117.3 (2)	C2—C9—H9B	109.00
N2—C5—C6	118.5 (2)	C2—C9—H9C	109.00
C4—C5—C6	124.2 (2)	H9A—C9—H9B	109.00
C1—C6—C5	115.4 (2)	H9A—C9—H9C	109.00
C1—C6—C7	122.4 (2)	H9B—C9—H9C	109.00
O1—N1—C1—C2	124.4 (3)	C6—C1—C2—C3	1.0 (4)
O11—N1—C1—C2	−55.6 (3)	N1—C1—C6—C5	178.6 (2)
O1—N1—C1—C6	−53.5 (3)	C1—C2—C3—C4	−2.2 (4)
O11—N1—C1—C6	126.6 (3)	C9—C2—C3—C4	178.9 (2)
O2—N2—C5—C6	−116.9 (3)	C2—C3—C4—C5	1.3 (4)
O2—N2—C5—C4	63.2 (3)	C2—C3—C4—C8	−179.9 (2)
O22—N2—C5—C4	−117.2 (3)	C3—C4—C5—C6	0.9 (4)
O22—N2—C5—C6	62.7 (3)	C8—C4—C5—N2	2.0 (4)
N1—C1—C2—C3	−176.7 (2)	C8—C4—C5—C6	−177.9 (2)
N1—C1—C6—C7	−4.4 (3)	C3—C4—C5—N2	−179.2 (2)
C2—C1—C6—C5	1.0 (3)	N2—C5—C6—C1	178.2 (2)
C2—C1—C6—C7	178.0 (2)	N2—C5—C6—C7	1.2 (4)
C6—C1—C2—C9	179.9 (2)	C4—C5—C6—C1	−1.9 (4)
N1—C1—C2—C9	2.2 (4)	C4—C5—C6—C7	−179.0 (2)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7*C*···O1[i]	0.96	2.60	3.232 (4)	124

Symmetry code: (i) *x*−1/2, −*y*+1/2, −*z*+1.