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2,3-Bis(bromomethyl)-1-methoxy-4-nitrobenzene

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.007 Å
R factor = 0.040
wR factor = 0.133
Data-to-parameter ratio = 17.9

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

In the title compound, $C_9H_9Br_2NO_3$, the molecules are linked by intermolecular C—H···O hydrogen bonds.

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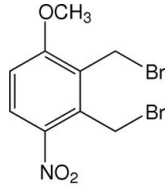
Comment

1,2-Bis(bromomethyl)benzene and its derivatives are important building blocks in supramolecular chemistry (Wu *et al.*, 2002). In this paper, we report the crystal structure of the title compound, (I) (Fig. 1). The bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The crystal structure is stabilized by intermolecular C—H···O interactions (Table 2 and Fig. 2)



(I)

Experimental

The title compound was synthesized by bromination of 1-methoxy-2,3-dimethyl-4-nitrobenzene with *N*-bromosuccinimide in CCl_4, according to the literature method (Lai & Yap, 1993; Knölker *et al.*, 1993). Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of a solution in hexane–MeOH (2:1).

Crystal data

$C_9H_9Br_2NO_3$
M_r = 338.99
Orthorhombic, $P2_12_12_1$
a = 4.7900 (9) Å
b = 13.772 (3) Å
c = 17.158 (3) Å
V = 1131.9 (4) Å^3
Z = 4
D_x = 1.989 Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 2142 reflections
θ = 2.4–23.8°
μ = 7.15 mm^{-1}
T = 292 (2) K
Block, colorless
0.20 × 0.20 × 0.20 mm

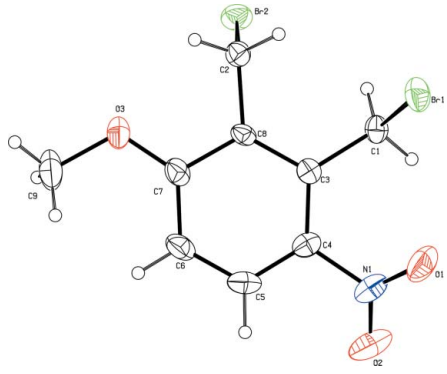


Figure 1
A view of the molecule of (I), showing the atom-labeling scheme and 50% probability displacement ellipsoids.

Data collection

Bruker SMART APEX CCD area detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.239, T_{max} = 0.243
6493 measured reflections
2454 independent reflections
1920 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 27.0°
$h = -5 \rightarrow 6$
$k = -14 \rightarrow 17$
$l = -20 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.133
S = 1.13
2454 reflections
137 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0709P)^2 + 0.0631P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.74 e Å^{-3}
$\Delta\rho_{min}$ = −0.68 e Å^{-3}
Absolute structure: Flack (1983), 1465 Friedel pairs
Flack parameter: 0.04 (3)

Table 1
Selected geometric parameters (Å, °).

Br1—C1	1.953 (5)	C9—O3	1.446 (6)
Br2—C2	1.993 (5)	N1—O2	1.199 (6)
C4—N1	1.452 (6)	N1—O1	1.235 (6)
C7—O3	1.327 (6)		
C3—C1—Br1	114.6 (3)	O2—N1—C4	118.4 (4)
C8—C2—Br2	108.7 (3)	O1—N1—C4	116.6 (4)
O2—N1—O1	125.0 (5)	C7—O3—C9	117.4 (4)
C8—C3—C4—N1	−175.5 (4)	C5—C6—C7—O3	177.4 (5)
N1—C4—C5—C6	174.5 (5)	O3—C7—C8—C3	−178.8 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O1^{i}	0.93	2.43	3.216 (6)	143

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

After their location in a difference Fourier map, H atoms were placed in calculated positions and allowed to ride on their parent atoms, with C—H = 0.93–0.97 Å, and U_{iso}(H) = 1.2 − 1.5 U_{eq}(C).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999)'; data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

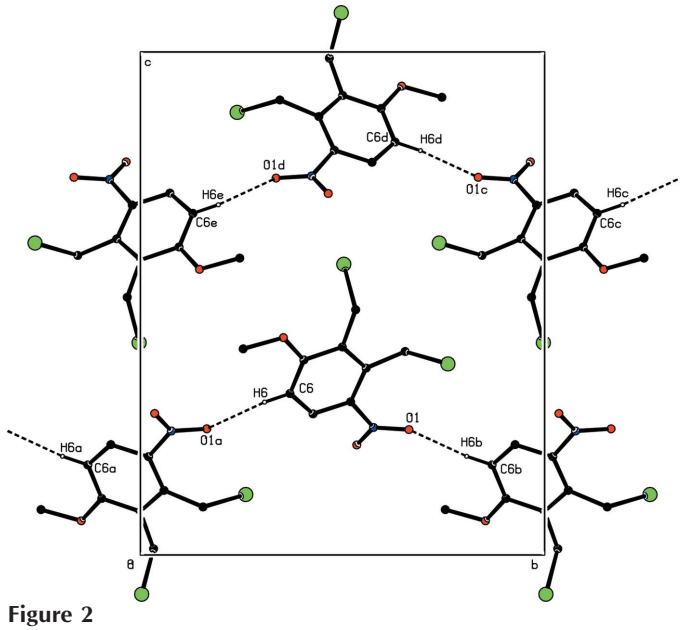


Figure 2
C—H···O intermolecular hydrogen-bonding, shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted. [Symmetry codes: (a) $1-x, -\frac{1}{2}+y, \frac{1}{2}-z$; (b) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$; (c) $\frac{1}{2}+x, -\frac{1}{2}-y, 1-z$; (d) $\frac{1}{2}-x, 1-y, \frac{1}{2}+z$; (e) $\frac{1}{2}+x, \frac{1}{2}-y, 1-z$.]

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