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1,3-Bis(chloromethyl)-2-methyl-5-nitrobenzene

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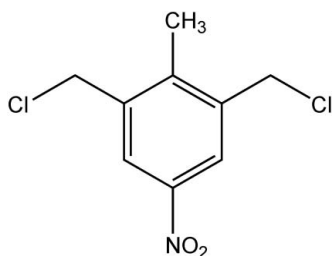
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 Key indicators: single-crystal X-ray study; $T = 273$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.036; wR factor = 0.106; data-to-parameter ratio = 16.7.

The title compound, $\text{C}_9\text{H}_9\text{Cl}_2\text{NO}_2$, is a natural product isolated from the endophytic fungus No. B77 of the mangrove tree from the South China Sea coast. In the crystal structure, the molecules lie on twofold axes and form offset stacks through face-to-face π - π interactions. Adjacent molecules in each stack are related by a centre of inversion and have an interplanar separation of 3.53 (1) Å, with a centroid-centroid distance of 3.76 (1) Å. Between stacks, there are $\text{C}-\text{H}\cdots\text{O}$ interactions to the nitro groups and $\text{Cl}\cdots\text{Cl}$ contacts of 3.462 (1) Å.

Related literature

For related literature, see: McBee (1951).



Experimental

Crystal data

$\text{C}_9\text{H}_9\text{Cl}_2\text{NO}_2$
 $M_r = 234.07$
 Monoclinic, $C2/c$
 $a = 8.921$ (3) Å
 $b = 16.141$ (6) Å
 $c = 7.511$ (3) Å
 $\beta = 111.929$ (6)°
 $V = 1003.3$ (6) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.62$ mm⁻¹
 $T = 273$ (2) K
 $0.47 \times 0.38 \times 0.18$ mm

Data collection

Bruker SMART CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2001)
 $T_{\min} = 0.760$, $T_{\max} = 0.897$
 2900 measured reflections
 1113 independent reflections
 976 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.106$
 $S = 1.06$
 1102 reflections
 66 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.34$ e Å⁻³
 $\Delta\rho_{\min} = -0.39$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C6}-\text{H6A}\cdots\text{O1}^1$	0.97	2.66	3.427 (3)	136

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

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2284).

References

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 Bruker (2001). SADABS and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
 McBee, E. T. (1951). *J. Am. Chem. Soc.* **73**, 3932-3934.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112-122.

supporting information

Acta Cryst. (2008). E64, o731 [doi:10.1107/S1600536808007290]

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S1. Comment

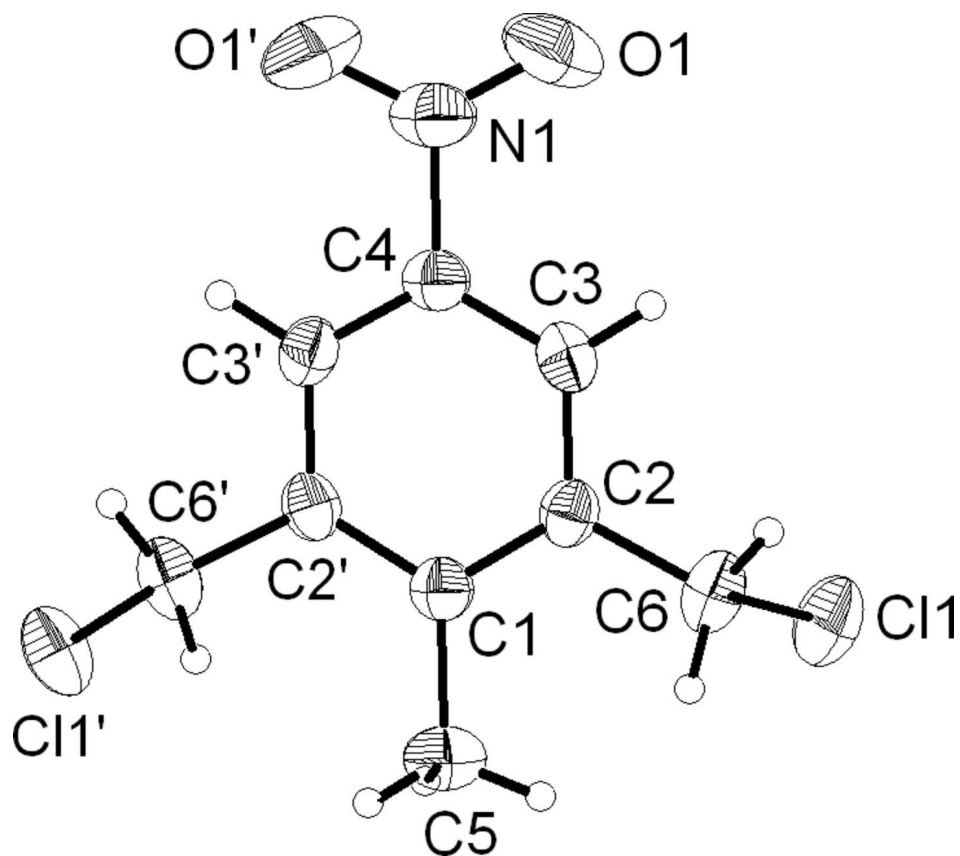
The title compound was isolated from the endophytic fungus No.B77 from the mangrove tree from the South China Sea coast. As far as we know, this compound has not been reported previously as a natural product, but only as a synthetic compound (Mcbee, 1951). The molecules lie on crystallographic twofold axes (Fig. 1) and form offset $\pi\cdots\pi$ stacks (Fig. 2).

S2. Experimental

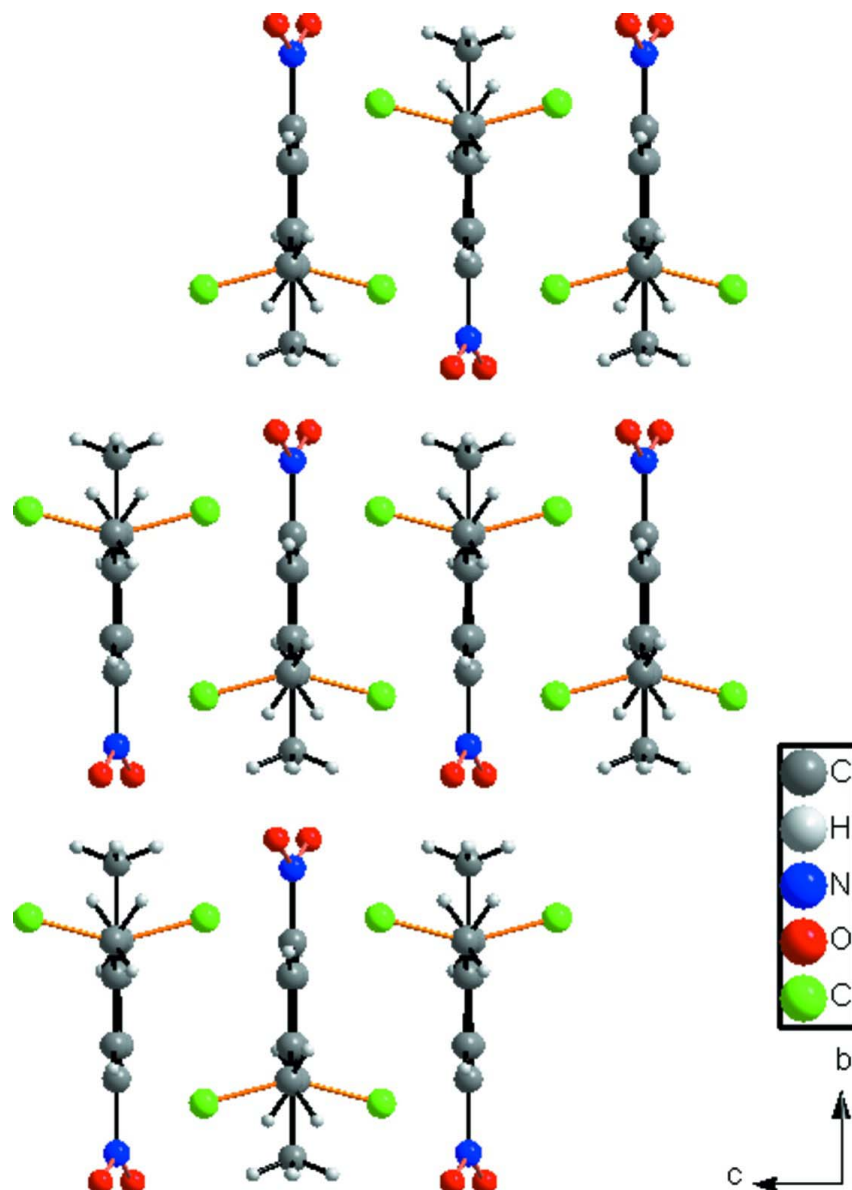
A strain of fungus (No. B77) was deposited in the Department of Applied Chemistry, Zhongshan University, Guangzhou, P. R. China. Culture conditions: GYT medium (glucose 10 g/L, peptone 2 g/L, yeast extract 1 g/L, NaCl 2.5 g/L) incubated at 298 K for 30 d. For the extraction and separation of the metabolite, the cultures (130 L) were filtered through cheesecloth, the filtrate was concentrated to 3 L below 323 K, then extracted three times by shaking with an equal volume of ethyl acetate. The extract was evaporated under reduced pressure and the combined organic extracts were subjected to silica-gel column chromatography, eluting with petroleum ether/ethyl acetate. Crystals of the title compound were obtained by evaporation of a methanol solution.

S3. Refinement

H atoms were positioned geometrically and treated as riding, with C—H = 0.93 (aromatic CH), 0.96 (methyl CH₃) or 0.97 Å (methylene CH₂), and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{CH})$ or $1.5U_{\text{eq}}(\text{CH}_3, \text{CH}_2)$.

**Figure 1**

The molecular structure with displacement ellipsoids at 30% probability for non-H atoms. Primed atoms are generated by the symmetry operator: $-x + 2, y, -z + 1/2$.

**Figure 2**View of the packing along the normal to the *bc*-plane.**11,3-Bis(chloromethyl)-2-methyl-5-nitrobenzene***Crystal data* $C_9H_6Cl_2NO_2$ $M_r = 234.07$ Monoclinic, *C2/c*Hall symbol: $-C\ 2yc$ $a = 8.921\ (3)\ \text{\AA}$ $b = 16.141\ (6)\ \text{\AA}$ $c = 7.511\ (3)\ \text{\AA}$ $\beta = 111.929\ (6)^\circ$ $V = 1003.3\ (6)\ \text{\AA}^3$ $Z = 4$ $F(000) = 480$ $D_x = 1.550\ \text{Mg m}^{-3}$ Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 711 reflections

 $\theta = 2.5\text{--}27.1^\circ$ $\mu = 0.62\ \text{mm}^{-1}$ $T = 273\ \text{K}$

Block, colourless

 $0.47 \times 0.38 \times 0.18\ \text{mm}$

Data collection

Bruker SMART CCD diffractometer	2900 measured reflections
Radiation source: fine-focus sealed tube	1113 independent reflections
Graphite monochromator	976 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.017$
Absorption correction: multi-scan (SADABS; Bruker, 2001)	$\theta_{\text{max}} = 27.1^\circ$, $\theta_{\text{min}} = 2.5^\circ$
$T_{\text{min}} = 0.760$, $T_{\text{max}} = 0.897$	$h = -11 \rightarrow 9$
	$k = -20 \rightarrow 19$
	$l = -9 \rightarrow 9$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.035$	H-atom parameters constrained
$wR(F^2) = 0.106$	$w = 1/[\sigma^2(F_o^2) + (0.0614P)^2 + 0.6055P]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
1102 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
66 parameters	$\Delta\rho_{\text{max}} = 0.34 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.39 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Cl1	0.58497 (5)	0.11383 (3)	-0.00170 (7)	0.0545 (2)	
N1	1.0000	-0.17528 (13)	0.2500	0.0507 (5)	
C3	0.86254 (17)	-0.04274 (9)	0.2433 (2)	0.0347 (3)	
H3A	0.7713	-0.0721	0.2379	0.042*	
C1	1.0000	0.08774 (13)	0.2500	0.0332 (4)	
C2	0.86253 (17)	0.04352 (9)	0.2446 (2)	0.0328 (3)	
C4	1.0000	-0.08388 (13)	0.2500	0.0347 (5)	
O1	0.8703 (2)	-0.21053 (9)	0.2040 (3)	0.0840 (6)	
C5	1.0000	0.18142 (15)	0.2500	0.0528 (6)	
H5A	0.8974	0.2012	0.2461	0.079*	0.50
H5B	1.0186	0.2012	0.1395	0.079*	0.50
H5C	1.0840	0.2012	0.3644	0.079*	0.50
C6	0.71110 (19)	0.08572 (12)	0.2402 (2)	0.0438 (4)	
H6A	0.7393	0.1351	0.3195	0.053*	
H6B	0.6520	0.0489	0.2925	0.053*	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0409 (3)	0.0665 (4)	0.0483 (3)	0.01565 (19)	0.0076 (2)	0.00775 (19)
N1	0.0643 (14)	0.0341 (10)	0.0476 (12)	0.000	0.0139 (10)	0.000
C3	0.0296 (7)	0.0409 (8)	0.0314 (7)	-0.0052 (6)	0.0088 (6)	0.0017 (6)
C1	0.0345 (10)	0.0339 (10)	0.0289 (10)	0.000	0.0092 (8)	0.000
C2	0.0292 (7)	0.0403 (8)	0.0276 (7)	0.0035 (6)	0.0091 (6)	0.0004 (5)
C4	0.0394 (11)	0.0311 (10)	0.0300 (10)	0.000	0.0087 (8)	0.000
O1	0.0821 (12)	0.0432 (8)	0.1149 (15)	-0.0220 (8)	0.0233 (11)	-0.0013 (9)
C5	0.0587 (16)	0.0335 (12)	0.0644 (17)	0.000	0.0209 (13)	0.000
C6	0.0346 (8)	0.0570 (10)	0.0398 (8)	0.0109 (7)	0.0139 (7)	0.0025 (7)

Geometric parameters (\AA , $^\circ$)

Cl1—C6	1.8017 (18)	C1—C5	1.512 (3)
N1—O1	1.2178 (19)	C2—C6	1.502 (2)
N1—C4	1.475 (3)	C5—H5A	0.960
C3—C4	1.3787 (19)	C5—H5B	0.960
C3—C2	1.392 (2)	C5—H5C	0.960
C3—H3A	0.930	C6—H6A	0.970
C1—C2	1.4064 (18)	C6—H6B	0.970
O1—N1—O1 ⁱ	124.3 (2)	C3—C4—N1	118.79 (10)
O1—N1—C4	117.85 (12)	C1—C5—H5A	109.5
O1 ⁱ —N1—C4	117.85 (12)	C1—C5—H5B	109.5
C4—C3—C2	118.94 (14)	H5A—C5—H5B	109.5
C4—C3—H3A	120.5	C1—C5—H5C	109.5
C2—C3—H3A	120.5	H5A—C5—H5C	109.5
C2—C1—C2 ⁱ	119.01 (19)	H5B—C5—H5C	109.5
C2—C1—C5	120.50 (10)	C2—C6—Cl1	110.75 (11)
C2 ⁱ —C1—C5	120.50 (10)	C2—C6—H6A	109.5
C3—C2—C1	120.34 (13)	Cl1—C6—H6A	109.5
C3—C2—C6	117.12 (14)	C2—C6—H6B	109.5
C1—C2—C6	122.53 (15)	Cl1—C6—H6B	109.5
C3 ⁱ —C4—C3	122.4 (2)	H6A—C6—H6B	108.1
C3 ⁱ —C4—N1	118.79 (10)		
C4—C3—C2—C1	0.91 (19)	C2—C3—C4—N1	179.54 (9)
C4—C3—C2—C6	-179.08 (11)	O1—N1—C4—C3 ⁱ	-164.92 (14)
C2 ⁱ —C1—C2—C3	-0.46 (10)	O1 ⁱ —N1—C4—C3 ⁱ	15.08 (14)
C5—C1—C2—C3	179.54 (10)	O1—N1—C4—C3	15.08 (14)
C2 ⁱ —C1—C2—C6	179.53 (15)	O1 ⁱ —N1—C4—C3	-164.92 (14)
C5—C1—C2—C6	-0.47 (15)	C3—C2—C6—Cl1	-95.84 (15)
C2—C3—C4—C3 ⁱ	-0.46 (9)	C1—C2—C6—Cl1	84.18 (15)

Symmetry code: (i) $-x+2, y, -z+1/2$.

Hydrogen-bond geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C6—H6 <i>A</i> ···O1 ⁱⁱ	0.97	2.66	3.427 (3)	136

Symmetry code: (ii) $-x+3/2, y+1/2, -z+1/2$.