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## Structure Reports

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## Dimethyl 5-nitroisophthalate

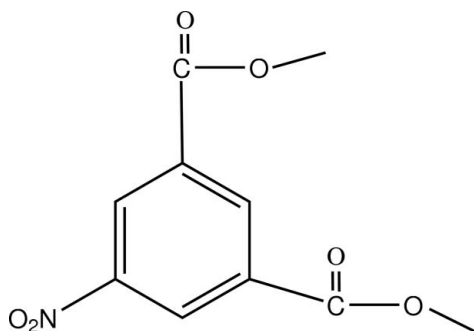
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Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  
 $R$  factor = 0.073;  $wR$  factor = 0.172; data-to-parameter ratio = 12.2.The nitro group in the title compound,  $\text{C}_{10}\text{H}_9\text{NO}_6$ , is rotated by  $10.9$  (5)° out of the plane of the benzene ring.

## Related literature

For related literature, see: Bjorsvik *et al.* (2001); Cutroneo *et al.* (2007); Enzweiler *et al.* (2006).

## Experimental

## Crystal data

 $\text{C}_{10}\text{H}_9\text{NO}_6$   
 $M_r = 239.18$ Triclinic,  $P\bar{1}$   
 $a = 4.0130$  (8) Å $b = 10.660$  (2) Å  
 $c = 12.643$  (3) Å  
 $\alpha = 106.11$  (3)°  
 $\beta = 93.74$  (3)°  
 $\gamma = 91.46$  (3)°  
 $V = 517.97$  (18) Å<sup>3</sup> $Z = 2$   
Mo  $K\alpha$  radiation  
 $\mu = 0.13$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 $0.40 \times 0.05 \times 0.05$  mm

## Data collection

Enraf–Nonius CAD-4  
diffractometer  
Absorption correction:  $\psi$  scan  
(North *et al.*, 1968)  
 $T_{\min} = 0.950$ ,  $T_{\max} = 0.994$   
2178 measured reflections1885 independent reflections  
1144 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.041$   
3 standard reflections  
every 200 reflections  
intensity decay: none

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.072$   
 $wR(F^2) = 0.172$   
 $S = 1.01$   
1885 reflections154 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.28$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.18$  e Å<sup>-3</sup>Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXL97*; software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CS2086).

## References

- Bjorsvik, H.-R., Priebe, H., Cervenka, J., Aabye, A. W., Gulbrandsen, T. & Bryde, A. C. (2001). *Org. Process Res. Dev.* **5**, 472–478.
- Cutroneo, P., Polimeni, G., Curcuruto, R., Calapai, G. & Caputi, A. P. (2007). *Pharmacol. Res.* **56**, 35–41.
- Enraf–Nonius (1989). *CAD-4 Software*. Enraf–Nonius, Delft, The Netherlands.
- Enzweiler, C. N. H., Höhn, S., Taupitz, M., Lembcke, A. E., Wiese, T. H., Hamm, B. & Kivelitz, D. E. (2006). *Acad. Radiol.* **13**, 95–103.
- Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

**supplementary materials**

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## Dimethyl 5-nitrosophthalate

M.-H. Xie, P. Zou, Y.-J. He, Y.-L. Liu and B. Huang

### Comment

The title molecule (Fig.1), is useful as an important intermediate for the preparation of iodinated *X*-ray contrast media, in particular non-ionic ones such as Iopamidol, Iohexol and Ioversol, being used clinically all over the world (Cutroneo *et al.*, 2007; Bjorsvik *et al.*, 2001; Enzweiler *et al.*, 2006). This crystal structure shows that the benzene ring and the nitro group are only slightly inclined, as shown by the torsion angles of O5—N—C7—C6 -10.6 (5)° and of O6—N—C7—C8 -11.2 (5)°.

### Experimental

5-nitrosophthalic acid (0.5 mmol, 100.6 mg) was dissolved in hot methanol (5 ml), then a drop of concentrated sulfuric acid was added and refluxed for 4 h. The precipitate was filtered off, washed with water and dissolved in 95% ethanol (20 mL). The solution was evaporated in air affording colourless needle crystals suitable for X-ray analysis (yield: 85.2%).

### Refinement

Positional parameters of all the H atoms bonded to C atoms were calculated geometrically and were allowed to ride on the C atoms to which they are bonded, with C—H = 0.93 (aromatic) and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  or 0.96 (methyl) and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ , respectively.

### Figures

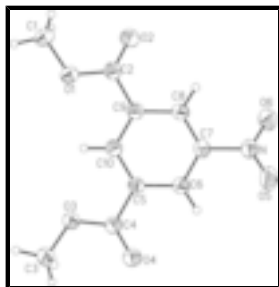


Fig. 1. A view of the title compound with the atomic numbering scheme. Displacement ellipsoids were drawn at the 30% probability level.

## Dimethyl 5-nitrobenzene-1,3-dicarboxylate

### Crystal data

$\text{C}_{10}\text{H}_9\text{NO}_6$

$M_r = 239.18$

Triclinic,  $P\bar{1}$

Hall symbol: -P 1

$Z = 2$

$F_{000} = 248$

$D_x = 1.534 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation

$\lambda = 0.71073 \text{ \AA}$

# supplementary materials

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$a = 4.0130$ (8) Å	Cell parameters from 25 reflections
$b = 10.660$ (2) Å	$\theta = 8\text{--}12^\circ$
$c = 12.643$ (3) Å	$\mu = 0.13$ mm <sup>-1</sup>
$\alpha = 106.11$ (3)°	$T = 293$ (2) K
$\beta = 93.74$ (3)°	Needle, colourless
$\gamma = 91.46$ (3)°	$0.40 \times 0.05 \times 0.05$ mm
$V = 517.97$ (18) Å <sup>3</sup>	

## Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.041$
Radiation source: fine-focus sealed tube	$\theta_{\text{max}} = 25.3^\circ$
Monochromator: graphite	$\theta_{\text{min}} = 1.7^\circ$
$T = 293$ (2) K	$h = -4 \rightarrow 4$
$\omega/2\theta$ scans	$k = -12 \rightarrow 12$
Absorption correction: $\psi$ scan (North <i>et al.</i> , 1968))	$l = 0 \rightarrow 15$
$T_{\text{min}} = 0.950$ , $T_{\text{max}} = 0.994$	3 standard reflections
2178 measured reflections	every 200 reflections
1885 independent reflections	intensity decay: none
1144 reflections with $I > 2\sigma(I)$	

## Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.073$	H-atom parameters constrained
$wR(F^2) = 0.172$	$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 0.6P]$
$S = 1.01$	where $P = (F_o^2 + 2F_c^2)/3$
1885 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
154 parameters	$\Delta\rho_{\text{max}} = 0.28$ e Å <sup>-3</sup>
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.18$ e Å <sup>-3</sup>
	Extinction correction: none

## Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N	0.5209 (9)	0.7040 (3)	0.8012 (3)	0.0572 (8)
O1	0.7307 (7)	0.1642 (2)	0.5059 (2)	0.0642 (8)
C1	0.8443 (12)	0.0907 (4)	0.4027 (3)	0.0717 (13)
H1A	0.8215	-0.0010	0.3965	0.108*
H1B	1.0748	0.1145	0.3992	0.108*
H1C	0.7126	0.1094	0.3431	0.108*
O2	0.8638 (9)	0.3487 (3)	0.4668 (2)	0.0825 (10)
C2	0.7532 (9)	0.2943 (3)	0.5284 (3)	0.0480 (9)

O3	0.2103 (7)	0.1601 (2)	0.8322 (2)	0.0608 (8)
C3	0.0691 (12)	0.0824 (4)	0.8961 (4)	0.0703 (12)
H3A	0.0833	-0.0086	0.8583	0.105*
H3B	-0.1610	0.1022	0.9054	0.105*
H3C	0.1906	0.1016	0.9671	0.105*
O4	0.1018 (8)	0.3426 (3)	0.9595 (2)	0.0739 (9)
C4	0.2108 (9)	0.2883 (3)	0.8735 (3)	0.0482 (9)
C5	0.3639 (8)	0.3588 (3)	0.8013 (3)	0.0424 (8)
O5	0.3683 (10)	0.7593 (3)	0.8783 (3)	0.0957 (12)
C6	0.3703 (9)	0.4947 (3)	0.8341 (3)	0.0465 (9)
H6A	0.2839	0.5401	0.8996	0.056*
O6	0.6840 (9)	0.7614 (3)	0.7524 (3)	0.0910 (11)
C7	0.5090 (8)	0.5598 (3)	0.7663 (3)	0.0445 (8)
C8	0.6352 (8)	0.4972 (3)	0.6687 (3)	0.0457 (8)
H8A	0.7276	0.5447	0.6254	0.055*
C9	0.6236 (9)	0.3612 (3)	0.6351 (3)	0.0448 (8)
C10	0.4871 (8)	0.2942 (3)	0.7022 (3)	0.0450 (8)
H10A	0.4781	0.2033	0.6802	0.054*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N	0.065 (2)	0.0474 (18)	0.061 (2)	0.0053 (16)	0.0112 (17)	0.0155 (16)
O1	0.091 (2)	0.0508 (15)	0.0511 (15)	0.0022 (14)	0.0238 (14)	0.0100 (12)
C1	0.099 (4)	0.062 (2)	0.054 (2)	0.013 (2)	0.027 (2)	0.009 (2)
O2	0.124 (3)	0.0604 (17)	0.0677 (19)	-0.0108 (17)	0.0448 (18)	0.0181 (15)
C2	0.049 (2)	0.0464 (19)	0.050 (2)	-0.0043 (16)	0.0047 (17)	0.0156 (17)
O3	0.085 (2)	0.0465 (14)	0.0549 (16)	0.0031 (13)	0.0257 (14)	0.0166 (12)
C3	0.089 (3)	0.057 (2)	0.072 (3)	-0.002 (2)	0.027 (2)	0.025 (2)
O4	0.105 (2)	0.0593 (17)	0.0644 (18)	0.0145 (16)	0.0411 (17)	0.0209 (14)
C4	0.049 (2)	0.049 (2)	0.048 (2)	0.0061 (16)	0.0103 (17)	0.0131 (17)
C5	0.0417 (19)	0.0443 (18)	0.0420 (19)	0.0034 (15)	0.0021 (15)	0.0139 (15)
O5	0.134 (3)	0.0523 (17)	0.107 (3)	0.0219 (18)	0.057 (2)	0.0198 (17)
C6	0.050 (2)	0.0473 (19)	0.0437 (19)	0.0042 (16)	0.0029 (16)	0.0145 (16)
O6	0.131 (3)	0.0527 (17)	0.089 (2)	-0.0210 (18)	0.036 (2)	0.0148 (16)
C7	0.0388 (19)	0.0405 (17)	0.054 (2)	0.0001 (15)	-0.0011 (16)	0.0137 (16)
C8	0.045 (2)	0.0507 (19)	0.0432 (19)	-0.0005 (16)	0.0012 (16)	0.0174 (16)
C9	0.047 (2)	0.0464 (19)	0.0412 (18)	0.0013 (16)	0.0028 (16)	0.0127 (15)
C10	0.044 (2)	0.0449 (18)	0.046 (2)	-0.0032 (15)	0.0042 (16)	0.0130 (16)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

N—O6	1.194 (4)	C3—H3B	0.9600
N—O5	1.204 (4)	C3—H3C	0.9600
N—C7	1.475 (4)	O4—C4	1.198 (4)
O1—C2	1.336 (4)	C4—C5	1.485 (5)
O1—C1	1.433 (4)	C5—C10	1.381 (4)
C1—H1A	0.9600	C5—C6	1.391 (5)
C1—H1B	0.9600	C6—C7	1.378 (5)

## supplementary materials

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C1—H1C	0.9600	C6—H6A	0.9300
O2—C2	1.193 (4)	C7—C8	1.366 (5)
C2—C9	1.475 (5)	C8—C9	1.392 (5)
O3—C4	1.320 (4)	C8—H8A	0.9300
O3—C3	1.438 (4)	C9—C10	1.381 (5)
C3—H3A	0.9600	C10—H10A	0.9300
O6—N—O5	122.5 (3)	O4—C4—C5	123.3 (3)
O6—N—C7	118.6 (3)	O3—C4—C5	112.4 (3)
O5—N—C7	118.9 (3)	C10—C5—C6	119.7 (3)
C2—O1—C1	116.9 (3)	C10—C5—C4	122.3 (3)
O1—C1—H1A	109.5	C6—C5—C4	117.9 (3)
O1—C1—H1B	109.5	C7—C6—C5	117.8 (3)
H1A—C1—H1B	109.5	C7—C6—H6A	121.1
O1—C1—H1C	109.5	C5—C6—H6A	121.1
H1A—C1—H1C	109.5	C8—C7—C6	123.1 (3)
H1B—C1—H1C	109.5	C8—C7—N	118.7 (3)
O2—C2—O1	122.5 (3)	C6—C7—N	118.2 (3)
O2—C2—C9	124.5 (3)	C7—C8—C9	119.0 (3)
O1—C2—C9	112.9 (3)	C7—C8—H8A	120.5
C4—O3—C3	116.9 (3)	C9—C8—H8A	120.5
O3—C3—H3A	109.5	C10—C9—C8	118.7 (3)
O3—C3—H3B	109.5	C10—C9—C2	122.6 (3)
H3A—C3—H3B	109.5	C8—C9—C2	118.7 (3)
O3—C3—H3C	109.5	C5—C10—C9	121.6 (3)
H3A—C3—H3C	109.5	C5—C10—H10A	119.2
H3B—C3—H3C	109.5	C9—C10—H10A	119.2
O4—C4—O3	124.3 (3)		
C1—O1—C2—O2	-0.4 (6)	O6—N—C7—C6	168.8 (4)
C1—O1—C2—C9	178.7 (3)	O5—N—C7—C6	-10.6 (5)
C3—O3—C4—O4	-0.1 (6)	C6—C7—C8—C9	0.2 (5)
C3—O3—C4—C5	179.7 (3)	N—C7—C8—C9	-179.7 (3)
O4—C4—C5—C10	-179.8 (4)	C7—C8—C9—C10	-0.6 (5)
O3—C4—C5—C10	0.4 (5)	C7—C8—C9—C2	178.6 (3)
O4—C4—C5—C6	-2.1 (5)	O2—C2—C9—C10	177.7 (4)
O3—C4—C5—C6	178.1 (3)	O1—C2—C9—C10	-1.4 (5)
C10—C5—C6—C7	-1.5 (5)	O2—C2—C9—C8	-1.4 (6)
C4—C5—C6—C7	-179.3 (3)	O1—C2—C9—C8	179.5 (3)
C5—C6—C7—C8	0.8 (5)	C6—C5—C10—C9	1.2 (5)
C5—C6—C7—N	-179.2 (3)	C4—C5—C10—C9	178.9 (3)
O6—N—C7—C8	-11.2 (5)	C8—C9—C10—C5	-0.1 (5)
O5—N—C7—C8	169.4 (4)	C2—C9—C10—C5	-179.3 (3)

Fig. 1

