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Dimethyl 5-nitroisophthalate

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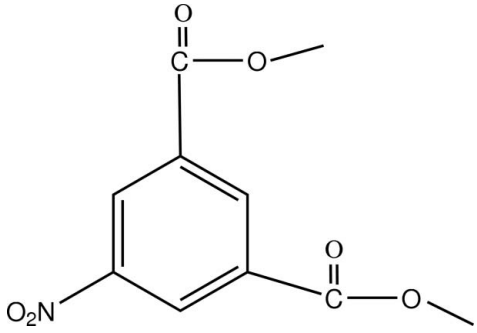
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.073; wR factor = 0.172; data-to-parameter ratio = 12.2.

The nitro group in the title compound, $C_{10}H_9NO_6$, 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

$C_{10}H_9NO_6$
M_r = 239.18
Triclinic, $P\overline{1}$
a = 4.0130 (8) Å
b = 10.660 (2) Å
c = 12.643 (3) Å
α = 106.11 (3)°
β = 93.74 (3)°
γ = 91.46 (3)°
V = 517.97 (18) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 293 (2) K
0.40 × 0.05 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968))
T_{min} = 0.950, T_{max} = 0.994
2178 measured reflections
1885 independent reflections
1144 reflections with $I > 2\sigma(I)$
R_{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 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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).

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Dimethyl 5-nitroisophthalate

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S1. 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)°.

S2. Experimental

5-nitroisophthalic 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%).

S3. 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_{iso}(H) = 1.2U_{eq}(C) or 0.96 (methyl) and U_{iso}(H) = 1.5U_{eq}(C), respectively.

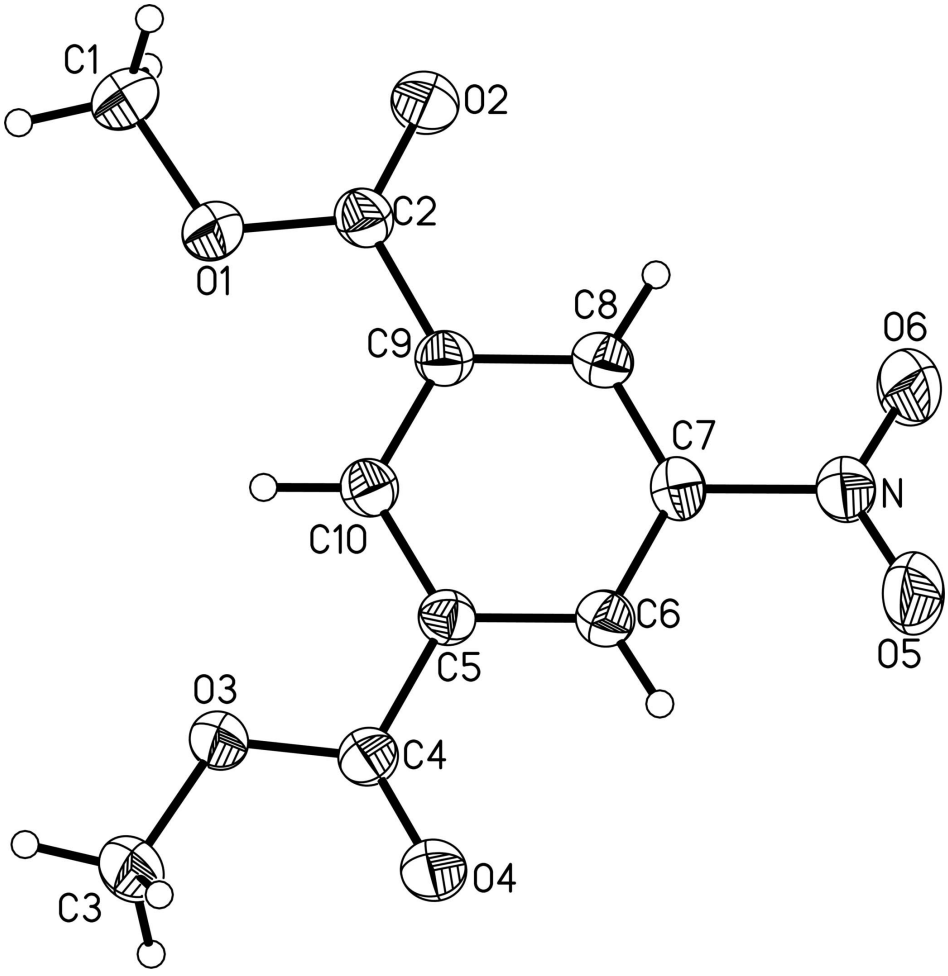

Figure 1

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

$C_{10}H_9NO_6$
$M_r = 239.18$
Triclinic, $P\overline{1}$
Hall symbol: -P 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^3
$Z = 2$
$F(000) = 248$
$D_x = 1.534$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8–12°$
$\mu = 0.13$ mm^{-1}
$T = 293$ K
Needle, colourless
0.40 × 0.05 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968))
T_{min} = 0.950, T_{max} = 0.994
2178 measured reflections
1885 independent reflections
1144 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 25.3°, θ_{min} = 1.7°
$h = -4\rightarrow4$
$k = -12\rightarrow12$
$l = 0\rightarrow15$
3 standard reflections every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.172
S = 1.01
1885 reflections
154 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 0.6P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{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 (Å²)

	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 (Å, º)

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)
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)