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# Isobutyl 3,5-dinitrobenzoate

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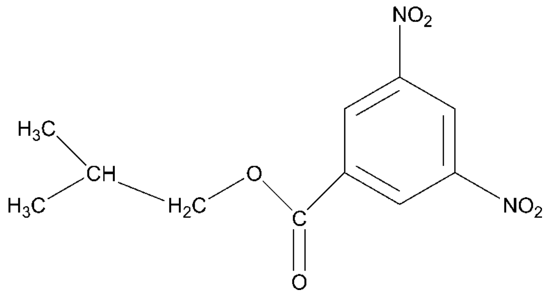
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.006 Å; $R$ factor = 0.069; $wR$ factor = 0.225; data-to-parameter ratio = 13.2.

In the structure of the title compound, $C_{11}H_{12}N_2O_6$, the molecules are stacked along the $b$ axis without any $\pi$–$\pi$ interactions. The stacked columns are linked together by non-classical intermolecular $C—H\cdots O$ interactions,. In the molecule, the nitro groups make dihedral angles of 9.4 (5) and 10.3 (5)° with the benzene ring.

## Related literature

For the properties and applications of dinitrobenzoate derivatives, see: Huang *et al.* (2004); Kagitani *et al.* (1984); Olive (1979). For the anti-creatinine effects of a series of 3,5-dinitrobenzoic acid esters, see: Yu & Yang (2002). For bond-length data, see: Allen *et al.* (1987).



## Experimental

### *Crystal data*

$C_{11}H_{12}N_2O_6$
$M_r$ = 268.23
Monoclinic, $P2_1/n$
$a$ = 16.666 (3) Å
$b$ = 4.776 (1) Å
$c$ = 16.678 (3) Å
$\beta$ = 110.30 (3)°
$V$ = 1245.1 (5) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.12 mm$^{-1}$
$T$ = 293 K
0.30 × 0.20 × 0.10 mm

### *Data collection*

Enraf–Nonius CAD-4 diffractometer
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.965, $T_{max}$ = 0.988
2348 measured reflections
2266 independent reflections
1402 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.061
3 standard reflections every 200 reflections intensity decay: 1%

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.225
$S$ = 1.11
2266 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.30 e Å$^{-3}$

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

| $D—H\cdots A$ | $D—H$ | $H\cdots A$ | $D\cdots A$ | $D—H\cdots A$ |
|---|---|---|---|---|
| C7—H7$A\cdots$O2$^i$ | 0.93 | 2.52 | 3.441 (5) | 168 |

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

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: *SHELXTL* (Sheldrick, 2008); 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: PV2148).

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# supporting information

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## Isobutyl 3,5-dinitrobenzoate

**Pei Zou, Min-Hao Xie, Shi-Neng Luo, Ya-Ling Liu and Yong-Jun He**

### S1. Comment

Due to their biological activities, dinitrobenzoate derivatives are widely used in pharmacology. Dinitrobenzoic acid derivatives are effective in tumour treatment as radiation sensitizers (Kagitani *et al.*, 1984). Moreover, some synthetic dinitrobenzoate compounds have shown useful properties in DNA and oligosaccharide synthesis (Olive, 1979; Huang *et al.*, 2004). Furthermore, a series of 3,5-dinitrobenzoic acid esters has also been synthesized and their anti-creatinine effects have been studied (Yu & Yang, 2002). To study their structures and activities, we report here the crystal structure of the title compound, (I).

The bond lengths and angles in (I) (Fig. 1) are within expected ranges (Allen *et al.*, 1987). The two nitro groups are inclined by 9.4 (5) and 169.7 (5)° to the benzene ring, respectively. Except for atoms C1 and C2, the other non-H atoms of the molecule lie in a plane. In the crystal structure, the molecules are stacked along the *b* axis, without any $\pi$-$\pi$ interaction. The stacked columns are linked together by non-classical intermolecular interactions of the type C—H···O, details have been given in Table 1.

### S2. Experimental

3,5-Dinitrobenzoylchloride (5200 mg, 23 mmol) was added in iso-butanol (25 ml, 271 mmol) and the mixture was refluxed for 4 h. White product appeared after cooling to room temperature. They were separated and washed with cold water. Single crystals of the title compound were grown by slow evaporation of a methanol solution: colourless needle-shaped crystals were formed after several days.

### 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 distances of 0.93 Å(aromatic), 0.98 Å(CH), 0.97 Å($CH_2$) and 0.96 Å($CH_3$); $U_{iso}$(H) = 1.2$U_{eq}$(C) for the aromatic H, CH and $CH_2$; $U_{iso}$(H) = 1.5$U_{eq}$(C) for $CH_3$.

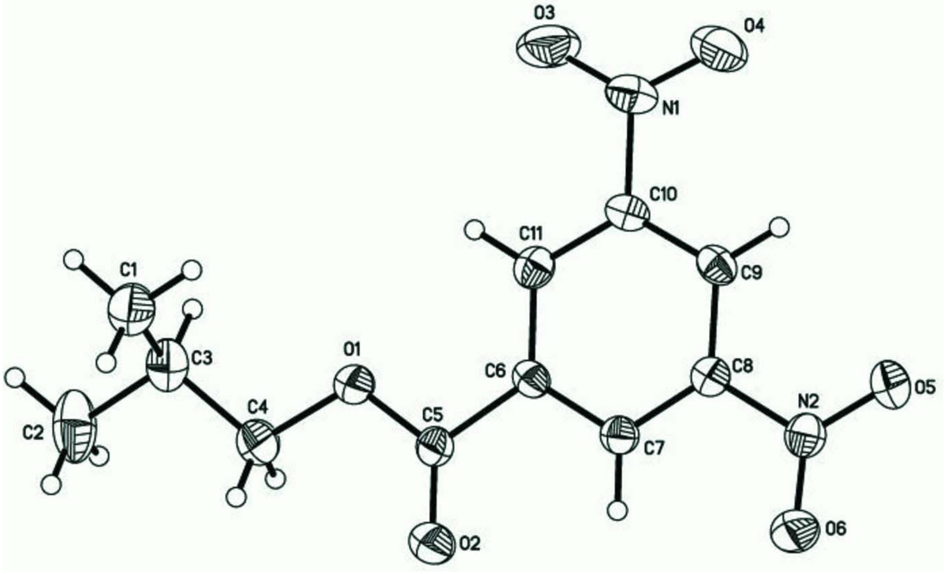

**Figure 1**

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

### Isobutyl 3,5-dinitrobenzoate

*Crystal data*

$C_{11}H_{12}N_2O_6$
$M_r = 268.23$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 16.666$ (3) Å
$b = 4.776$ (1) Å
$c = 16.678$ (3) Å
$\beta = 110.30$ (3)°
$V = 1245.1$ (5) Å$^3$
$Z = 4$

$F(000) = 560$
$D_x = 1.431$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–12°
$\mu = 0.12$ mm$^{-1}$
$T = 293$ K
Needle, colourless
0.30 × 0.20 × 0.10 mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{\min} = 0.965$, $T_{\max} = 0.988$
2348 measured reflections

2266 independent reflections
1402 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.061$
$\theta_{\max} = 25.3°$, $\theta_{\min} = 1.5°$
$h = 0\rightarrow20$
$k = 0\rightarrow5$
$l = -20\rightarrow18$
3 standard reflections every 200 reflections
intensity decay: 1%

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.069$
$wR(F^2) = 0.225$
$S = 1.11$

2266 reflections
172 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

$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.25$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.30$ e Å$^{-3}$

*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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{\rm iso}$*/$U_{\rm eq}$ |
|---|---|---|---|---|
| O1 | −0.17180 (16) | −0.2811 (6) | 0.58690 (17) | 0.0567 (8) |
| O2 | −0.08913 (17) | −0.4218 (7) | 0.51387 (18) | 0.0616 (8) |
| O3 | −0.0648 (2) | 0.3897 (9) | 0.8166 (2) | 0.0933 (13) |
| O4 | 0.0522 (2) | 0.6102 (7) | 0.8340 (2) | 0.0767 (10) |
| O5 | 0.23983 (17) | 0.2979 (6) | 0.68814 (18) | 0.0603 (8) |
| O6 | 0.20462 (18) | −0.0604 (6) | 0.60527 (19) | 0.0637 (9) |
| N1 | −0.0004 (2) | 0.4291 (8) | 0.8000 (2) | 0.0542 (9) |
| N2 | 0.18975 (19) | 0.1116 (7) | 0.6515 (2) | 0.0463 (8) |
| C1 | −0.2813 (3) | −0.6160 (11) | 0.6546 (3) | 0.0745 (14) |
| H1A | −0.2346 | −0.5124 | 0.6936 | 0.112* |
| H1B | −0.2629 | −0.8031 | 0.6491 | 0.112* |
| H1C | −0.3278 | −0.6224 | 0.6761 | 0.112* |
| C2 | −0.3856 (3) | −0.6279 (12) | 0.5060 (3) | 0.0864 (17) |
| H2A | −0.4308 | −0.6444 | 0.5289 | 0.130* |
| H2B | −0.3680 | −0.8112 | 0.4951 | 0.130* |
| H2C | −0.4055 | −0.5237 | 0.4535 | 0.130* |
| C3 | −0.3102 (3) | −0.4769 (9) | 0.5697 (3) | 0.0593 (11) |
| H3A | −0.3289 | −0.2870 | 0.5771 | 0.071* |
| C4 | −0.2420 (3) | −0.4536 (10) | 0.5323 (3) | 0.0645 (12) |
| H4A | −0.2208 | −0.6386 | 0.5264 | 0.077* |
| H4B | −0.2651 | −0.3702 | 0.4759 | 0.077* |
| C5 | −0.1000 (2) | −0.2845 (8) | 0.5687 (2) | 0.0418 (8) |
| C6 | −0.0337 (2) | −0.0901 (7) | 0.6262 (2) | 0.0363 (8) |
| C7 | 0.0447 (2) | −0.0816 (7) | 0.6143 (2) | 0.0388 (8) |
| H7A | 0.0553 | −0.1948 | 0.5736 | 0.047* |
| C8 | 0.1063 (2) | 0.0988 (7) | 0.6641 (2) | 0.0381 (8) |
| C9 | 0.0934 (2) | 0.2683 (7) | 0.7252 (2) | 0.0406 (8) |
| H9A | 0.1356 | 0.3901 | 0.7578 | 0.049* |
| C10 | 0.0157 (2) | 0.2503 (7) | 0.7359 (2) | 0.0407 (8) |
| C11 | −0.0482 (2) | 0.0730 (8) | 0.6876 (2) | 0.0430 (9) |

| | | | | |
|---|---|---|---|---|
| H11A | −0.1002 | 0.0641 | 0.6965 | 0.052* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0495 (15) | 0.0679 (19) | 0.0601 (17) | −0.0169 (14) | 0.0283 (13) | −0.0232 (14) |
| O2 | 0.0559 (17) | 0.070 (2) | 0.0611 (17) | −0.0124 (15) | 0.0226 (14) | −0.0231 (16) |
| O3 | 0.072 (2) | 0.123 (3) | 0.096 (3) | 0.002 (2) | 0.043 (2) | −0.047 (2) |
| O4 | 0.093 (2) | 0.066 (2) | 0.070 (2) | −0.0068 (19) | 0.0260 (18) | −0.0222 (18) |
| O5 | 0.0550 (16) | 0.0615 (19) | 0.0638 (18) | −0.0203 (15) | 0.0200 (14) | −0.0124 (15) |
| O6 | 0.0572 (17) | 0.067 (2) | 0.075 (2) | −0.0002 (15) | 0.0328 (15) | −0.0174 (17) |
| N1 | 0.056 (2) | 0.053 (2) | 0.0485 (19) | 0.0094 (18) | 0.0128 (16) | −0.0119 (17) |
| N2 | 0.0420 (17) | 0.048 (2) | 0.0478 (18) | 0.0002 (15) | 0.0143 (14) | 0.0031 (16) |
| C1 | 0.073 (3) | 0.085 (4) | 0.069 (3) | −0.020 (3) | 0.029 (2) | −0.008 (3) |
| C2 | 0.051 (3) | 0.100 (4) | 0.090 (4) | −0.023 (3) | 0.001 (2) | 0.023 (3) |
| C3 | 0.052 (2) | 0.054 (3) | 0.073 (3) | −0.004 (2) | 0.022 (2) | 0.006 (2) |
| C4 | 0.056 (2) | 0.081 (3) | 0.057 (2) | −0.024 (2) | 0.020 (2) | −0.020 (2) |
| C5 | 0.048 (2) | 0.040 (2) | 0.0394 (19) | −0.0065 (17) | 0.0181 (16) | 0.0011 (17) |
| C6 | 0.0414 (18) | 0.0324 (18) | 0.0320 (17) | −0.0024 (15) | 0.0088 (14) | −0.0001 (15) |
| C7 | 0.0446 (19) | 0.0347 (18) | 0.0377 (18) | 0.0001 (16) | 0.0151 (15) | 0.0015 (15) |
| C8 | 0.0384 (18) | 0.0371 (19) | 0.0378 (18) | 0.0030 (15) | 0.0118 (15) | 0.0040 (15) |
| C9 | 0.047 (2) | 0.0325 (19) | 0.0378 (18) | −0.0016 (16) | 0.0091 (15) | −0.0001 (15) |
| C10 | 0.0456 (19) | 0.038 (2) | 0.0352 (18) | 0.0048 (16) | 0.0095 (15) | −0.0036 (15) |
| C11 | 0.0422 (19) | 0.048 (2) | 0.0387 (18) | 0.0011 (17) | 0.0134 (16) | 0.0060 (17) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C5 | 1.332 (4) | C2—H2C | 0.9600 |
| O1—C4 | 1.462 (5) | C3—C4 | 1.479 (5) |
| O2—C5 | 1.190 (4) | C3—H3A | 0.9800 |
| O3—N1 | 1.213 (4) | C4—H4A | 0.9700 |
| O4—N1 | 1.222 (4) | C4—H4B | 0.9700 |
| O5—N2 | 1.228 (4) | C5—C6 | 1.505 (5) |
| O6—N2 | 1.210 (4) | C6—C11 | 1.373 (5) |
| N1—C10 | 1.463 (5) | C6—C7 | 1.390 (5) |
| N2—C8 | 1.478 (4) | C7—C8 | 1.377 (5) |
| C1—C3 | 1.485 (6) | C7—H7A | 0.9300 |
| C1—H1A | 0.9600 | C8—C9 | 1.375 (5) |
| C1—H1B | 0.9600 | C9—C10 | 1.369 (5) |
| C1—H1C | 0.9600 | C9—H9A | 0.9300 |
| C2—C3 | 1.517 (6) | C10—C11 | 1.381 (5) |
| C2—H2A | 0.9600 | C11—H11A | 0.9300 |
| C2—H2B | 0.9600 | | |
| | | | |
| C5—O1—C4 | 116.0 (3) | O1—C4—H4A | 109.6 |
| O3—N1—O4 | 123.5 (4) | C3—C4—H4A | 109.6 |
| O3—N1—C10 | 118.5 (4) | O1—C4—H4B | 109.6 |
| O4—N1—C10 | 117.9 (3) | C3—C4—H4B | 109.6 |

| | |
|---|---|
| O6—N2—O5 | 123.7 (3) |
| O6—N2—C8 | 118.3 (3) |
| O5—N2—C8 | 118.0 (3) |
| C3—C1—H1A | 109.5 |
| C3—C1—H1B | 109.5 |
| H1A—C1—H1B | 109.5 |
| C3—C1—H1C | 109.5 |
| H1A—C1—H1C | 109.5 |
| H1B—C1—H1C | 109.5 |
| C3—C2—H2A | 109.5 |
| C3—C2—H2B | 109.5 |
| H2A—C2—H2B | 109.5 |
| C3—C2—H2C | 109.5 |
| H2A—C2—H2C | 109.5 |
| H2B—C2—H2C | 109.5 |
| C4—C3—C1 | 113.2 (4) |
| C4—C3—C2 | 108.1 (4) |
| C1—C3—C2 | 111.8 (4) |
| C4—C3—H3A | 107.9 |
| C1—C3—H3A | 107.9 |
| C2—C3—H3A | 107.9 |
| O1—C4—C3 | 110.2 (3) |
| H4A—C4—H4B | 108.1 |
| O2—C5—O1 | 124.8 (3) |
| O2—C5—C6 | 123.7 (3) |
| O1—C5—C6 | 111.5 (3) |
| C11—C6—C7 | 120.5 (3) |
| C11—C6—C5 | 123.0 (3) |
| C7—C6—C5 | 116.5 (3) |
| C8—C7—C6 | 118.2 (3) |
| C8—C7—H7A | 120.9 |
| C6—C7—H7A | 120.9 |
| C7—C8—C9 | 122.7 (3) |
| C7—C8—N2 | 118.8 (3) |
| C9—C8—N2 | 118.6 (3) |
| C10—C9—C8 | 117.3 (3) |
| C10—C9—H9A | 121.4 |
| C8—C9—H9A | 121.4 |
| C9—C10—C11 | 122.3 (3) |
| C9—C10—N1 | 118.7 (3) |
| C11—C10—N1 | 119.0 (3) |
| C6—C11—C10 | 118.9 (3) |
| C6—C11—H11A | 120.5 |
| C10—C11—H11A | 120.5 |

| | |
|---|---|
| C5—O1—C4—C3 | 168.4 (4) |
| C1—C3—C4—O1 | −62.2 (5) |
| C2—C3—C4—O1 | 173.4 (4) |
| C4—O1—C5—O2 | −2.2 (6) |
| C4—O1—C5—C6 | 177.4 (3) |
| O2—C5—C6—C11 | 178.1 (4) |
| O1—C5—C6—C11 | −1.5 (5) |
| O2—C5—C6—C7 | −1.8 (5) |
| O1—C5—C6—C7 | 178.6 (3) |
| C11—C6—C7—C8 | −1.5 (5) |
| C5—C6—C7—C8 | 178.4 (3) |
| C6—C7—C8—C9 | 0.4 (5) |
| C6—C7—C8—N2 | −179.5 (3) |
| O6—N2—C8—C7 | −8.8 (5) |
| O5—N2—C8—C7 | 170.7 (3) |
| O6—N2—C8—C9 | 171.3 (3) |
| O5—N2—C8—C9 | −9.2 (5) |
| C7—C8—C9—C10 | 0.6 (5) |
| N2—C8—C9—C10 | −179.5 (3) |
| C8—C9—C10—C11 | −0.6 (5) |
| C8—C9—C10—N1 | −179.6 (3) |
| O3—N1—C10—C9 | −171.3 (4) |
| O4—N1—C10—C9 | 8.5 (5) |
| O3—N1—C10—C11 | 9.7 (5) |
| O4—N1—C10—C11 | −170.5 (3) |
| C7—C6—C11—C10 | 1.5 (5) |
| C5—C6—C11—C10 | −178.4 (3) |
| C9—C10—C11—C6 | −0.4 (5) |
| N1—C10—C11—C6 | 178.5 (3) |

### *Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C7—H7*A*···O2$^{\text{i}}$ | 0.93 | 2.52 | 3.441 (5) | 168 |

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