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# 4-Nitrophenyl methacrylate

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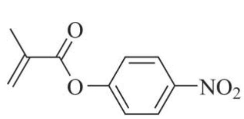
Key indicators: single-crystal X-ray study; $T$ = 90 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.054; $wR$ factor = 0.146; data-to-parameter ratio = 16.0.

The title compound, $C_{10}H_9NO_4$, was obtained serendipitously during the preparation of benzyl cyclohexylcarbamate. The molecule consists of two approximately planar parts, the nitrophenyl ring and the rest of the non-H atoms, with a dihedral angle of 55.05 (6)° between the two segments. The crystal structure is stabilized by weak C—H···O interactions and $\pi$ stacking [3.753 (1) Å] along the $b$ axis.

## Related literature

For related literature, see: Banks *et al.* (1977); Hwang *et al.* (2007); Li *et al.* (2007); Otsu *et al.* (1968); Tang *et al.* (2007).



## Experimental

*Crystal data*

$C_{10}H_9NO_4$
$M_r$ = 207.18
Monoclinic, $C2/c$
$a$ = 24.491 (6) Å
$b$ = 3.753 (1) Å
$c$ = 23.428 (6) Å
$\beta$ = 116.98 (1)°
$V$ = 1919.0 (9) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.11 mm$^{-1}$
$T$ = 90.0 (2) K
0.30 × 0.10 × 0.04 mm

*Data collection*

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min}$ = 0.967, $T_{max}$ = 0.996
3936 measured reflections
2193 independent reflections
1380 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.049

*Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.145
$S$ = 1.04
2193 reflections
137 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.28 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C7—H7···O1$^{i}$ | 0.95 | 2.41 | 3.130 (2) | 133 |
| C1—H1$B$···O4$^{ii}$ | 0.95 | 2.64 | 3.546 (3) | 159 |
| C2—H2$A$···O3$^{ii}$ | 0.98 | 2.68 | 3.611 (2) | 159 |
| C9—H9···O4$^{iii}$ | 0.95 | 2.46 | 3.282 (2) | 145 |

Symmetry codes: (i) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x+\frac{1}{2}, y-\frac{1}{2}, z$; (iii) $-x, -y+2, -z$.

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* and local procedures.

YX and FQ thank Dr Sihui Long for helpful discussions and invaluable suggestions.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2212).

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# supplementary materials

*Acta Cryst.* (2008). E**64**, o1751 [ **doi:10.1107/S1600536808025130** ]

## 4-Nitrophenyl methacrylate

**Y.-H. Xu and F. Qu**

### Comment

The title compound (I) is an important building block in the preparation of functional block polymers (Tang, *et al.* 2007; Hwang, *et al.* 2007; Li, *et al.* 2007). Although it has been widely used as a monomer in polymerization reactions for a long time (Otsu, *et al.* 1968), the crystal structure, as far as we know, has never been reported before.

Traditonally, (I) has been synthesized by refluxing methacryloyl chloride and *para*-nitrophenol (Banks, *et al.* 1977). Here it was obtained unexpectedly during an attempt to make benzyl cyclohexylcarbamate as described in the experimental section.

The asymmetric unit of (I) (Fig. 1) contains one molecule and bond lengths and angles are within normal ranges. The molecule consists of two approximately planar parts: the nitrophenyl ring and the rest of the non-hydrogen atoms (dihedral angle between the two segments is 55.05 (6)°). The nitro group is nearly coplanar with the phenyl ring as indicated by the torsion angle O3-N1-C8-C7 of -7.48 °. The remaining non-hydrogen atoms are almost coplanar as suggested by the torsion angle C2-C3-C4-O1 at 9.35 °. Since (I) has no classic hydrogen bonding donors, the crystal packng is stabilized by C—H···O interactions (Table 1)in two directions with aromatic C-H atoms as the donors and both oxygen atoms of the nitro group and the carbonyl oxygen as the acceptors. There is also π-stacking along the third direction, the shortest (*b*), where the aromatic rings are separated by a unit cell translation of 3.753 (1) Å (Fig. 2).

### Experimental

4-nitrophenyl cyclohexylcarbamate (0.95 g, 3.5 mmol), phenylmethanol (0.40 g, 3.7 mmol) and triethylamine (0.36 g, 3.6 mmol) were reflxued overnight in 20 ml methylene chloride. The solution was washed with 1 N NaOH, water and brine, and then dried with anhydrous $Na_2SO_4$. After removal of the solvent, the product was recovered as a colorless solid (0.5 g). Crystals of (I) were obtained by recrystallization from ethyl acetate as colorless rods.

### Refinement

H atoms were found in difference Fourier maps and subsequently placed in idealized positions with constrained C—H distances of 0.95 Å ($C_{Ar}$H) and 0.98 Å (Csp3H). $U_{iso}$(H) values were set to 1.2$U_{eq}$ for all H atoms.

### Figures

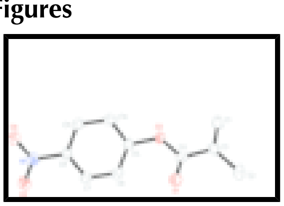

Fig. 1. The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level (arbitrary spheres for the H atoms).

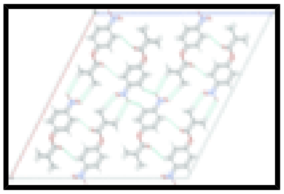

Fig. 2. A packing diagram of (I) shown looking down the *b* axis.

### 4-Nitrophenyl methacrylate

*Crystal data*

| | |
|---|---|
| $C_{10}H_9NO_4$ | $F_{000}$ = 864 |
| $M_r$ = 207.18 | $D_x$ = 1.434 Mg m$^{-3}$ |
| Monoclinic, *C*2/*c* | Mo *K*α radiation<br>λ = 0.71073 Å |
| *a* = 24.491 (6) Å | Cell parameters from 2523 reflections |
| *b* = 3.753 (1) Å | θ = 1.0–27.5° |
| *c* = 23.428 (6) Å | μ = 0.11 mm$^{-1}$ |
| β = 116.98 (1)° | *T* = 90.0 (2) K |
| *V* = 1919.0 (9) Å$^3$ | Thin rod, colorless |
| *Z* = 8 | 0.30 × 0.10 × 0.04 mm |

*Data collection*

| | |
|---|---|
| Nonius KappaCCD<br>diffractometer | 2193 independent reflections |
| Radiation source: fine-focus sealed tube | 1380 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.049 |
| Detector resolution: 18 pixels mm$^{-1}$ | $\theta_{max}$ = 27.5° |
| *T* = 90.0(2) K | $\theta_{min}$ = 1.9° |
| ω scans at fixed χ = 55° | $h = -31 \rightarrow 31$ |
| Absorption correction: multi-scan<br>(SCALEPACK; Otwinowski & Minor, 1997) | $k = -4 \rightarrow 4$ |
| $T_{min}$ = 0.967, $T_{max}$ = 0.996 | $l = -30 \rightarrow 29$ |
| 3936 measured reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)]$ = 0.053 | H-atom parameters constrained |
| $wR(F^2)$ = 0.145 | $w = 1/[\sigma^2(F_o^2) + (0.0788P)^2 + 0.0268P]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| *S* = 1.04 | $(\Delta/\sigma)_{max}$ < 0.001 |
| 2193 reflections | $\Delta\rho_{max}$ = 0.32 e Å$^{-3}$ |
| 137 parameters | $\Delta\rho_{min}$ = −0.28 e Å$^{-3}$ |

Primary atom site location: structure-invariant direct methods

Extinction correction: none

*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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.34238 (9) | 0.3043 (6) | 0.08125 (9) | 0.0350 (5) |
| H1A | 0.3022 | 0.2636 | 0.0481 | 0.042* |
| H1B | 0.3769 | 0.2388 | 0.0753 | 0.042* |
| C2 | 0.41131 (8) | 0.5324 (6) | 0.19114 (9) | 0.0327 (5) |
| H2A | 0.4437 | 0.4551 | 0.1803 | 0.049* |
| H2B | 0.4153 | 0.4042 | 0.2293 | 0.049* |
| H2C | 0.4150 | 0.7891 | 0.1998 | 0.049* |
| C3 | 0.35044 (8) | 0.4545 (6) | 0.13665 (9) | 0.0258 (5) |
| C4 | 0.29729 (8) | 0.5595 (5) | 0.14726 (9) | 0.0247 (5) |
| C5 | 0.18934 (8) | 0.4978 (5) | 0.10638 (9) | 0.0232 (5) |
| C6 | 0.18535 (8) | 0.3892 (5) | 0.16081 (8) | 0.0248 (5) |
| H6 | 0.2196 | 0.2858 | 0.1958 | 0.030* |
| C7 | 0.13046 (8) | 0.4341 (5) | 0.16321 (9) | 0.0249 (5) |
| H7 | 0.1262 | 0.3607 | 0.1998 | 0.030* |
| C8 | 0.08165 (8) | 0.5879 (5) | 0.11143 (8) | 0.0225 (5) |
| C9 | 0.08549 (8) | 0.6948 (5) | 0.05692 (8) | 0.0239 (5) |
| H9 | 0.0511 | 0.7963 | 0.0218 | 0.029* |
| C10 | 0.14063 (8) | 0.6505 (5) | 0.05472 (8) | 0.0243 (5) |
| H10 | 0.1449 | 0.7242 | 0.0182 | 0.029* |
| N1 | 0.02377 (7) | 0.6382 (5) | 0.11468 (7) | 0.0273 (4) |
| O1 | 0.30028 (5) | 0.7338 (4) | 0.19146 (6) | 0.0309 (4) |
| O2 | 0.24257 (5) | 0.4394 (4) | 0.09952 (6) | 0.0265 (4) |
| O3 | 0.01867 (6) | 0.5094 (4) | 0.16001 (7) | 0.0379 (4) |
| O4 | −0.01732 (6) | 0.8076 (4) | 0.07152 (6) | 0.0356 (4) |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0292 (11) | 0.0418 (15) | 0.0387 (11) | −0.0006 (11) | 0.0195 (9) | −0.0047 (11) |
| C2 | 0.0274 (11) | 0.0333 (14) | 0.0390 (12) | 0.0015 (10) | 0.0165 (10) | −0.0001 (10) |
| C3 | 0.0258 (10) | 0.0259 (12) | 0.0286 (11) | −0.0004 (9) | 0.0148 (9) | 0.0030 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C4 | 0.0239 (10) | 0.0250 (12) | 0.0228 (10) | 0.0004 (9) | 0.0084 (8) | 0.0007 (9) |
| C5 | 0.0217 (10) | 0.0231 (12) | 0.0272 (10) | −0.0010 (9) | 0.0133 (8) | −0.0051 (9) |
| C6 | 0.0215 (10) | 0.0237 (12) | 0.0246 (10) | −0.0001 (9) | 0.0066 (8) | −0.0014 (9) |
| C7 | 0.0258 (10) | 0.0254 (12) | 0.0238 (10) | −0.0008 (9) | 0.0116 (8) | −0.0005 (8) |
| C8 | 0.0199 (9) | 0.0233 (12) | 0.0251 (10) | −0.0017 (9) | 0.0107 (8) | −0.0032 (9) |
| C9 | 0.0230 (10) | 0.0233 (12) | 0.0219 (9) | 0.0006 (9) | 0.0070 (8) | −0.0013 (9) |
| C10 | 0.0269 (10) | 0.0248 (12) | 0.0204 (9) | 0.0004 (9) | 0.0102 (8) | −0.0007 (9) |
| N1 | 0.0238 (9) | 0.0312 (11) | 0.0268 (9) | 0.0008 (8) | 0.0113 (7) | −0.0005 (8) |
| O1 | 0.0260 (7) | 0.0375 (10) | 0.0295 (7) | −0.0029 (7) | 0.0129 (6) | −0.0088 (7) |
| O2 | 0.0201 (7) | 0.0343 (8) | 0.0258 (7) | −0.0005 (6) | 0.0111 (6) | −0.0043 (6) |
| O3 | 0.0317 (8) | 0.0518 (11) | 0.0365 (8) | 0.0040 (7) | 0.0210 (7) | 0.0094 (7) |
| O4 | 0.0251 (7) | 0.0489 (11) | 0.0319 (7) | 0.0098 (7) | 0.0121 (6) | 0.0059 (7) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—C3 | 1.345 (3) | C5—O2 | 1.402 (2) |
| C1—H1A | 0.9500 | C6—C7 | 1.381 (3) |
| C1—H1B | 0.9500 | C6—H6 | 0.9500 |
| C2—C3 | 1.487 (2) | C7—C8 | 1.385 (3) |
| C2—H2A | 0.9800 | C7—H7 | 0.9500 |
| C2—H2B | 0.9800 | C8—C9 | 1.382 (2) |
| C2—H2C | 0.9800 | C8—N1 | 1.466 (2) |
| C3—C4 | 1.485 (3) | C9—C10 | 1.385 (3) |
| C4—O1 | 1.199 (2) | C9—H9 | 0.9500 |
| C4—O2 | 1.375 (2) | C10—H10 | 0.9500 |
| C5—C10 | 1.381 (3) | N1—O3 | 1.224 (2) |
| C5—C6 | 1.384 (3) | N1—O4 | 1.231 (2) |
| C3—C1—H1A | 120.0 | C7—C6—H6 | 120.7 |
| C3—C1—H1B | 120.0 | C5—C6—H6 | 120.7 |
| H1A—C1—H1B | 120.0 | C6—C7—C8 | 119.00 (17) |
| C3—C2—H2A | 109.5 | C6—C7—H7 | 120.5 |
| C3—C2—H2B | 109.5 | C8—C7—H7 | 120.5 |
| H2A—C2—H2B | 109.5 | C9—C8—C7 | 122.47 (17) |
| C3—C2—H2C | 109.5 | C9—C8—N1 | 118.91 (16) |
| H2A—C2—H2C | 109.5 | C7—C8—N1 | 118.62 (16) |
| H2B—C2—H2C | 109.5 | C8—C9—C10 | 118.42 (17) |
| C1—C3—C4 | 121.11 (17) | C8—C9—H9 | 120.8 |
| C1—C3—C2 | 124.19 (18) | C10—C9—H9 | 120.8 |
| C4—C3—C2 | 114.70 (17) | C5—C10—C9 | 119.11 (17) |
| O1—C4—O2 | 122.47 (17) | C5—C10—H10 | 120.4 |
| O1—C4—C3 | 125.08 (17) | C9—C10—H10 | 120.4 |
| O2—C4—C3 | 112.45 (16) | O3—N1—O4 | 123.34 (16) |
| C10—C5—C6 | 122.41 (17) | O3—N1—C8 | 118.44 (15) |
| C10—C5—O2 | 116.30 (16) | O4—N1—C8 | 118.22 (15) |
| C6—C5—O2 | 121.19 (16) | C4—O2—C5 | 118.00 (14) |
| C7—C6—C5 | 118.58 (17) | | |
| C1—C3—C4—O1 | −170.1 (2) | C6—C5—C10—C9 | −0.5 (3) |
| C2—C3—C4—O1 | 9.4 (3) | O2—C5—C10—C9 | 176.01 (16) |
| C1—C3—C4—O2 | 8.8 (3) | C8—C9—C10—C5 | 0.8 (3) |

| | | | |
|---|---|---|---|
| C2—C3—C4—O2 | −171.74 (17) | C9—C8—N1—O3 | 172.43 (18) |
| C10—C5—C6—C7 | 0.3 (3) | C7—C8—N1—O3 | −7.5 (3) |
| O2—C5—C6—C7 | −176.06 (18) | C9—C8—N1—O4 | −7.5 (3) |
| C5—C6—C7—C8 | −0.4 (3) | C7—C8—N1—O4 | 172.65 (18) |
| C6—C7—C8—C9 | 0.8 (3) | O1—C4—O2—C5 | −5.8 (3) |
| C6—C7—C8—N1 | −179.34 (17) | C3—C4—O2—C5 | 175.30 (15) |
| C7—C8—C9—C10 | −1.0 (3) | C10—C5—O2—C4 | 129.61 (19) |
| N1—C8—C9—C10 | 179.14 (16) | C6—C5—O2—C4 | −53.8 (2) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C7—H7···O1$^{i}$ | 0.95 | 2.41 | 3.130 (2) | 133 |
| C1—H1B···O4$^{ii}$ | 0.95 | 2.64 | 3.546 (3) | 159 |
| C2—H2A···O3$^{ii}$ | 0.98 | 2.68 | 3.611 (2) | 159 |
| C9—H9···O4$^{iii}$ | 0.95 | 2.46 | 3.282 (2) | 145 |

Symmetry codes: (i) −*x*+1/2, *y*−1/2, −*z*+1/2; (ii) *x*+1/2, *y*−1/2, *z*; (iii) −*x*, −*y*+2, −*z*.

**Fig. 1**

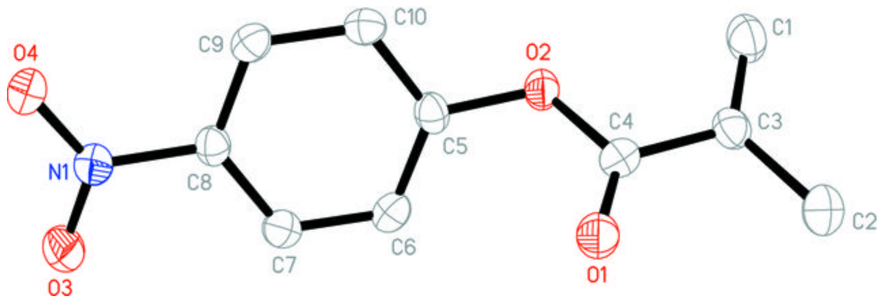

**Fig. 2**