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N-[4-(Prop-2-ynyloxy)phenyl]maleimide

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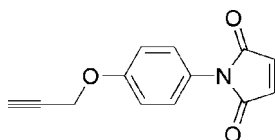
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 Key indicators: single-crystal X-ray study; $T = 292$ K; mean $\sigma(\text{C}-\text{C}) = 0.012$ Å; R factor = 0.072; wR factor = 0.224; data-to-parameter ratio = 13.3.

In the title compound, $\text{C}_{13}\text{H}_9\text{NO}_3$, the dihedral angle between the benzene and maleimide rings is $64.1(2)^\circ$. In the crystal structure, molecules interact *via* $\text{C}-\text{H}\cdots\text{O}$ interactions.

Related literature

N-substituted maleimides can be used in free-radical-initiated polymerization processes upon exposure to light, see: Chang *et al.* (1999); Hoyle *et al.* (1999). For related structures, see: Moreno-Fuquen *et al.* (2006, 2008*a,b*). For the effect of benzene ring substituents on the dihedral angle between the benzene and imidic rings, see: Miller *et al.* (2000).



Experimental

Crystal data

 $\text{C}_{13}\text{H}_9\text{NO}_3$
 $M_r = 227.21$

 Monoclinic, $P2_1/n$
 $a = 9.0428(18)$ Å

 $b = 11.491(2)$ Å

 $c = 11.492(2)$ Å

 $\beta = 102.00(3)^\circ$
 $V = 1168.0(4)$ Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 0.09$ mm⁻¹
 $T = 292(3)$ K

 $0.30 \times 0.26 \times 0.20$ mm

Data collection

 Bruker SMART 1K CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2000)
 $T_{\min} = 0.968$, $T_{\max} = 0.981$

 6107 measured reflections
 2053 independent reflections
 1368 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.088$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.072$
 $wR(F^2) = 0.224$
 $S = 1.35$

2053 reflections

154 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.48$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.43$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}2-\text{H}2\cdots\text{O}2^i$	0.93	2.50	3.179 (12)	130
$\text{C}9-\text{H}9\cdots\text{O}2^{ii}$	0.93	2.18	3.103 (10)	169

 Symmetry codes: (i) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{3}{2}$; (ii) $x + \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$.

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; 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: HG2436).

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supplementary materials

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N-[4-(Prop-2-ynyloxy)phenyl]maleimide

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Comment

N-substituted maleimides can be used in free radical initiated polymerization process upon exposure to light (Chang, *et al.*, 1999; Hoyle, *et al.*, 1999). *N*-(3-Nitrophenyl)maleimide (Moreno-Fuquen, *et al.*, 2006), *N*-(3-Chlorophenyl)maleimide (Moreno-Fuquen, *et al.*, 2008a) and *N*-(4-Chlorophenyl)maleimide (Moreno-Fuquen, *et al.*, 2008b) has reported and could be taken as a reference to compare with the structural characteristics of (I).

Perspective view of (I), showing the atomic numbering scheme, can be seen in Fig. 1. The dihedral angle between the benzene and imidic rings influences on the polymerization process, and substituents of the benzene ring can effect the value of dihedral angle (Miller *et al.* 2000). In the title compound (I), the dihedral angle between the benzene and maleimide is 64.1 (2)°. This angle is 46.46 (5)° for *N*-(3-Chlorophenyl)maleimide (Moreno-Fuquen, *et al.*, 2008:1) and 47.54 (9)° for *N*-(4-Chlorophenyl)maleimide (Moreno-Fuquen, *et al.*, 2008:2). The crystal structure of (I) is stabilized by weak intermolecular C—H···O hydrogen bonds.

Experimental

The title compound was prepared by taking equimolar quantities of 4-(prop-2-ynyloxy)benzenamine (14.7 g, 0.1 mol) and maleic anhydride (9.8 g, 0.1 mol) in 80 ml benzene and 20 ml DMF and refluxing 4 h in the presence of *p*-toluenesulfonic acid. The reaction product was poured into 500 ml ice water, yellow precipitate product was formed. The crude product was recrystallized from ethanol. Yield 90%. ¹H NMR (300 MHz, DMSO-*d*₆) δ 7.30, 7.13(d, aromatic), 7.02(s, 2H, maleimide), 4.85(s, 2H, —H₂—), 3.14(s, 1H, ≡H). Analysis. calculated for C₁₃H₉NO₃: C 68.72, H 3.99, N 6.16%. Found: C 68.39, H 4.02, N 6.21%. The product added in 50 ml ethanol and crystals of (I) suitable for X-ray analysis were obtained by slow evaporation at room temperature.

Refinement

The H atoms bound to C atoms were placed in calculated positions with C—H = 0.93 Å and included in the refinement with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

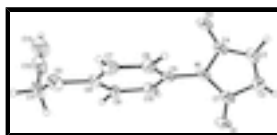


Fig. 1. A view of complex (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

N-[4-(Prop-2-ynyloxy)phenyl]maleimide

Crystal data

$C_{13}H_9NO_3$	$F(000) = 472$
$M_r = 227.21$	$D_x = 1.292 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2yn	Cell parameters from 2053 reflections
$a = 9.0428 (18) \text{ \AA}$	$\theta = 2.5\text{--}25.0^\circ$
$b = 11.491 (2) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$c = 11.492 (2) \text{ \AA}$	$T = 292 \text{ K}$
$\beta = 102.00 (3)^\circ$	Block, yellow
$V = 1168.0 (4) \text{ \AA}^3$	$0.30 \times 0.26 \times 0.20 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART 1K CCD area-detector diffractometer	2053 independent reflections
Radiation source: fine-focus sealed tube graphite	1368 reflections with $I > 2\sigma(I)$
phi and ω scans	$R_{\text{int}} = 0.088$
Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 2000)	$\theta_{\text{max}} = 25.0^\circ$, $\theta_{\text{min}} = 2.5^\circ$
$T_{\text{min}} = 0.968$, $T_{\text{max}} = 0.981$	$h = -10 \rightarrow 10$
6107 measured reflections	$k = -13 \rightarrow 10$
	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.072$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.224$	H-atom parameters constrained
$S = 1.35$	$w = 1/[\sigma^2(F_o^2) + (0.0733P)^2 + 3.1182P]$
2053 reflections	where $P = (F_o^2 + 2F_c^2)/3$
154 parameters	$(\Delta/\sigma)_{\text{max}} < 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 0.49 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.43 \text{ e \AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C3	0.2361 (11)	0.5326 (8)	0.6724 (8)	0.088 (4)
H3	0.2039	0.5394	0.5904	0.106*
O3	0.0885 (6)	0.1202 (5)	1.2278 (5)	0.0694 (19)
C5	0.1785 (7)	0.3652 (6)	0.9539 (6)	0.052 (2)
O1	0.3695 (7)	0.5806 (5)	0.9313 (5)	0.083 (2)
O2	0.0834 (8)	0.3616 (6)	0.7229 (6)	0.094 (3)
C8	0.1352 (8)	0.1986 (7)	1.1335 (6)	0.048 (2)
C9	0.2725 (8)	0.2328 (7)	1.0900 (6)	0.054 (2)
H9	0.3586	0.1960	1.1323	0.065*
C10	0.3076 (7)	0.3112 (7)	0.9958 (6)	0.053 (2)
H10	0.3992	0.3200	0.9720	0.064*
C7	0.0089 (9)	0.2487 (8)	1.0914 (8)	0.066 (3)
H7	-0.0834	0.2363	1.1129	0.079*
C6	0.0429 (7)	0.3326 (7)	0.9989 (7)	0.059 (2)
H6	-0.0424	0.3736	0.9613	0.071*
C1	0.3150 (9)	0.5431 (8)	0.8502 (7)	0.062 (3)
C4	0.1745 (10)	0.4410 (7)	0.7421 (7)	0.070 (3)
C11	0.2080 (11)	0.0481 (9)	1.2619 (8)	0.077 (3)
H11A	0.1862	0.0016	1.3268	0.093*
H11B	0.2935	0.0971	1.2958	0.093*
C2	0.3280 (11)	0.5958 (8)	0.7276 (8)	0.078 (3)
H2	0.3866	0.6553	0.7059	0.094*
C12	0.2667 (12)	-0.0409 (10)	1.1735 (10)	0.081 (3)
C13	0.3170 (15)	-0.1138 (12)	1.1030 (12)	0.113 (5)
H13	0.3526	-0.1656	1.0530	0.136*
N1	0.2168 (7)	0.4475 (6)	0.8557 (5)	0.0543 (19)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C3	0.115 (8)	0.070 (6)	0.055 (6)	0.005 (6)	-0.038 (6)	0.016 (5)
O3	0.068 (4)	0.078 (4)	0.052 (3)	0.000 (3)	-0.010 (3)	0.011 (3)
C5	0.053 (4)	0.053 (5)	0.038 (4)	-0.006 (4)	-0.017 (4)	-0.001 (4)
O1	0.093 (5)	0.077 (4)	0.058 (4)	-0.021 (4)	-0.038 (4)	-0.001 (3)
O2	0.107 (5)	0.068 (4)	0.074 (4)	-0.006 (4)	-0.056 (4)	0.003 (3)
C8	0.064 (5)	0.049 (5)	0.026 (4)	-0.009 (4)	-0.003 (4)	0.009 (3)
C9	0.059 (5)	0.054 (5)	0.039 (4)	-0.021 (4)	-0.010 (4)	0.016 (4)

supplementary materials

C10	0.039 (4)	0.068 (5)	0.045 (5)	-0.006 (4)	-0.007 (4)	-0.009 (4)
C7	0.045 (5)	0.075 (6)	0.069 (6)	-0.013 (4)	-0.006 (4)	0.001 (5)
C6	0.057 (5)	0.051 (5)	0.058 (5)	-0.002 (4)	-0.014 (4)	0.007 (4)
C1	0.057 (5)	0.069 (6)	0.047 (5)	0.002 (4)	-0.018 (4)	-0.003 (5)
C4	0.082 (6)	0.052 (5)	0.054 (5)	0.013 (5)	-0.034 (5)	-0.002 (4)
C11	0.082 (6)	0.096 (8)	0.042 (5)	-0.010 (6)	-0.012 (5)	0.036 (5)
C2	0.088 (7)	0.058 (6)	0.072 (6)	-0.011 (5)	-0.020 (5)	0.016 (5)
C12	0.085 (7)	0.083 (8)	0.068 (7)	0.019 (6)	0.000 (6)	0.028 (6)
C13	0.134 (11)	0.098 (9)	0.092 (9)	0.023 (9)	-0.014 (8)	0.019 (8)
N1	0.051 (4)	0.064 (4)	0.036 (4)	0.004 (3)	-0.018 (3)	0.000 (3)

Geometric parameters (Å, °)

C3—C2	1.184 (11)	C10—H10	0.9300
C3—C4	1.498 (9)	C7—C6	1.513 (11)
C3—H3	0.9300	C7—H7	0.9300
O3—C11	1.353 (10)	C6—H6	0.9300
O3—C8	1.535 (9)	C1—N1	1.422 (11)
C5—C10	1.321 (7)	C1—C2	1.560 (12)
C5—C6	1.475 (8)	C4—N1	1.284 (10)
C5—N1	1.565 (10)	C11—C12	1.608 (16)
O1—C1	1.052 (9)	C11—H11A	0.9700
O2—C4	1.218 (10)	C11—H11B	0.9700
C8—C7	1.281 (10)	C2—H2	0.9300
C8—C9	1.484 (8)	C12—C13	1.311 (16)
C9—C10	1.493 (10)	C13—H13	0.9300
C9—H9	0.9300		
C2—C3—C4	116.3 (8)	C7—C6—H6	112.3
C2—C3—H3	121.9	O1—C1—N1	117.3 (9)
C4—C3—H3	121.9	O1—C1—C2	122.1 (9)
C11—O3—C8	104.1 (6)	N1—C1—C2	120.4 (7)
C10—C5—C6	119.3 (7)	O2—C4—N1	106.0 (8)
C10—C5—N1	103.6 (6)	O2—C4—C3	137.9 (8)
C6—C5—N1	136.9 (6)	N1—C4—C3	115.9 (8)
C7—C8—C9	119.8 (7)	O3—C11—C12	123.7 (7)
C7—C8—O3	100.1 (7)	O3—C11—H11A	106.4
C9—C8—O3	139.9 (6)	C12—C11—H11A	106.4
C8—C9—C10	136.3 (6)	O3—C11—H11B	106.4
C8—C9—H9	111.9	C12—C11—H11B	106.4
C10—C9—H9	111.9	H11A—C11—H11B	106.5
C5—C10—C9	104.1 (6)	C3—C2—C1	93.9 (8)
C5—C10—H10	128.0	C3—C2—H2	133.0
C9—C10—H10	128.0	C1—C2—H2	133.0
C8—C7—C6	104.9 (7)	C13—C12—C11	178.9 (10)
C8—C7—H7	127.6	C12—C13—H13	180.0
C6—C7—H7	127.6	C4—N1—C1	93.2 (7)
C5—C6—C7	135.5 (6)	C4—N1—C5	129.4 (7)
C5—C6—H6	112.3	C1—N1—C5	137.1 (5)
C11—O3—C8—C7	169.0 (7)	C4—C3—C2—C1	-4.4 (12)

C11—O3—C8—C9	-16.0 (12)	O1—C1—C2—C3	-172.0 (11)
C7—C8—C9—C10	-5.8 (14)	N1—C1—C2—C3	2.3 (12)
O3—C8—C9—C10	179.9 (8)	O2—C4—N1—C1	-179.3 (7)
C6—C5—C10—C9	-4.1 (9)	C3—C4—N1—C1	-3.7 (9)
N1—C5—C10—C9	179.7 (5)	O2—C4—N1—C5	6.4 (12)
C8—C9—C10—C5	6.6 (12)	C3—C4—N1—C5	-178.0 (7)
C9—C8—C7—C6	1.8 (10)	O1—C1—N1—C4	175.7 (9)
O3—C8—C7—C6	178.1 (6)	C2—C1—N1—C4	1.2 (10)
C10—C5—C6—C7	2.5 (14)	O1—C1—N1—C5	-10.7 (15)
N1—C5—C6—C7	177.1 (8)	C2—C1—N1—C5	174.7 (8)
C8—C7—C6—C5	-0.8 (13)	C10—C5—N1—C4	109.2 (9)
C2—C3—C4—O2	-179.8 (12)	C6—C5—N1—C4	-65.9 (13)
C2—C3—C4—N1	6.5 (14)	C10—C5—N1—C1	-62.5 (11)
C8—O3—C11—C12	-61.5 (10)	C6—C5—N1—C1	122.4 (10)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C2—H2 \cdots O2 ⁱ	0.93	2.50	3.179 (12)	130
C9—H9 \cdots O2 ⁱⁱ	0.93	2.18	3.103 (10)	169

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

Fig. 1

