

# Two polymorphs of 2-(prop-2-yn-1-yloxy)-naphthalene-1,4-dione: solvent-dependent crystallization

Flaviano Melo Ottoni,<sup>a\*</sup> Raquel Geralda Isidório,<sup>a</sup> Ricardo José Alves<sup>a</sup> and Nivaldo Lúcio Speziali<sup>b</sup>

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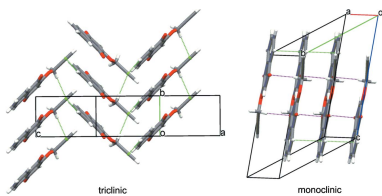
**Supporting information:** this article has supporting information at journals.iucr.org/e

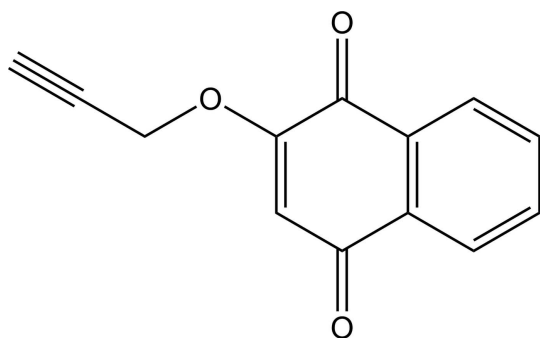
<sup>a</sup>Departamento de Produtos Farmacêuticos, Faculdade de Farmácia, Universidade Federal de Minas Gerais, Avenida Antônio Carlos, 6627, Belo Horizonte, MG, CEP 31.270-901, Brazil, and <sup>b</sup>Departamento de Física, Instituto de Ciências Exatas, Universidade Federal de Minas Gerais, Avenida Antônio Carlos, 6627, Belo Horizonte, MG, CEP 31.270-901, Brazil. \*Correspondence e-mail: fmottoni@hotmail.com

The title compound, C<sub>13</sub>H<sub>8</sub>O<sub>3</sub>, crystallizes in two polymorphs, namely the monoclinic (space group  $P2_1/c$ ) and triclinic (space group  $P\bar{1}$ ) forms, obtained from *N,N*-dimethylformamide and isopropyl alcohol solutions, respectively. The molecular structures and conformations in the two forms are essentially the same as each other. The naphthoquinone ring systems are essentially planar with r.m.s. deviations of 0.015 and 0.029 Å for the monoclinic and triclinic forms, respectively. The *O*-propargyl groups are coplanar with the naphthoquinone units with r.m.s. deviations ranging from 0.04 to 0.09 Å. In the monoclinic crystal, molecules are linked *via* pairs of C—H···O hydrogen bonds, forming a tape structure running along [120]. The tapes are further linked by a C—H··· $\pi$  interaction into a layer parallel to the *ab* plane. Adjacent layers are linked by another C—H··· $\pi$  interaction. In the triclinic crystal, molecules are linked *via* C—H···O and  $\pi$ – $\pi$  interactions, forming a layer parallel to the *ab* plane. Adjacent layers are linked by a C—H··· $\pi$  interaction.

## 1. Chemical context

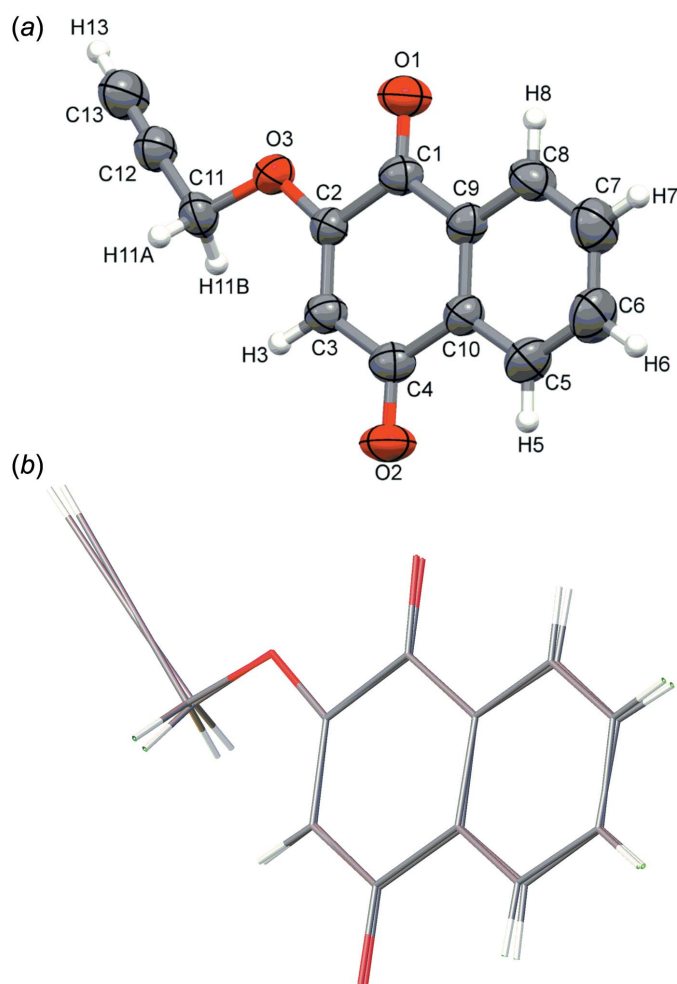
Naphthoquinone derivatives have been studied intensively over the past few decades, mostly because of their numerous biological activities, mainly antimicrobial and antitumor (Fujii *et al.*, 1992; Hussain *et al.*, 2007; Epifano *et al.*, 2014). The main mechanism of the activity is related to the formation of reactive oxygen species (ROS) through semiquinonic radicals, which cause damage to cell macromolecules and consequently cell death (Da Silva *et al.*, 2003). Among the substances that comprise this class, some synthetic bioactive derivatives have been obtained from lawsone (2-hydroxynaphthalene-1,4-dione) (Jordão *et al.*, 2015). In a basic medium, lawsone shows three sites able to be alkylated (Lamoureux *et al.*, 2008), resulting in *O*-alkyl and *C*-alkyl derivatives difficult to purify in some cases in some cases (Kongkathip *et al.*, 2003). The title compound was obtained in higher yields since oxygen better accommodates the negative charge generated in the enolate formation, using a weak base, propargyl bromide, aprotic solvent and heat. The product has an alkyne terminal chain and can be used as the starting material in the synthesis of triazole derivatives, which are widely exploited in medicinal chemistry (Haider *et al.*, 2014). The present study shows that the title compound has two polymorphs, monoclinic (space group  $P2_1/c$ ) and triclinic (space group  $P\bar{1}$ ), crystallized from *N,N*-dimethylformamide (DMF) and isopropyl alcohol, respectively.





## 2. Structural commentary

The molecular structures in the two polymorphs are essentially the same (Fig. 1). The naphthoquinone ring systems in the monoclinic and triclinic forms are both planar, with r.m.s. deviations of 0.015 and 0.029 Å, respectively, for the non-H atoms. Each propargyl group is coplanar with the naphtho-



**Figure 1**  
(a) The molecular structure of the title compound (monoclinic form) with the atom labelling. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level. (b) A view of the overlay of the molecular structures of the monoclinic and triclinic forms of the title compound.

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

Cg2 is the midpoint of the C12≡C13 bond.

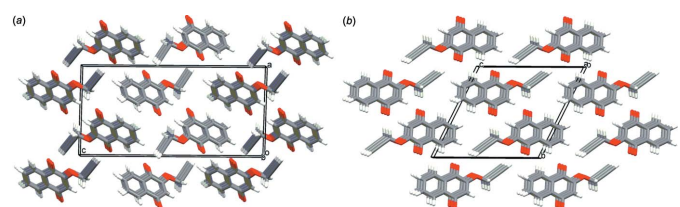
<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
<b>Monoclinic form</b>				
C3—H3...O2 <sup>i</sup>	0.93	2.58	3.436 (2)	153
C13—H13...O1 <sup>ii</sup>	0.93	2.33	3.350 (2)	173
C11—H11A...Cg2 <sup>iii</sup>	0.97	2.91	3.740 (4)	145
C7—H7...Cg2 <sup>iv</sup>	0.93	2.87	3.703 (4)	151
<b>Triclinic form</b>				
C3—H3...O2 <sup>i</sup>	0.93	2.49	3.409 (4)	173
C11—H11B...O2 <sup>ii</sup>	0.97	2.52	3.380 (4)	149
C13—H13...O1 <sup>iii</sup>	0.93	2.44	3.340 (5)	164
C7—H7...Cg2 <sup>v</sup>	0.93	2.93	3.829 (4)	162

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

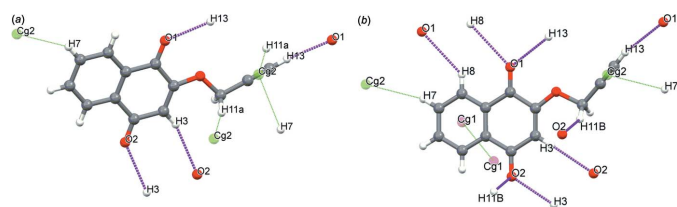
quinone ring system, with C1—C2—O3—C11 and C2—O3—C11—C12 torsion angles being  $-178.8$  (1) and  $175.9$  (1) $^\circ$ , respectively, for the monoclinic form, and  $-177.1$  (3) and  $-171.9$  (3) $^\circ$ , respectively, for the triclinic form.

## 3. Supramolecular features

The molecular arrangements in both crystals are similar (Fig. 2) with nearly the same crystal densities ( $\rho = 1.383$  and  $1.392$  Mg m $^{-3}$  for the monoclinic and triclinic forms, respectively). In the monoclinic crystal, molecules are linked *via* pairs of C—H...O hydrogen bonds (C3—H3...O2<sup>i</sup> and C13—H13...O1<sup>ii</sup>; symmetry codes as in Table 1), forming a tape structure running along [120]. The tapes are further linked by a C—H... $\pi$  interaction (C11—H11A...Cg2<sup>iii</sup>; Table 1) into a layer parallel to the *ab* plane; Cg2 is the centroid of the C12≡C13 triple bond [Fig. 3(a)]. In the layer, molecules are arranged parallel to each other and adjacent layers are linked by another C—H... $\pi$  interaction (C7—H7...Cg2<sup>iv</sup>; Table 1), forming a three-dimensional network [Fig. 4(a)]. In the triclinic crystal, molecules are linked *via* C—H...O interactions (C3—H3...O2<sup>i</sup>, C11—H11B...O2<sup>ii</sup> and C13—H13...O1<sup>iii</sup>; Table 1) and  $\pi$ — $\pi$  interactions with centroid-centroid distances of 3.9906 (18) and 3.991 (2) Å, respectively, between C1—C4/C10/C9 rings and between C5—C10 rings, forming a layer parallel to the *ab* plane [Fig. 3(b)]. Adjacent layers are linked by a C—H... $\pi$  interaction [Fig. 4(b); C7—H7...Cg2<sup>v</sup>; Table 1].



**Figure 2**  
Packing diagrams of the title compound, showing the stacked naphthoquinone molecules: (a) monoclinic form viewed along the *b* axis and (b) triclinic form viewed along the *a* axis.


**Figure 3**

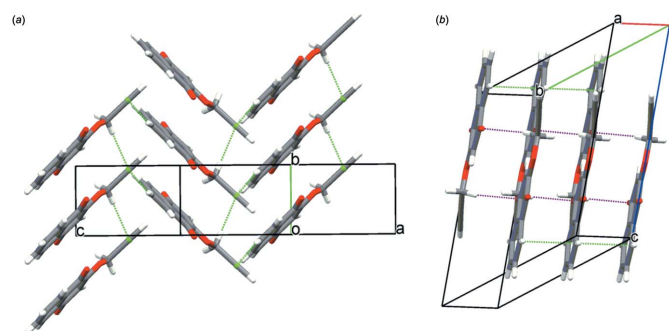
Selected intermolecular interactions in the crystals of (a) the monoclinic form and (b) the triclinic form. Purple dashed lines represent the C–H···O hydrogen bonds and green dashed lines the C–H··· $\pi$  and  $\pi$ – $\pi$  interactions. Cg1 is the centroid of the C5–C10 ring, while Cg2 is the midpoint of the C12=C13 bond.

#### 4. Database survey

A search of the Cambridge Structural Database (Version 5.38; Groom *et al.*, 2016) for naphthalene-1,4-dione gave about 790 structures. Among them, 2-methoxynaphthalene-1,4-dione (Jin *et al.*, 2011) and 2-[[1-(4-bromobenzyl)-1*H*-1,2,3-triazol-4-yl]methoxy]naphthalene-1,4-dione (Raja *et al.*, 2015) are very similar to the title compound. These compounds exhibit additional functional groups linked at O3 and essentially planar naphthoquinone ring systems and C–H···O and  $\pi$ – $\pi$  interactions are also observed in their crystal structures.

#### 5. Synthesis and crystallization

The synthesis of the title compound was achieved in one step according to the literature method (Raja *et al.*, 2015). To a solution of lawsone (0.20 g, 1.15 mmol) in DMF (10 ml) was


**Figure 4**

Partial packing diagrams of (a) the monoclinic form and (b) the triclinic form. Purple dashed lines represent the C–H···O hydrogen bonds and green dashed lines the C–H··· $\pi$  and  $\pi$ – $\pi$  interactions.

added  $K_2CO_3$  (0.16 g, 1.15 mmol) and propargyl bromide (0.48 g, 4.07 mmol). The mixture was stirred at 363 K for 24 h. Then hydrochloric acid (1.0 mol l<sup>−1</sup>, 0.34 ml) was added and the resulting solution was extracted with dichloromethane (3 × 25 ml). The organic layers were washed with water (60 ml), dried over anhydrous sodium sulfate and concentrated. The solid obtained was purified by column chromatography using silica gel and hexane–ethyl acetate (9:1) and furnished the title compound in 70% yield. Yellow single crystals of the monoclinic and triclinic forms (m.p. 420.0–423.1 K) suitable for X-ray diffraction were obtained by slow evaporation of DMF and isopropyl alcohol solutions (about 0.5 mg ml<sup>−1</sup>), respectively, at room temperature.

**Table 2**  
Experimental details.

	Monoclinic	Triclinic
Crystal data		
Chemical formula	C <sub>13</sub> H <sub>8</sub> O <sub>3</sub>	C <sub>13</sub> H <sub>8</sub> O <sub>3</sub>
<i>M<sub>r</sub></i>	212.19	212.19
Crystal system, space group	Monoclinic, <i>P</i> 2 <sub>1</sub> / <i>c</i>	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	293	293
<i>a</i> , <i>b</i> , <i>c</i> (Å)	10.0911 (7), 4.8021 (3), 20.8939 (15)	3.9906 (6), 11.6943 (16), 12.3413 (18)
$\alpha$ , $\beta$ , $\gamma$ (°)	90, 91.174 (7), 90	63.347 (14), 83.343 (12), 83.018 (12)
<i>V</i> (Å <sup>3</sup> )	1012.27 (12)	509.69 (14)
<i>Z</i>	4	2
Radiation type	Mo <i>K</i> $\alpha$	Mo <i>K</i> $\alpha$
$\mu$ (mm <sup>−1</sup> )	0.10	0.10
Crystal size (mm)	0.35 × 0.2 × 0.1	0.35 × 0.2 × 0.1
Data collection		
Diffractometer	Rigaku Xcalibur Atlas Gemini ultra	Rigaku Xcalibur Atlas Gemini ultra
Absorption correction	Multi-scan ( <i>CrysAlis PRO</i> ; Rigaku OD, 2015)	Multi-scan ( <i>CrysAlis PRO</i> ; Rigaku OD, 2015)
<i>T<sub>min</sub></i> , <i>T<sub>max</sub></i>	0.764, 1.000	0.773, 1.000
No. of measured, independent and observed [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] reflections	10652, 3452, 1765	7730, 2503, 923
<i>R<sub>int</sub></i>	0.058	0.084
Refinement		
<i>R</i> [ <i>F</i> <sup>2</sup> > 2 $\sigma$ ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.059, 0.156, 1.01	0.064, 0.184, 1.00
No. of reflections	3452	2503
No. of parameters	145	145
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$ , $\Delta\rho_{min}$ (e Å <sup>−3</sup> )	0.19, −0.20	0.24, −0.24

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXT* (Sheldrick, 2015a), *SHELXL2014* (Sheldrick, 2015b) and *OLEX2* (Dolomanov *et al.*, 2009).

Spectrometric data. IR  $\nu_{\max}$  ( $\text{cm}^{-1}$ ): The spectrum show the characteristic absorption bands of the main functional groups for title compound at IR ( $\nu$  max/ $\text{cm}^{-1}$ ): 3250 (C–H alkyne), 3053 (C–H aromatic), 2130 (C $\equiv$ C) 1649, 1680 (C=O quinone), 1575–1604 (C–C aromatic), 1016, 1208 and 1245 (C–O).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta_{\text{H}}$  8.12 (*dd*, 1H,  $J_{5,6}$  7.1 Hz,  $J_{8,7}$  1.9 Hz, H-5), 8.07 (*dd*, 1H,  $J_{8,7}$  7.0 Hz,  $J_{8,6}$  1.9 Hz, H-8), 7.74 (*td*, 1H,  $J_{6,5}$  7.5 Hz,  $J_{6,7}$  7.5 Hz,  $J_{6,8}$  1.7 Hz, H-6), 7.70 (*td*, 1H,  $J_{7,6}$  7.4 Hz,  $J_{7,8}$  7.4 Hz,  $J_{7,5}$  1.6 Hz, H-7), 6.33 (*s*, 1H, H-3), 4.78 (*d*, 2H,  $J_{11,13}$  2.4 Hz, H-11), 2.63 (*t*, 1H,  $J_{13,11}$  2.4 Hz, H-13).  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta_{\text{C}}$  184.6 (C-4), 179.8 (C-1), 158.1 (C-2), 134.3 (C-6), 133.4 (C-7), 131.9 (C-10), 131.1 (C-9), 126.7 (C-5), 126.2 (C-8), 111.6 (C-3), 78.2 (C-13), 75.5 (C-12), 56.7 (C-11).

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. C-bound H atoms were constrained to an ideal geometry with C–H = 0.93–0.97 Å and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

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## supporting information

*Acta Cryst.* (2018). E74, 1731-1734 [https://doi.org/10.1107/S2056989018015438]

## Two polymorphs of 2-(prop-2-yn-1-yloxy)naphthalene-1,4-dione: solvent-dependent crystallization

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### Computing details

For both structures, data collection: *CrysAlis PRO* (Rigaku OD, 2015); cell refinement: *CrysAlis PRO* (Rigaku OD, 2015); data reduction: *CrysAlis PRO* (Rigaku OD, 2015); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

### 2-(Prop-2-yn-1-yloxy)naphthalene-1,4-dione (Monoclinic)

#### Crystal data

$C_{13}H_8O_3$	$F(000) = 440$
$M_r = 212.19$	$D_x = 1.392 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 10.0911 (7) \text{ \AA}$	Cell parameters from 1655 reflections
$b = 4.8021 (3) \text{ \AA}$	$\theta = 3.9\text{--}30.6^\circ$
$c = 20.8939 (15) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$\beta = 91.174 (7)^\circ$	$T = 293 \text{ K}$
$V = 1012.27 (12) \text{ \AA}^3$	Rod, yellow
$Z = 4$	$0.35 \times 0.2 \times 0.1 \text{ mm}$

#### Data collection

Rigaku Xcalibur Atlas Gemini ultra diffractometer	$T_{\min} = 0.764$ , $T_{\max} = 1.000$
Radiation source: fine-focus sealed X-ray tube, Enhance (Mo) X-ray Source	10652 measured reflections
Graphite monochromator	3452 independent reflections
Detector resolution: $10.4186 \text{ pixels mm}^{-1}$	1765 reflections with $I > 2\sigma(I)$
$\omega$ scans	$R_{\text{int}} = 0.058$
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2015)	$\theta_{\max} = 32.8^\circ$ , $\theta_{\min} = 2.8^\circ$
	$h = -13 \rightarrow 15$
	$k = -7 \rightarrow 7$
	$l = -30 \rightarrow 31$

#### Refinement

Refinement on $F^2$	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.059$	$w = 1/[\sigma^2(F_o^2) + (0.0571P)^2]$
$wR(F^2) = 0.156$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{\max} < 0.001$
3452 reflections	$\Delta\rho_{\max} = 0.19 \text{ e \AA}^{-3}$
145 parameters	$\Delta\rho_{\min} = -0.20 \text{ e \AA}^{-3}$
0 restraints	
Primary atom site location: dual	

*Special details*

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.08336 (12)	0.4803 (2)	0.34723 (5)	0.0512 (3)
O2	0.50658 (13)	1.0905 (3)	0.41264 (6)	0.0646 (4)
O3	0.19024 (11)	0.3978 (2)	0.46005 (5)	0.0493 (3)
C1	0.17768 (15)	0.6268 (3)	0.36127 (7)	0.0381 (4)
C2	0.24795 (16)	0.5949 (3)	0.42459 (7)	0.0390 (4)
C3	0.35379 (16)	0.7492 (3)	0.44063 (7)	0.0434 (4)
H3	0.3937	0.7248	0.4807	0.052*
C4	0.40849 (16)	0.9546 (3)	0.39706 (8)	0.0419 (4)
C5	0.38922 (16)	1.1922 (3)	0.29135 (8)	0.0445 (4)
H5	0.4645	1.2953	0.3021	0.053*
C6	0.32525 (17)	1.2340 (4)	0.23311 (8)	0.0495 (4)
H6	0.3576	1.3651	0.2046	0.059*
C7	0.21324 (18)	1.0817 (4)	0.21695 (8)	0.0528 (5)
H7	0.1701	1.1116	0.1778	0.063*
C8	0.16518 (16)	0.8854 (4)	0.25877 (8)	0.0460 (4)
H8	0.0899	0.7832	0.2477	0.055*
C9	0.22882 (15)	0.8402 (3)	0.31716 (7)	0.0362 (3)
C10	0.34126 (15)	0.9961 (3)	0.33397 (7)	0.0363 (4)
C11	0.24569 (19)	0.3468 (3)	0.52323 (7)	0.0488 (4)
H11A	0.2477	0.5175	0.5480	0.059*
H11B	0.3354	0.2761	0.5204	0.059*
C12	0.16027 (19)	0.1408 (4)	0.55324 (8)	0.0510 (5)
C13	0.0907 (2)	-0.0220 (4)	0.57682 (9)	0.0649 (6)
H13	0.0352	-0.1518	0.5956	0.078*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0476 (7)	0.0589 (7)	0.0465 (7)	-0.0115 (6)	-0.0106 (5)	0.0087 (5)
O2	0.0593 (8)	0.0758 (9)	0.0578 (9)	-0.0230 (7)	-0.0195 (6)	0.0080 (7)
O3	0.0576 (8)	0.0521 (7)	0.0377 (6)	-0.0075 (6)	-0.0101 (5)	0.0117 (5)
C1	0.0386 (8)	0.0394 (8)	0.0362 (8)	0.0037 (7)	-0.0047 (6)	-0.0009 (7)
C2	0.0474 (9)	0.0381 (8)	0.0315 (8)	0.0030 (7)	-0.0020 (7)	0.0039 (6)
C3	0.0494 (9)	0.0474 (9)	0.0330 (8)	0.0008 (8)	-0.0112 (7)	0.0019 (7)
C4	0.0415 (9)	0.0438 (9)	0.0402 (9)	0.0014 (7)	-0.0068 (7)	-0.0023 (7)
C5	0.0397 (8)	0.0453 (9)	0.0485 (10)	-0.0006 (7)	0.0016 (7)	0.0030 (8)
C6	0.0524 (10)	0.0523 (10)	0.0439 (10)	0.0008 (8)	0.0037 (8)	0.0122 (8)
C7	0.0571 (11)	0.0630 (11)	0.0380 (9)	0.0015 (9)	-0.0095 (8)	0.0115 (8)
C8	0.0453 (9)	0.0519 (10)	0.0405 (9)	-0.0034 (8)	-0.0086 (7)	0.0065 (7)

C9	0.0367 (8)	0.0374 (8)	0.0343 (8)	0.0040 (7)	-0.0028 (6)	0.0017 (6)
C10	0.0361 (8)	0.0369 (8)	0.0356 (8)	0.0060 (6)	-0.0025 (6)	-0.0011 (6)
C11	0.0655 (11)	0.0482 (10)	0.0323 (8)	-0.0007 (9)	-0.0078 (7)	0.0043 (7)
C12	0.0674 (12)	0.0489 (10)	0.0366 (9)	0.0023 (9)	-0.0013 (8)	0.0006 (8)
C13	0.0780 (15)	0.0633 (12)	0.0536 (12)	-0.0049 (11)	0.0071 (10)	0.0095 (10)

*Geometric parameters (Å, °)*

O1—C1	1.2145 (18)	C8—C7	1.380 (2)
O2—C4	1.2241 (18)	C8—H8	0.9300
O3—C2	1.3423 (18)	C9—C10	1.398 (2)
O3—C11	1.4442 (19)	C9—C1	1.478 (2)
C1—C2	1.496 (2)	C9—C8	1.384 (2)
C2—C3	1.337 (2)	C10—C4	1.483 (2)
C3—H3	0.9300	C10—C5	1.390 (2)
C4—C3	1.458 (2)	C11—C12	1.462 (3)
C5—C6	1.380 (2)	C11—H11A	0.9700
C5—H5	0.9300	C11—H11B	0.9700
C6—C7	1.382 (2)	C12—C13	1.167 (2)
C6—H6	0.9300	C13—H13	0.9300
C7—H7	0.9300		
O1—C1—C9	122.19 (14)	C6—C5—H5	120.0
O1—C1—C2	120.61 (14)	C6—C7—H7	119.9
O2—C4—C10	121.15 (15)	C7—C8—C9	120.16 (16)
O2—C4—C3	120.59 (15)	C7—C6—H6	119.9
O3—C2—C1	110.92 (14)	C7—C8—H8	119.9
O3—C11—H11A	110.4	C8—C9—C10	119.80 (14)
O3—C11—H11B	110.4	C8—C9—C1	119.77 (14)
O3—C11—C12	106.62 (14)	C8—C7—C6	120.20 (16)
C2—O3—C11	117.37 (13)	C8—C7—H7	119.9
C2—C3—C4	121.94 (14)	C9—C10—C4	120.35 (14)
C2—C3—H3	119.0	C9—C1—C2	117.20 (14)
C3—C2—O3	127.29 (15)	C9—C8—H8	119.9
C3—C2—C1	121.78 (14)	C10—C9—C1	120.42 (14)
C3—C4—C10	118.26 (14)	C10—C5—H5	120.0
C4—C3—H3	119.0	H11A—C11—H11B	108.6
C5—C6—H6	119.9	C12—C11—H11A	110.4
C5—C6—C7	120.24 (15)	C12—C11—H11B	110.4
C5—C10—C9	119.55 (14)	C12—C13—H13	180.0
C5—C10—C4	120.10 (15)	C13—C12—C11	179.1 (2)
C6—C5—C10	120.05 (16)		
O1—C1—C2—O3	-1.5 (2)	C8—C9—C1—C2	-177.9 (1)
O1—C1—C2—C3	178.5 (1)	C9—C1—C2—O3	179.1 (1)
O2—C4—C3—C2	-179.0 (2)	C9—C10—C4—O2	-179.6 (2)
O3—C2—C3—C4	178.9 (1)	C9—C10—C5—C6	0.6 (2)
C1—C9—C8—C7	-179.2 (2)	C9—C8—C7—C6	0.1 (3)

C1—C9—C10—C5	178.8 (1)	C9—C1—C2—C3	-1.0 (2)
C1—C9—C10—C4	-1.7 (2)	C9—C10—C4—C3	-0.3 (2)
C1—C2—C3—C4	-1.0 (2)	C10—C9—C1—O1	-177.1 (1)
C2—O3—C11—C12	175.9 (1)	C10—C5—C6—C7	0.1 (3)
C5—C10—C4—O2	-0.1 (2)	C10—C9—C8—C7	0.7 (2)
C5—C6—C7—C8	-0.5 (3)	C10—C9—C1—C2	2.3 (2)
C8—C9—C1—O1	2.7 (2)	C10—C4—C3—C2	1.7 (2)
C8—C9—C10—C5	-1.0 (2)	C11—O3—C2—C3	1.2 (2)
C8—C9—C10—C4	178.5 (1)	C11—O3—C2—C1	-178.8 (1)

## 2-(Prop-2-yn-1-yloxy)naphthalene-1,4-dione (Triclinic)

### Crystal data

$C_{13}H_8O_3$

$M_r = 212.19$

Triclinic,  $P\bar{1}$

$a = 3.9906$  (6) Å

$b = 11.6943$  (16) Å

$c = 12.3413$  (16) Å

$\alpha = 63.347$  (14)°

$\beta = 83.343$  (12)°

$\gamma = 83.018$  (12)°

$V = 509.69$  (14) Å<sup>3</sup>

$Z = 2$

$F(000) = 220$

$D_x = 1.383$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 796 reflections

$\theta = 3.2$ – $22.3$ °

$\mu = 0.10$  mm<sup>-1</sup>

$T = 293$  K

Rod, yellow

$0.35 \times 0.2 \times 0.1$  mm

### Data collection

Rigaku Xcalibur Atlas Gemini ultra  
diffractometer

Radiation source: fine-focus sealed X-ray tube,  
Enhance (Mo) X-ray Source

Graphite monochromator

Detector resolution: 10.4186 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: multi-scan  
(CrysAlis PRO; Rigaku OD, 2015)

$T_{\min} = 0.773$ ,  $T_{\max} = 1.000$

7730 measured reflections

2503 independent reflections

923 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.084$

$\theta_{\max} = 29.5$ °,  $\theta_{\min} = 3.2$ °

$h = -5 \rightarrow 5$

$k = -14 \rightarrow 15$

$l = -16 \rightarrow 16$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.064$

$wR(F^2) = 0.184$

$S = 1.00$

2503 reflections

145 parameters

0 restraints

Primary atom site location: dual

Hydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0469P)^2]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.24$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.23$  e Å<sup>-3</sup>

### Special details

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.



Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.1734 (6)	0.9294 (2)	0.1987 (2)	0.0641 (7)
O2	0.9527 (6)	0.4894 (2)	0.3422 (2)	0.0669 (8)
O3	0.3732 (5)	0.82577 (18)	0.41940 (19)	0.0502 (6)
C1	0.3597 (7)	0.8310 (3)	0.2279 (3)	0.0434 (8)
C2	0.4859 (7)	0.7629 (3)	0.3529 (3)	0.0403 (8)
C3	0.6822 (7)	0.6529 (3)	0.3868 (3)	0.0446 (8)
H3	0.7612	0.6142	0.4642	0.053*
C4	0.7756 (7)	0.5920 (3)	0.3068 (3)	0.0469 (8)
C5	0.7466 (8)	0.6005 (3)	0.1040 (3)	0.0623 (10)
H5	0.8690	0.5209	0.1307	0.075*
C6	0.6507 (9)	0.6636 (4)	−0.0147 (4)	0.0734 (12)
H6	0.7105	0.6264	−0.0676	0.088*
C7	0.4685 (9)	0.7804 (4)	−0.0543 (3)	0.0693 (11)
H7	0.4083	0.8226	−0.1343	0.083*
C8	0.3734 (8)	0.8360 (3)	0.0234 (3)	0.0544 (9)
H8	0.2467	0.9147	−0.0038	0.065*
C9	0.4678 (7)	0.7737 (3)	0.1427 (3)	0.0428 (8)
C10	0.6598 (7)	0.6563 (3)	0.1823 (3)	0.0447 (8)
C11	0.4891 (8)	0.7741 (3)	0.5405 (3)	0.0532 (9)
H11A	0.7294	0.7831	0.5362	0.064*
H11B	0.4510	0.6837	0.5849	0.064*
C12	0.2992 (8)	0.8455 (3)	0.6013 (3)	0.0518 (9)
C13	0.1377 (9)	0.9034 (3)	0.6476 (3)	0.0692 (11)
H13	0.0087	0.9496	0.6846	0.083*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0807 (17)	0.0553 (14)	0.0518 (16)	0.0214 (13)	−0.0130 (12)	−0.0243 (12)
O2	0.0784 (17)	0.0510 (14)	0.0670 (17)	0.0249 (13)	−0.0154 (13)	−0.0273 (12)
O3	0.0621 (14)	0.0495 (13)	0.0460 (14)	0.0123 (10)	−0.0128 (11)	−0.0294 (11)
C1	0.0431 (18)	0.0423 (18)	0.044 (2)	0.0002 (14)	0.0003 (15)	−0.0204 (16)
C2	0.0425 (18)	0.0415 (18)	0.040 (2)	0.0020 (14)	−0.0068 (14)	−0.0206 (16)
C3	0.0479 (19)	0.0440 (18)	0.0411 (19)	0.0029 (15)	−0.0038 (14)	−0.0195 (15)
C4	0.0456 (19)	0.0450 (19)	0.048 (2)	−0.0002 (15)	−0.0002 (15)	−0.0204 (16)
C5	0.068 (2)	0.068 (2)	0.064 (3)	0.0107 (19)	−0.0070 (19)	−0.044 (2)
C6	0.077 (3)	0.095 (3)	0.069 (3)	0.009 (2)	−0.008 (2)	−0.057 (3)
C7	0.073 (3)	0.089 (3)	0.052 (2)	0.003 (2)	−0.0072 (19)	−0.038 (2)
C8	0.059 (2)	0.055 (2)	0.043 (2)	0.0090 (17)	−0.0107 (17)	−0.0171 (18)
C9	0.0392 (18)	0.0492 (19)	0.045 (2)	−0.0027 (14)	0.0019 (15)	−0.0268 (16)
C10	0.0482 (19)	0.0462 (19)	0.043 (2)	0.0013 (15)	−0.0035 (15)	−0.0238 (16)
C11	0.051 (2)	0.066 (2)	0.050 (2)	0.0075 (17)	−0.0159 (17)	−0.0329 (19)
C12	0.056 (2)	0.058 (2)	0.046 (2)	0.0043 (17)	−0.0090 (17)	−0.0274 (18)
C13	0.078 (3)	0.079 (3)	0.054 (2)	0.018 (2)	−0.0136 (19)	−0.036 (2)

## Geometric parameters (Å, °)

O1—C1	1.220 (3)	C7—H7	0.9300
O2—C4	1.236 (3)	C8—C7	1.378 (4)
O3—C2	1.339 (3)	C8—H8	0.9300
O3—C11	1.447 (3)	C9—C8	1.394 (4)
C1—C2	1.499 (4)	C10—C9	1.393 (4)
C1—C9	1.481 (4)	C10—C4	1.478 (4)
C2—C3	1.339 (3)	C10—C5	1.382 (4)
C3—H3	0.9300	C11—H11A	0.9700
C4—C3	1.449 (4)	C11—H11B	0.9700
C5—H5	0.9300	C12—C11	1.454 (4)
C5—C6	1.389 (5)	C12—C13	1.164 (4)
C6—H6	0.9300	C13—H13	0.9300
C6—C7	1.369 (4)		
O1—C1—C2	120.7 (3)	C6—C5—H5	120.1
O1—C1—C9	121.8 (3)	C7—C8—C9	119.7 (3)
O3—C2—C1	111.2 (2)	C7—C8—H8	120.1
O3—C11—C12	107.6 (2)	C7—C6—C5	120.2 (3)
O3—C11—H11A	110.2	C7—C6—H6	119.9
O3—C11—H11B	110.2	C8—C7—H7	119.7
O2—C4—C10	121.0 (3)	C8—C9—C1	120.2 (3)
O2—C4—C3	120.2 (3)	C9—C8—H8	120.1
C2—C3—C4	122.2 (3)	C9—C10—C4	120.3 (3)
C2—O3—C11	117.2 (2)	C9—C1—C2	117.5 (3)
C2—C3—H3	118.9	C10—C9—C1	120.1 (3)
C3—C2—O3	127.8 (3)	C10—C9—C8	119.7 (3)
C3—C2—C1	121.0 (3)	C10—C5—H5	120.1
C3—C4—C10	118.7 (3)	C10—C5—C6	119.9 (3)
C4—C3—H3	118.9	H11A—C11—H11B	108.5
C5—C10—C9	119.8 (3)	C12—C11—H11A	110.2
C5—C10—C4	119.9 (3)	C12—C11—H11B	110.2
C5—C6—H6	119.9	C12—C13—H13	180.0
C6—C7—C8	120.6 (4)	C13—C12—C11	177.6 (3)
C6—C7—H7	119.7		
O1—C1—C2—O3	-1.3 (4)	C5—C10—C9—C1	177.0 (3)
O1—C1—C2—C3	178.0 (3)	C5—C10—C9—C8	-2.0 (5)
O1—C1—C9—C10	-175.2 (3)	C5—C10—C4—O2	0.8 (5)
O1—C1—C9—C8	3.8 (5)	C5—C10—C4—C3	179.7 (3)
O2—C4—C3—C2	-178.8 (3)	C5—C6—C7—C8	-1.0 (6)
O3—C2—C3—C4	177.5 (3)	C9—C1—C2—O3	179.1 (3)
C1—C2—C3—C4	-1.7 (5)	C9—C1—C2—C3	-1.5 (5)
C1—C9—C8—C7	-178.5 (3)	C9—C10—C5—C6	1.9 (5)
C2—O3—C11—C12	-171.9 (3)	C9—C8—C7—C6	1.0 (6)
C2—C1—C9—C10	4.4 (5)	C10—C9—C8—C7	0.5 (5)
C2—C1—C9—C8	-176.6 (3)	C10—C4—C3—C2	2.3 (5)

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C4—C10—C9—C1	-3.9 (5)	C10—C5—C6—C7	-0.5 (6)
C4—C10—C9—C8	177.1 (3)	C11—O3—C2—C1	-177.1 (3)
C4—C10—C5—C6	-177.1 (3)	C11—O3—C2—C3	3.7 (5)

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