3-(3-Nitrophenyl)-1-[4-(prop-2-ynyloxy)phenyl]-prop-2-en-1-one

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The structure of the title compound, C_{18}H_{13}NO_{4}, shows that the whole molecule is almost planar but with a dihedral angle between the two phenyl rings of 19.22 (5)°. The molecules are linked by C—H···O interactions, forming sheets in the (211) plane.

Structure description

Chalcones are among the leading bioactive flavonoids, with a therapeutic potential implicated to an array of bioactivities that have been investigated by a series of pre-clinical and clinical studies. They contain an α-β unsaturated carbonyl system, which is present in open-chain form, and two aromatic rings are joined through three-carbon atoms (Kozlowski et al., 2007; Raghav & Garg, 2014). Studies depicting the biological activities of chalcones and their derivatives describe their immense significance as anti-diabetic, anticancer, anti-inflammatory, antimicrobial, antioxidant, antiparasitic, psychoactive and neuroprotective agents, and their antioxidant and enzyme inhibitory activities (Lin et al., 2002; Bhat et al., 2005; Trivedi et al., 2007; Lahtchev et al., 2008; Aneja et al., 2018).

Chalcone as a privileged structure in medicinal chemistry has been reviewed by Zhuang et al. (2017). A comprehensive review of chalcone derivatives as antileishmanial agents has also been published (de Mello et al., 2018). The crystal structures of (2E)-1-(4-methylphenyl)-3-(4-nitrophenyl)prop-2-en-1-one (Butcher et al., 2007), (2E)-1-(3-bromophenyl)-3-(4,5-dimethoxy-2-nitrophenyl)prop-2-en-1-one (Jasinski et al., 2010), (2E)-3-(3-nitrophenyl)-1-[4-(piperidin-1-yl)phenyl]prop-2-en-1-one (Fun et al., 2012) and
The present work describes the synthesis and crystal structure of the title compound 3-(3-nitrophenyl)-1-[4-(prop-2-yn-1-yloxy)phenyl]prop-2-en-1-one, which crystallizes in the triclinic space group $P\overline{1}$ with one molecule in the asymmetric unit. It consists both a 3-nitrophenyl group and a (prop-2-yn-1-yloxy)benzene group linked by the chalcone is 19.22 (5)$^\circ$.

The molecules are linked by $C$–$H$⋯$O$ interactions (Table 1), which form sheets in the (211) plane as shown in Fig. 2. There are no $\pi$–$\pi$ interactions between the phenyl rings.

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

|            | $D$–$H$⋯$A$ | $D$–$H$ | $H$–$A$ | $D$⋯$A$ | $D$–$H$⋯$A$ |
|-----------|------------|---------|---------|--------|-------------|
| C18–H18A–O2 | 0.901 (18) | 2.519 (18) | 3.3927 (18) | 163.6 (15) |

Symmetry code: (i) $x - 1, y + 2, z$.  

### Table 2
Experimental details.

| Crystal data | Chemical formula | $C_{18}H_{13}NO_4$ |
|-------------|----------------|-------------------|
| $M_r$       |                | 307.29            |
| Crystal system, space group | Triclinic, $P\overline{1}$ |
| Temperature (K) | 100 |
| $a$, $b$, $c$ (Å) | 7.6534 (16), 8.6079 (15), 11.369 (2) |
| $\alpha$, $\beta$, $\gamma$ (°) | 94.433 (7), 97.953 (8), 97.019 (7) |
| $V$ (Å³) | 732.8 (3) |
| No. of measured, independent and observed $|F| > 2\sigma(|F|)$ reflections | 47166, 3638, 2700 |
| $R_{	ext{int}}$, $\sigma(\beta)_{\text{max}}$ (Å$^{-1}$) | 0.089, 0.667 |
| No. of reflections | 3638 |
| No. of parameters | 212 |
| H-atom treatment | H-atoms treated by a mixture of independent and constrained refinement |
| $\Delta R_{	ext{max}}$, $\Delta R_{	ext{min}}$ (e Å$^{-3}$) | 0.28, −0.21 |

Computer programs: APEX2 and $\text{SAINT}$ (Bruker, 2016), $\text{SHELXTL}$ (Sheldrick, 2015a), $\text{SHELXL}2016/5$ (Sheldrick, 2015b) and $\text{SHELXL}$ (Sheldrick, 2008).

### Synthesis and crystallization
A well-stirred solution of 1-[4-(prop-2-yn-1-yloxy)phenyl]ethanone (1 g, 1 mmol) in 20 ml of ethanol was added slowly to alcoholic potassium hydroxide (0.48 g, 1.5 mmol). To this solution, $m$-nitro benzoaldehyde (1.03 g, 1.2 mmol) was added. The resulting mixture was stirred at room temperature for 30 min. Then, the separated solid from the reaction mixture was filtered, washed with cold water, dried and recrystallized from ethanol:dimethylformamide mixture (9:1). Golden yellow crystals (yield: 86%, m.p. 453–454 K). The reaction scheme is shown in Fig. 3. FT–IR: $\nu_{\text{max}}$, cm$^{-1}$ (KBr): 2987 (C–H aliphatic), 2117 (C≡C str), 1650 (C≡O), 1518 (asym NO$_2$ stretch), 1444 (sym NO$_2$ stretch), 1252 (C–O stretch). $^1$H NMR (400 MHz, CDC$_3$, δ p.p.m.): 7.55 (d, 1H, $J = 15.7$ Hz, olefinic-β), 7.36 (d, 2H, $J = 8.8$ Hz, Ar–H), 7.28 (d, 2H, $J = 8.6$ Hz, Ar–H), 7.16 (d, 2H, $J = 8.8$ Hz, Ar–H), 7.09 (d, 2H, $J = 8.3$ Hz, Ar–H), 6.73 (d, 1H, $J = 15.7$ Hz, olefinic-α), 4.46 (s, 2H, O–CH$_2$), 2.79 (s, 1H, acetylene proton).
Refinement

Crystal data, data collection and structure refinement details for the title compound are summarized in Table 2.

Acknowledgements

V is grateful to the DST–PURSE Project, Vignan Bhavana, UOM, for providing research facilities.

Funding information

HSY and BK are grateful to UGC, New Delhi, for the award of BSR Faculty Fellowship.

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3-(3-Nitrophenyl)-1-[4-(prop-2-ynyloxy)phenyl]prop-2-en-1-one

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Crystal data

$\text{C}_{18}\text{H}_{13}\text{NO}_4$

$M_r = 307.29$

Triclinic, $P\overline{1}$

$a = 7.6534$ (16) Å

$b = 8.6079$ (15) Å

$c = 11.369$ (2) Å

$\alpha = 94.433$ (7)°

$\beta = 97.953$ (8)°

$\gamma = 97.019$ (7)°

$V = 732.8$ (3) Å$^3$

$Z = 2$

$F(000) = 320$

$D_a = 1.393$ Mg m$^{-3}$

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

Cell parameters from 9976 reflections

$\theta = 2.7$–32.9°

$\mu = 0.10$ mm$^{-1}$

$T = 100$ K

Prism, yellow

0.33 × 0.19 × 0.14 mm

Data collection

Bruker APEXII CCD diffractometer

$\phi$ and $\omega$ scans

Absorption correction: multi-scan (SADABS; Bruker, 2016)

$T_{\text{min}} = 0.634$, $T_{\text{max}} = 0.729$

47166 measured reflections

3638 independent reflections

2700 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.089$

$\theta_{\text{max}} = 28.3$°, $\theta_{\text{min}} = 2.4$°

$h = -10$→10

$k = -11$→10

$l = -15$→15

Refinement

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.046$

$wR(F^2) = 0.137$

$S = 1.08$

3638 reflections

212 parameters

0 restraints

Primary atom site location: dual

Secondary atom site location: difference Fourier map

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F^2) + (0.0642P)^2 + 0.1082P]$

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

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

$\Delta \rho_{\text{max}} = 0.28$ e Å$^{-3}$

$\Delta \rho_{\text{min}} = -0.21$ e Å$^{-3}$

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.
**Refinement.** The acetylenic H atom was freely refined. All remaining hydrogen atoms were placed geometrically and refined as riding atoms with their $U_{iso}$ values 1.2 times that of their attached atoms.

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

|      | x     | y     | z     | $U_{iso}$/$U_{eq}$ |
|------|-------|-------|-------|--------------------|
| O1   | 0.8224 (2) | −0.53179 (13) | 0.40582 (11) | 0.0732 (4) |
| O2   | 0.78869 (16) | −0.48667 (12) | 0.22192 (10) | 0.0557 (3) |
| O3   | 0.38666 (16) | 0.19330 (12) | 0.03294 (9) | 0.0557 (3) |
| O4   | 0.12863 (14) | 0.82184 (10) | 0.22678 (8) | 0.0419 (3) |
| N1   | 0.79204 (16) | −0.44535 (13) | 0.32710 (11) | 0.0436 (3) |
| C1   | 0.63484 (18) | −0.05300 (15) | 0.30785 (12) | 0.0358 (3) |
| C2   | 0.67213 (17) | −0.20372 (14) | 0.27518 (11) | 0.0342 (3) |
| H2A  | 0.639784 | −0.249615 | 0.195197 | 0.041* |
| C3   | 0.75705 (18) | −0.28509 (15) | 0.36138 (12) | 0.0362 (3) |
| C4   | 0.8101 (2) | −0.22355 (17) | 0.47820 (12) | 0.0446 (3) |
| H4A  | 0.868961 | −0.282414 | 0.535011 | 0.054* |
| C5   | 0.7751 (2) | −0.07331 (18) | 0.51018 (13) | 0.0518 (4) |
| C6   | 0.811026 | −0.027252 | 0.589874 | 0.062* |
| H6A  | 0.6882 (2) | 0.00972 (17) | 0.42650 (13) | 0.0469 (4) |
| H7A  | 0.663892 | 0.112233 | 0.450078 | 0.056* |
| C7   | 0.54529 (18) | 0.03375 (15) | 0.21647 (12) | 0.0378 (3) |
| C8   | 0.524932 | −0.013883 | 0.136707 | 0.045* |
| H8A  | 0.49007 (19) | 0.17290 (15) | 0.23512 (12) | 0.0402 (3) |
| C9   | 0.505167 | 0.222444 | 0.314155 | 0.048* |
| C10  | 0.40553 (19) | 0.25253 (15) | 0.13558 (12) | 0.0386 (3) |
| C11  | 0.34071 (18) | 0.40617 (14) | 0.16303 (11) | 0.0345 (3) |
| C12  | 0.32576 (18) | 0.46816 (15) | 0.27815 (11) | 0.0366 (3) |
| H11A | 0.365827 | 0.414762 | 0.344742 | 0.044* |
| H12A | 0.25357 (19) | 0.60589 (15) | 0.29624 (11) | 0.0382 (3) |
| H13A | 0.242477 | 0.645757 | 0.374749 | 0.046* |
| C13  | 0.19719 (17) | 0.68610 (14) | 0.19960 (11) | 0.0343 (3) |
| C14  | 0.2132 (2) | 0.62771 (16) | 0.08447 (12) | 0.0418 (3) |
| H14A | 0.176033 | 0.682647 | 0.018101 | 0.050* |
| C15  | 0.2840 (2) | 0.48861 (16) | 0.06802 (12) | 0.0418 (3) |
| H15A | 0.293985 | 0.448410 | −0.010617 | 0.050* |
| C16  | 0.0652 (2) | 0.90385 (15) | 0.12751 (12) | 0.0412 (3) |
| H16A | −0.037244 | 0.838377 | 0.076985 | 0.049* |
| H16B | 0.160640 | 0.926181 | 0.078194 | 0.049* |
| C17  | 0.01114 (18) | 1.05106 (15) | 0.17380 (12) | 0.0397 (3) |
| C18  | −0.0334 (2) | 1.17155 (17) | 0.20482 (14) | 0.0466 (4) |
| H18A | −0.067 (2) | 1.266 (2) | 0.2248 (15) | 0.057 (5)* |

**Atomic displacement parameters ($\AA^2$)**

|      | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|------|----------|----------|----------|----------|----------|----------|
| O1   | 0.1189 (11) | 0.0461 (6) | 0.0560 (7) | 0.0324 (7) | −0.0072 (7) | 0.0173 (5) |
| O2   | 0.0791 (8) | 0.0448 (6) | 0.0449 (6) | 0.0280 (5) | 0.0012 (5) | −0.0014 (5) |
|   | x     | y     | z     | dx    | dy    | dz    |
|---|-------|-------|-------|-------|-------|-------|
| O3 | 0.0887 (9) | 0.0452 (6) | 0.0366 (6) | 0.0323 (6) | 0.0041 (5) | -0.0009 (4) |
| O4 | 0.0605 (6) | 0.0339 (5) | 0.0339 (5) | 0.0210 (4) | 0.0040 (4) | 0.0022 (4) |
| N1 | 0.0506 (7) | 0.0363 (6) | 0.0440 (7) | 0.0146 (5) | -0.0018 (5) | 0.0069 (5) |
| C1 | 0.0395 (7) | 0.0357 (7) | 0.0357 (7) | 0.0101 (7) | 0.0041 (7) | 0.0022 (7) |
| C2 | 0.0507 (7) | 0.0316 (6) | 0.0087 (5) | 0.0027 (5) | 0.0031 (5) | 0.0026 (5) |
| C3 | 0.0391 (7) | 0.0317 (6) | 0.0102 (5) | 0.0052 (5) | 0.0027 (5) | 0.0063 (5) |
| C4 | 0.0465 (8) | 0.0352 (7) | 0.0153 (6) | 0.0012 (6) | 0.0090 (6) | 0.0090 (6) |
| C5 | 0.0591 (10) | 0.0320 (7) | 0.0183 (7) | -0.0018 (7) | -0.0026 (6) | 0.0078 (6) |
| C6 | 0.0609 (9) | 0.0388 (7) | 0.0173 (7) | 0.0026 (6) | 0.0032 (6) | 0.0021 (6) |
| C7 | 0.0457 (7) | 0.0339 (6) | 0.0121 (5) | 0.0040 (5) | 0.0016 (5) | 0.0016 (5) |
| C8 | 0.0514 (8) | 0.0359 (7) | 0.0148 (6) | 0.0025 (6) | 0.0014 (5) | 0.0014 (5) |
| C9 | 0.0484 (8) | 0.0366 (7) | 0.0130 (5) | 0.0061 (6) | 0.0021 (5) | 0.0021 (5) |
| C10 | 0.0406 (7) | 0.0339 (6) | 0.0096 (5) | 0.0038 (5) | 0.0025 (5) | 0.0025 (5) |
| C11 | 0.0472 (7) | 0.0317 (6) | 0.0115 (5) | 0.0037 (5) | 0.0066 (5) | 0.0066 (5) |
| C12 | 0.0514 (8) | 0.0299 (6) | 0.0109 (6) | 0.0064 (5) | 0.0014 (5) | 0.0014 (5) |
| C13 | 0.0396 (7) | 0.0346 (5) | 0.0090 (5) | 0.0033 (5) | 0.0007 (5) | 0.0007 (5) |
| C14 | 0.0596 (9) | 0.0307 (6) | 0.0188 (6) | 0.0018 (6) | 0.0047 (5) | 0.0047 (5) |
| C15 | 0.0603 (9) | 0.0306 (5) | 0.0177 (6) | 0.0044 (6) | 0.0011 (5) | 0.0011 (5) |
| C16 | 0.0526 (8) | 0.0368 (7) | 0.0160 (6) | -0.0016 (6) | 0.0028 (5) | 0.0028 (5) |
| C17 | 0.0429 (7) | 0.0408 (7) | 0.0107 (6) | 0.0019 (6) | 0.0062 (5) | 0.0062 (5) |
| C18 | 0.0518 (9) | 0.0527 (9) | 0.0170 (6) | 0.0063 (7) | 0.0052 (6) | 0.0052 (6) |

**Geometric parameters (Å, °)**

| Bond/Angle | Distance (Å) | Angle (°) |
|------------|--------------|-----------|
| O1—N1      | 1.2232 (15)  |           |
| O2—N1      | 1.2168 (16)  | 1.4815 (18) |
| O3—C9      | 1.2189 (16)  | 0.9500    |
| O4—C13     | 1.3693 (14)  | 1.4940 (17) |
| O4—C16     | 1.4362 (15)  | 1.3866 (17) |
| N1—C3      | 1.4763 (17)  | 1.3997 (18) |
| C1—C2      | 1.3957 (17)  | 1.3809 (17) |
| C1—C6      | 1.3996 (19)  | 0.9500    |
| C1—C7      | 1.4680 (18)  | 1.3888 (17) |
| C2—C3      | 1.3838 (17)  | 0.9500    |
| C2—H2A     | 0.9500       | 1.3924 (18) |
| C3—C4      | 1.3780 (19)  | 1.3835 (18) |
| C4—C5      | 1.384 (2)    | 0.9500    |
| C4—H4A     | 0.9500       | 1.4627 (18) |
| C5—C6      | 1.380 (2)    | 0.9900    |
| C5—H5A     | 0.9500       | 0.9900    |
| C6—H6A     | 0.9500       | 1.1746 (19) |
| C7—C8      | 1.3295 (17)  | 0.901 (18) |
| C7—H7A     | 0.9500       | 0.9500    |

IUCrData (2022). 7, x220957 data-3
| Bond                  | Angle (Deg)       | Bond                  | Angle (Deg)       |
|----------------------|-------------------|----------------------|-------------------|
| C2—C1—C6            | 118.12 (12)       | C11—C10—C9          | 124.01 (11)       |
| C2—C1—C7            | 118.90 (11)       | C12—C11—C10         | 120.89 (11)       |
| C6—C1—C7            | 122.97 (12)       | C12—C11—H11A        | 119.6             |
| C3—C2—C1            | 118.80 (12)       | C10—C11—H11A        | 119.6             |
| C3—C2—H2A           | 120.6             | C11—C12—C13         | 119.98 (12)       |
| C1—C2—H2A           | 120.6             | C11—C12—H12A        | 120.0             |
| C4—C3—C2            | 123.22 (12)       | C13—C12—H12A        | 120.0             |
| C4—C3—N1            | 118.22 (11)       | O4—C13—C12          | 115.58 (11)       |
| C2—C3—N1            | 118.56 (11)       | O4—C13—C14          | 124.35 (11)       |
| C3—C4—C5            | 117.94 (12)       | C12—C13—C14         | 120.07 (11)       |
| C3—C4—H4A           | 121.0             | C15—C14—C13         | 119.11 (12)       |
| C5—C4—H4A           | 121.0             | C15—C14—H14A        | 120.4             |
| C6—C5—C4            | 120.11 (13)       | C13—C14—H14A        | 120.4             |
| C6—C5—H5A           | 119.9             | C14—C15—C10         | 121.89 (12)       |
| C4—C5—H5A           | 119.9             | C14—C15—H15A        | 119.1             |
| C5—C6—C1            | 121.80 (13)       | C10—C15—H15A        | 119.1             |
| C5—C6—H6A           | 119.1             | O4—C16—C17          | 108.45 (11)       |
| C1—C6—H6A           | 119.1             | O4—C16—H16A         | 110.0             |
| C8—C7—C1            | 125.96 (12)       | C17—C16—H16A        | 110.0             |
| C8—C7—H7A           | 117.0             | O4—C16—H16B         | 110.0             |
| C1—C7—H7A           | 117.0             | C17—C16—H16B        | 110.0             |
| C7—C8—C9            | 121.55 (12)       | H16A—C16—H16B       | 108.4             |
| C7—C8—H8A           | 119.2             | C18—C17—C16         | 176.40 (14)       |
| C9—C8—H8A           | 119.2             | C17—C18—H18A        | 177.0 (11)        |
| O3—C9—C8            | 120.87 (12)       | C7—C8—C9—C10        | 177.91 (13)       |
| C7—C1—C2—C3         | 0.0 (2)           | O3—C9—C10—C15       | -9.4 (2)          |
| C7—C1—C2—C3         | -0.3 (2)          | C8—C9—C10—C15       | 171.57 (13)       |
| C1—C2—C3—N1         | 178.51 (11)       | O3—C9—C10—C11       | 167.42 (14)       |
| O2—N1—C3—C4         | -161.56 (14)      | C8—C9—C10—C11       | -11.6 (2)         |
| O1—N1—C3—C4         | 18.8 (2)          | C15—C10—C11—C12    | 1.2 (2)           |
| O2—N1—C3—C2         | 18.64 (19)        | C9—C10—C11—C12     | -175.62 (13)      |
| O1—N1—C3—C2         | -160.96 (13)      | C10—C11—C12—C13    | -1.0 (2)          |
| C2—C3—C4—C5         | 0.5 (2)           | C16—O4—C13—C12     | -178.35 (11)      |
| N1—C3—C4—C5         | -179.33 (13)      | C16—O4—C13—C14     | 2.0 (2)           |
| C3—C4—C5—C6         | 0.6 (2)           | C11—C12—C13—O4     | -179.60 (12)      |
| C4—C5—C6—C1         | -0.7 (3)          | C11—C12—C13—C14    | 0.1 (2)           |
| C2—C1—C6—C5         | -0.1 (2)          | O4—C13—C14—C15     | -179.70 (13)      |
| C7—C1—C6—C5         | -178.78 (15)      | C12—C13—C14—C15    | 0.7 (2)           |
| C2—C1—C7—C8         | 175.05 (13)       | C13—C14—C15—C10    | -0.5 (2)          |
| C6—C1—C7—C8         | -6.3 (2)          | C11—C10—C15—C14    | -0.4 (2)          |
| C1—C7—C8—C9         | 178.18 (13)       | C9—C10—C15—C14     | 176.58 (13)       |
| C7—C8—C9—O3         | -1.1 (2)          | C13—O4—C16—C17     | -175.40 (11)      |
Hydrogen-bond geometry (Å, °)

| D—H···A     | D—H | H···A | D···A    | D—H···A |
|-------------|------|-------|----------|---------|
| C18—H18A···O2i | 0.901 (18) | 2.519 (18) | 3.3927 (18) | 163.6 (15) |

Symmetry code: (i) x−1, y+2, z.