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

Vinaya,a Yeriyr B. Basavaraju,a Holalagudu A. Nagma Banu,b Balakrishna Kalluraya,b Himmige S. Yathirajan,a* Rishik Baleraoc and Ray J. Butcherd

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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){\degree}. 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 antidiabetic, 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 (Fig. 1), 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 to a central chalcone moiety. Even though the C—N bond length is 1.4706 (17) Å and thus single, the nitro group is almost coplanar with its phenyl ring [dihedral angle of 18.94 (6)$^\circ$] as a result of the steric clash between O1 and H4 and between O2 and H2, respectively. The chalcone group is planar (average deviation from plane of 0.004 Å) and makes dihedral angles of 7.69 (8) and 10.96 (6)$^\circ$ with the 3-nitrophenyl ring and the phenyl ring of the (prop-2-yn-1-yloxy)benzene group, respectively. Lastly, the twist between the two phenyl rings which are 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 π–π interactions between the phenyl rings.

**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 one (1 g, 1 mmol) in 20 ml of ethanol was added slowly to alcoholic potassium hydroxide (0.48 g, 1.5 mmol). The reaction 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, CDC13, $\delta$ 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).

**Table 1**

<table>
<thead>
<tr>
<th>Hydrogen-bond geometry ($\overline{\AA}, ^\circ$).</th>
</tr>
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<tbody>
<tr>
<td>$D$–H···$A$</td>
</tr>
<tr>
<td>---------------</td>
</tr>
<tr>
<td>C18–H18A···O2</td>
</tr>
</tbody>
</table>

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

4'-dimethylamino-3-nitrochalcone, 3-dimethylamino-3'-nitrochalcone and 3'-nitrochalcone (Hall et al., 2020) have been reported.

![Diagram of molecules showing the atom-labelling scheme. Atomic displacement parameters are at the 30% probability level.](image1)

![Packing diagram for the title compound showing the C–H···O interactions linking the molecules into sheets in the (211) plane.](image2)

![Reaction scheme for the synthesis of the title compound.](image3)

**Table 2**

<table>
<thead>
<tr>
<th>Crystal data</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical formula</td>
</tr>
<tr>
<td>$M_r$</td>
</tr>
<tr>
<td>Crystal system, space group</td>
</tr>
<tr>
<td>Temperature (K)</td>
</tr>
<tr>
<td>$a$, $b$, $c$ (Å)</td>
</tr>
<tr>
<td>$\alpha$, $\beta$, $\gamma$ (°)</td>
</tr>
<tr>
<td>$V$ (Å$^3$)</td>
</tr>
<tr>
<td>No. of measured, independent and observed $</td>
</tr>
<tr>
<td>$R_{	ext{int}}$, ($\sin \theta/\lambda)_{\text{max}}$ (Å$^{-1}$)</td>
</tr>
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</table>

<table>
<thead>
<tr>
<th>Data collection</th>
</tr>
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<tr>
<td>Diffractometer</td>
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<tr>
<td>Absorption correction</td>
</tr>
<tr>
<td>$\overline{\mu}$ (e Å$^{-1}$)</td>
</tr>
<tr>
<td>No. of reflections</td>
</tr>
<tr>
<td>No. of parameters</td>
</tr>
<tr>
<td>H-atom treatment</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Refinement</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\overline{R}(F^2 &gt; 2\sigma(F^2))$, $wR(F^2)$, $S$</td>
</tr>
<tr>
<td>$\Delta$ρ$<em>{\text{max}}$, $\Delta$ρ$</em>{\text{min}}$ (e Å$^{-3}$)</td>
</tr>
</tbody>
</table>

Computer programs: APEX2 and SAINT (Bruker, 2016), SHELXTL (Sheldrick, 2008).
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.

References


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

Crystal data

C_{18}H_{13}NO_{4}  
Mr = 307.29
Triclinic, P\bar{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) Å³

Z = 2  
F(000) = 320  
\(D_{\text{x}} = 1.393\) Mg m⁻³  
Mo Ka 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  
\(R[F^2 > 2\sigma(F^2)] = 0.046\)  
w\(R(F^2) = 0.137\)  
\(S = 1.08\)

3638 reflections  
212 parameters  
0 restraints

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 $U_{	ext{iso}}$ values 1.2 times that of their attached atoms.

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

<table>
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<th></th>
<th>$x$</th>
<th>$y$</th>
<th>$z$</th>
<th>$U_{	ext{iso}}/U_{eq}$</th>
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<td>O1</td>
<td>0.8224 (2)</td>
<td>−0.53179 (13)</td>
<td>0.40582 (11)</td>
<td>0.0732 (4)</td>
</tr>
<tr>
<td>O2</td>
<td>0.78869 (16)</td>
<td>−0.48667 (12)</td>
<td>0.22192 (10)</td>
<td>0.0557 (3)</td>
</tr>
<tr>
<td>O3</td>
<td>0.38666 (16)</td>
<td>0.19330 (12)</td>
<td>0.03294 (9)</td>
<td>0.0557 (3)</td>
</tr>
<tr>
<td>O4</td>
<td>0.12863 (14)</td>
<td>0.82184 (10)</td>
<td>0.22678 (8)</td>
<td>0.0419 (3)</td>
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<tr>
<td>N1</td>
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<td>−0.44535 (13)</td>
<td>0.32710 (11)</td>
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</tr>
<tr>
<td>C1</td>
<td>0.63484 (18)</td>
<td>−0.05300 (15)</td>
<td>0.30785 (12)</td>
<td>0.0358 (3)</td>
</tr>
<tr>
<td>C2</td>
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<td>−0.20372 (14)</td>
<td>0.27518 (11)</td>
<td>0.0342 (3)</td>
</tr>
<tr>
<td>H2A</td>
<td>0.639784</td>
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<td>0.195197</td>
<td>0.041*</td>
</tr>
<tr>
<td>C3</td>
<td>0.75705 (18)</td>
<td>−0.28509 (15)</td>
<td>0.36138 (12)</td>
<td>0.0362 (3)</td>
</tr>
<tr>
<td>C4</td>
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<tr>
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</tr>
<tr>
<td>C5</td>
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<td>−0.07331 (18)</td>
<td>0.51018 (13)</td>
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</tr>
<tr>
<td>H5A</td>
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<td>0.589874</td>
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</tr>
<tr>
<td>C6</td>
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<td>0.00972 (17)</td>
<td>0.42650 (13)</td>
<td>0.0469 (4)</td>
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<tr>
<td>H6A</td>
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<td>0.112233</td>
<td>0.450078</td>
<td>0.056*</td>
</tr>
<tr>
<td>C7</td>
<td>0.54529 (18)</td>
<td>0.03375 (15)</td>
<td>0.21647 (12)</td>
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<tr>
<td>H7A</td>
<td>0.524932</td>
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</tr>
<tr>
<td>C8</td>
<td>0.49007 (19)</td>
<td>0.17290 (15)</td>
<td>0.23512 (12)</td>
<td>0.0402 (3)</td>
</tr>
<tr>
<td>H8A</td>
<td>0.505167</td>
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<td>0.314155</td>
<td>0.048*</td>
</tr>
<tr>
<td>C9</td>
<td>0.40553 (19)</td>
<td>0.25253 (15)</td>
<td>0.13558 (12)</td>
<td>0.0386 (3)</td>
</tr>
<tr>
<td>C10</td>
<td>0.34071 (18)</td>
<td>0.40617 (14)</td>
<td>0.16303 (11)</td>
<td>0.0345 (3)</td>
</tr>
<tr>
<td>C11</td>
<td>0.32576 (18)</td>
<td>0.46816 (15)</td>
<td>0.27815 (11)</td>
<td>0.0366 (3)</td>
</tr>
<tr>
<td>H11A</td>
<td>0.365827</td>
<td>0.414762</td>
<td>0.344742</td>
<td>0.044*</td>
</tr>
<tr>
<td>C12</td>
<td>0.25357 (19)</td>
<td>0.60589 (15)</td>
<td>0.29624 (11)</td>
<td>0.0382 (3)</td>
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<tr>
<td>H12A</td>
<td>0.242477</td>
<td>0.645757</td>
<td>0.374749</td>
<td>0.046*</td>
</tr>
<tr>
<td>C13</td>
<td>0.19719 (17)</td>
<td>0.68610 (14)</td>
<td>0.19960 (11)</td>
<td>0.0343 (3)</td>
</tr>
<tr>
<td>C14</td>
<td>0.2132 (2)</td>
<td>0.62771 (16)</td>
<td>0.08447 (12)</td>
<td>0.0418 (3)</td>
</tr>
<tr>
<td>H14A</td>
<td>0.176033</td>
<td>0.682647</td>
<td>0.018101</td>
<td>0.050*</td>
</tr>
<tr>
<td>C15</td>
<td>0.2840 (2)</td>
<td>0.48861 (16)</td>
<td>0.06802 (12)</td>
<td>0.0418 (3)</td>
</tr>
<tr>
<td>H15A</td>
<td>0.293985</td>
<td>0.448410</td>
<td>−0.010617</td>
<td>0.050*</td>
</tr>
<tr>
<td>C16</td>
<td>0.0652 (2)</td>
<td>0.90385 (15)</td>
<td>0.12751 (12)</td>
<td>0.0412 (3)</td>
</tr>
<tr>
<td>H16A</td>
<td>−0.037244</td>
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<td>0.076985</td>
<td>0.049*</td>
</tr>
<tr>
<td>H16B</td>
<td>0.160640</td>
<td>0.926181</td>
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<td>0.049*</td>
</tr>
<tr>
<td>C17</td>
<td>0.01114 (18)</td>
<td>1.05106 (15)</td>
<td>0.17380 (12)</td>
<td>0.0397 (3)</td>
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<tr>
<td>C18</td>
<td>−0.0334 (2)</td>
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<td>0.20482 (14)</td>
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<tr>
<td>H18A</td>
<td>−0.067 (2)</td>
<td>1.266 (2)</td>
<td>0.2248 (15)</td>
<td>0.057 (5)*</td>
</tr>
</tbody>
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**Atomic displacement parameters ($\AA^2$)**

<table>
<thead>
<tr>
<th></th>
<th>$U_{11}$</th>
<th>$U_{22}$</th>
<th>$U_{33}$</th>
<th>$U_{12}$</th>
<th>$U_{13}$</th>
<th>$U_{23}$</th>
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<tbody>
<tr>
<td>O1</td>
<td>0.1189 (11)</td>
<td>0.0461 (6)</td>
<td>0.0560 (7)</td>
<td>0.0324 (7)</td>
<td>−0.0072 (7)</td>
<td>0.0173 (5)</td>
</tr>
<tr>
<td>O2</td>
<td>0.0791 (8)</td>
<td>0.0448 (6)</td>
<td>0.0449 (6)</td>
<td>0.0280 (5)</td>
<td>0.0012 (5)</td>
<td>−0.0014 (5)</td>
</tr>
</tbody>
</table>
**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)  0.0333 (6)  0.0357 (7)  0.0103 (5)  0.0049 (5)  0.0040 (5)  
**C2**  0.0384 (7)  0.0328 (6)  0.0316 (6)  0.0087 (5)  0.0027 (5)  0.0026 (5)  
**C3**  0.0397 (7)  0.0333 (6)  0.0372 (7)  0.0102 (5)  0.0052 (5)  0.0063 (5)  
**C4**  0.0535 (8)  0.0465 (8)  0.0352 (7)  0.0153 (6)  0.0012 (6)  0.0090 (6)  
**C5**  0.0691 (10)  0.0530 (9)  0.0320 (7)  0.0183 (7)  −0.0018 (7)  −0.0026 (6)  
**C6**  0.0633 (9)  0.0391 (7)  0.0388 (7)  0.0179 (7)  0.0032 (6)  −0.0021 (6)  
**C7**  0.0457 (7)  0.0333 (6)  0.0389 (7)  0.0121 (5)  0.0040 (5)  0.0016 (5)  
**C8**  0.0541 (8)  0.0340 (7)  0.0359 (7)  0.0148 (6)  0.0025 (6)  0.0014 (5)  
**C9**  0.0484 (8)  0.0324 (6)  0.0366 (7)  0.0130 (5)  0.0061 (6)  0.0021 (5)  
**C10**  0.0406 (7)  0.0296 (6)  0.0339 (6)  0.0096 (5)  0.0038 (5)  0.0025 (5)  
**C11**  0.0472 (7)  0.0324 (6)  0.0317 (6)  0.0115 (5)  0.0037 (5)  0.0066 (5)  
**C12**  0.0514 (8)  0.0343 (6)  0.0299 (6)  0.0109 (6)  0.0064 (5)  0.0014 (5)  
**C13**  0.0396 (7)  0.0281 (6)  0.0352 (6)  0.0090 (5)  0.0033 (5)  0.0007 (5)  
**C14**  0.0596 (9)  0.0371 (7)  0.0307 (6)  0.0188 (6)  0.0018 (6)  0.0047 (5)  
**C15**  0.0603 (9)  0.0365 (7)  0.0305 (6)  0.0177 (6)  0.0044 (6)  0.0111 (5)  
**C16**  0.0526 (8)  0.0343 (7)  0.0368 (7)  0.0160 (6)  −0.0016 (6)  0.0028 (5)  
**C17**  0.0429 (7)  0.0361 (7)  0.0408 (7)  0.0107 (6)  0.0019 (6)  0.0062 (5)  
**C18**  0.0518 (9)  0.0378 (7)  0.0527 (9)  0.0170 (6)  0.0063 (7)  0.0052 (6)  

**Geometric parameters (Å, °)**

<table>
<thead>
<tr>
<th>Bond/Angle</th>
<th>Distance/Angle</th>
<th>Distance/Angle</th>
<th>Distance/Angle</th>
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<tbody>
<tr>
<td>O1—N1</td>
<td>1.2232 (15)</td>
<td>C8—C9</td>
<td>1.4815 (18)</td>
</tr>
<tr>
<td>O2—N1</td>
<td>1.2168 (16)</td>
<td>C8—H8A</td>
<td>0.9500</td>
</tr>
<tr>
<td>O3—C9</td>
<td>1.2189 (16)</td>
<td>C9—C10</td>
<td>1.4940 (17)</td>
</tr>
<tr>
<td>O4—C13</td>
<td>1.3693 (14)</td>
<td>C10—C15</td>
<td>1.3866 (17)</td>
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<tr>
<td>O4—C16</td>
<td>1.4362 (15)</td>
<td>C10—C11</td>
<td>1.3997 (18)</td>
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<tr>
<td>N1—C3</td>
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<td>C11—C12</td>
<td>1.3809 (17)</td>
</tr>
<tr>
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O1—N1—C3—C4 18.8 (2)  C18—C17—C16 120.4
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C4—C5—C6—C1 −0.7 (3)  C16—O4—C13—C14 −178.35 (11)
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Hydrogen-bond geometry (Å, °)

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Symmetry code: (i) x−1, y+2, z.