Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal structures of (*E*)-5-(4-methylphenyl)-1-(pyridin-2-yl)pent-2-en-4-yn-1-one and [3,4-bis-(phenylethynyl)cyclobutane-1,2-diyl]bis(pyridin-2-ylmethanone)

Ivan E. Ushakov,[a] Ivan S. Odin,[b] Pavel A. Gloukhov,[b] Alexander A. Golovanov,[b] Pavel V. Dorovatovskii[c] and Anna V. Vologzhanina[a]*

[a]A.N. Nesmeyanov Institute of Organoelement Compounds of RAS, 28, Vavilova Str., Moscow, 119991, Russian Federation, [b]Togliatti State University; 14 Belorusskaya Str., Togliatti, 445667, Russian Federation, and [c]National Research Center 'Kurchatov Institute', pl. Akad. Kurchatova, 1, Moscow, 123098, Russian Federation. *Correspondence e-mail: vologzhanina@mail.ru

Received 21 November 2019
Accepted 3 January 2020

Edited by K. Fejfarova, Institute of Biotechnology CAS, Czech Republic

Keywords: vinyl ketones with acetylene fragment; cyclobutane derivatives; photoreaction; crystal structure.

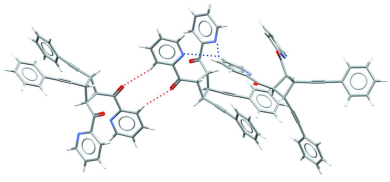
CCDC references: 1975319; 1975318

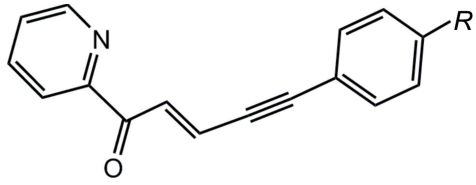
Supporting information: this article has supporting information at journals.iucr.org/e

Recrystallization of (*E*)-5-phenyl-1-(pyridin-2-yl)pent-2-en-4-yn-1-one at room temperature from ethylene glycol in daylight afforded [3,4-bis(phenylethynyl)cyclobutane-1,2-diyl)bis(pyridin-2-ylmethanone], $C_{32}H_{22}N_2O_2$ (**3**), while (*E*)-5-(4-methylphenyl)-1-(pyridin-2-yl)pent-2-en-4-yn-1-one, $C_{17}H_{13}NO$ (**2**), remained photoinert. This is the first experimental evidence that pentenynones can be photoreactive when fixed in nearly coplanar parallel positions. During the photoreaction, the bond lengths and angles along the pentenyne chain changed significantly, while the disposition of the pyridyl ring towards the keto group was almost unchanged. The cyclobutane ring adopts an *rctt* conformation.

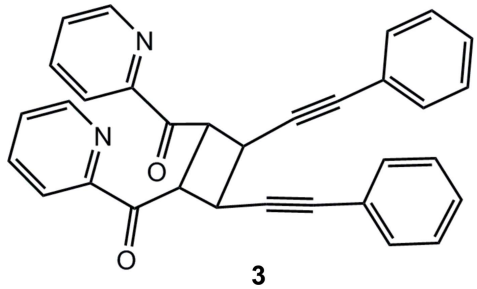
1. Chemical context

Vinyl-substituted ketones are known to take part in photo-initiated reactions both in the solid state and in solution (Hopkin *et al.*, 1991; Vatsadze *et al.*, 2006). Both *trans–cis* isomerization and [2 + 2] cycloaddition reactions can be observed depending on the nature of the substituents on the alkyl chain (Vatsadze *et al.*, 2006). Many of the compounds previously reported by us, including 1,5-diarylpentenynones (Golovanov *et al.*, 2013; Vologzhanina *et al.*, 2014; Voronova *et al.*, 20161) and cyclic ketones with vinylacetylene fragments (Voronova *et al.*, 2018) in crystals exhibit coplanar packing with a distance between the olefin fragments of less than 4.2 Å; thus, they satisfy the Schmidt (1971) criteria for a solid-state [2 + 2] cycloaddition to occur. However, our numerous attepts to carry out [2 + 2] photocycloaddition in these compounds were unsuccessful. We aimed to synthesize pyridine-substituted representatives of this family in order to fix olefin fragments in photoreactive positions using hydrogen bonding or coordination bonding as described by Nagarathinam *et al.* (2008). Two novel pyridine-2-yl-containing ketones, **1** and **2** (Scheme and Fig. 1), were synthesized as described below, and recrystallized from ethanol. Single-crystal XRD data for **2** could only be obtained using synchrotron radiation, while we failed to obtain a crystal structure of **1** using single-crystal or powder X-ray diffraction. Recrystallization of **1** and **2** from ethylene glycol afforded, respectively, a dimerization reaction product, **3**, and the initial solid phase.





1 *R* = H
2 *R* = Me

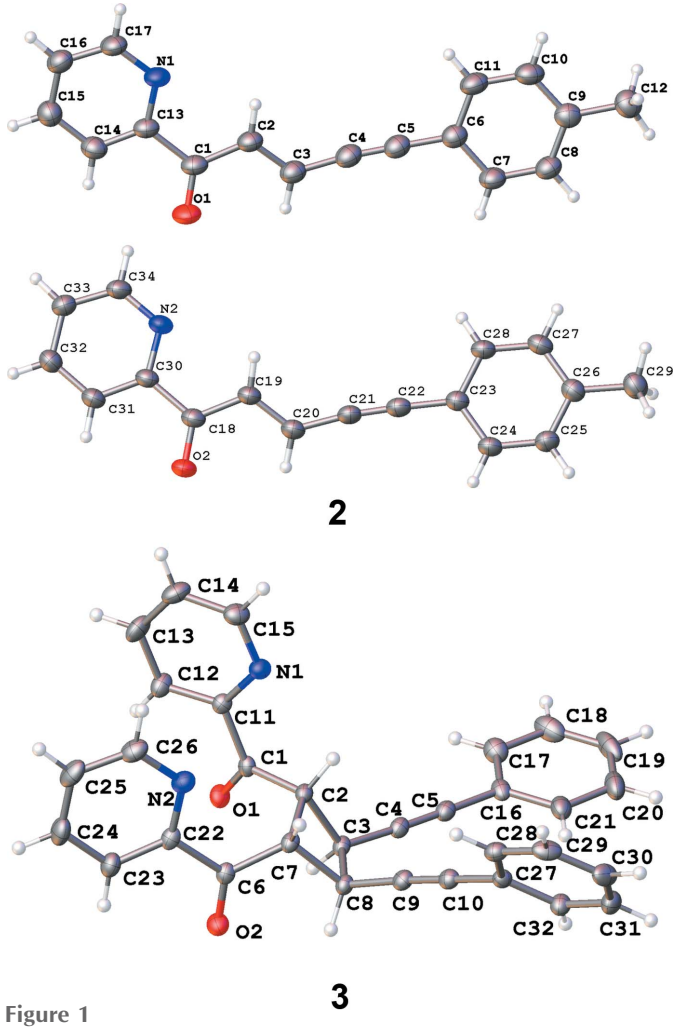


3

2. Structural commentary

The asymmetric unit of ketone **2** contains two independent molecules (Fig. 1). Their conformations are very similar to each other as shown in Fig. 2. Both molecules of **2** exhibit delocalization of charge density along the alkyl chain, as can be concluded from the bond lengths given in Table 1, the single bonds between a double and a triple bond being much shorter than the average value of 1.53–1.54 Å for a C—C bond. The corresponding values for the C═O ketone fragments in **3** are similar to those in **2**, while the absence of double bonds along the alkyl chain causes shortening of the allyl bonds and elongation of single bonds. The bond lengths in the cyclobutane ring of **3** are unequal: those corresponding to a previously 'double' bond are characteristic of a C—C bond (*ca* 1.55 Å), while the single bonds between two 'monomers' are elongated to 1.575 (2) Å. Only the *rctt* isomer of a 1,2,3,4-tetrasubstituted cyclobutane was obtained of four theoretically possible (based on XRD data).

The conformations of the molecules of both **2** and **3** is probably affected by intramolecular C—H···N contacts (Tables 2 and 3) involving the nitrogen atoms of the pyridine-2-yl rings and hydrogen atoms of ethenyl or cyclobutane moieties. The C—H···N angle does not exceed 102°; however, such a mutual disposition of the conjugated pyridine ring and a double bond was found not only in **2** and **3**, but also in previously reported pyridine-2-yl-containing chalcones. The chalcones in the Cambridge Structural Database (CSD, Version 5.40, update of November 2019; Groom *et al.*, 2016) [ABADUE (Fun *et al.*, 2011*b*), AFOPOC (Chantrapromma *et*



2

3

Figure 1
The molecular structure of **2** and **3**, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

Table 1
Selected geometry parameters (Å, °) for **2** and **3**.

The carbon atoms of the pentenynone fragment are numbered from 1 to 5. Φ_1 is the dihedral angle between the pyridine ring and the ketone fragment and Φ_2 is the dihedral angle between the pyridine and phenyl rings.

Bond	**2**	**3**
C1═O	1.226 (2), 1.228 (2)	1.212 (2), 1.215 (2)
C1—C_{py}	1.498 (3)–1.498 (2)	1.495 (2), 1.498 (2)
C1—C2	1.474 (3)–1.477 (3)	1.509 (2), 1.513 (2)
C2═C3	1.335 (3), 1.336 (3)	–
C_{cb}—C_{cb}	–	1.549 (2), 1.554 (2)
C3—C4	1.411 (3), 1.420 (3)	1.454 (2), 1.460 (2)
C4≡C5	1.206 (3), 1.203 (3)	1.195 (2), 1.194 (2)
C5—C_{Ph}	1.426 (3), 1.430 (3)	1.441 (2), 1.439 (2)
Φ_1	11.0 (1), 11.1 (1)	14.8 (1), 0.9 (1)
Φ_2	7.4 (1), 5.1 (1)	84.8 (1), 47.0 (1)

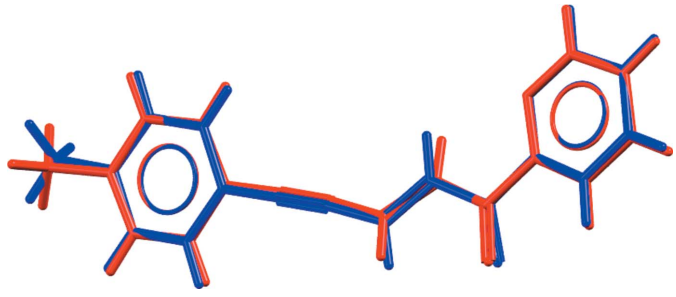


Figure 2
Conformation of the two symmetrically independent molecules in **2** (red and blue) in superimposed representation.

Table 2
Hydrogen-bond geometry (Å, °) for (2).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···N1	0.95	2.52	2.832 (3)	100
C16—H16···N2^{i}	0.95	2.66	3.555 (3)	158
C20—H20···N2^{i}	0.95	2.71	3.465 (3)	136
C3—H3···O1^{ii}	0.95	2.43	3.206 (3)	139
C19—H19···O2^{iii}	0.95	2.57	3.379 (2)	143
C25—H25···O2^{iv}	0.95	2.65	3.561 (2)	161

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

Table 3
Hydrogen-bond geometry (Å, °) for (3).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···N1	1.00	2.45	2.835 (3)	102
C3—H3···O1	1.00	2.49	2.899 (2)	104
C8—H8···O2	1.00	2.39	2.804 (2)	104
C25—H25···N1^{i}	0.95	2.60	3.445 (3)	148
C19—H19···N1^{ii}	0.95	2.73	3.665 (2)	167
C20—H20···O1^{iii}	0.95	2.62	3.263 (2)	125
C32—H32···O2^{iv}	0.95	2.55	3.487 (2)	168

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

al., 2013), AYUYOJ (Fun *et al.*, 2011*a*), BERXEC (Wang *et al.*, 2004), CIBYIY (Brennan *et al.*, 2018), COBJEJ (Prajapati *et al.*, 2008), ENINOG (Lee *et al.*, 2016), GARMAP (Fan & Wang, 2012), IJUSAI (Jasinski *et al.*, 2011), IXOXOJ (Dudek *et al.*, 2011), LANTAY (Qian *et al.*, 2017), OGIZIP and VUZVET (Tan *et al.*, 2016), PUKVEY (Rout & Mondal, 2015), QEMJOK and QEMJUQ (Albaladejo *et al.*, 2018), SOXHAP (Lin *et al.*, 2009), TISCEF (Jayarama *et al.*, 2013) and YUQTEK (Li *et al.*, 2010)] demonstrate similar conformations, but different crystal packing in the region of pyridyl ring. The majority of 1-phenyl-substituted chalcones and 1-phenyl-substituted pentenyn-1-ones also exhibit a nearly coplanar arrangement of the aryl and ketone fragments and thus no hindrance occurs between the hydrogen atoms of these fragments.

3. Supramolecular features

As the independent molecules of ketone **2** have similar conformations, their crystalline environment becomes of particular interest because it can rationalize why $Z \neq 1$. Previously, we found that the most abundant C—H···O-bonded associates in the crystals of chalcones, polyenones and pentenynones include dimers, head-to-tail chains and zigzag C—H···O chains with the most acidic proton of a molecule (Vologzhanina *et al.*, 2014). The two independent molecules of ketone **2** demonstrate two of these motifs (Fig. 3). In the C—H···O-connected dimers, r(C···O) = 3.206 (3) Å, and in the head-to-tail chains r(C···O) and r(C···N) = 3.379 (2) and 3.465 (3) Å, respectively. The corresponding C—H···O and C—H···N angles are, respectively, 139, 143 and 136°. Note,

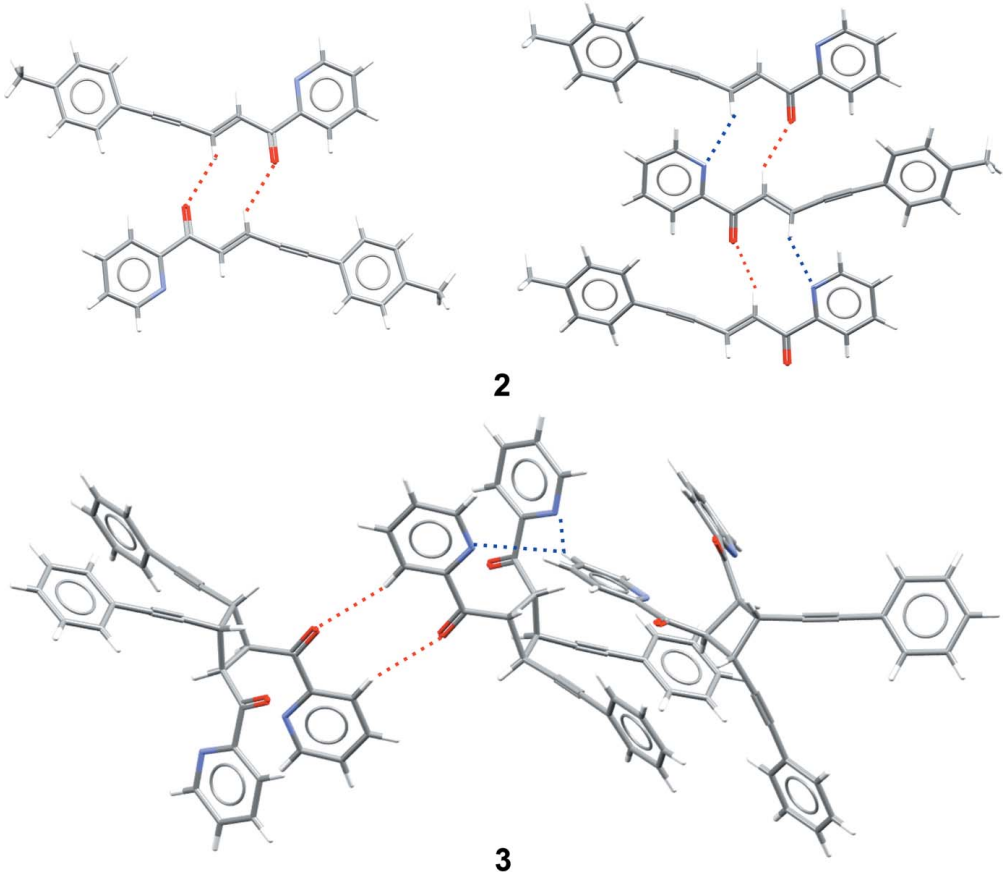


Figure 3
Supramolecular aggregates in the crystals of **2** and **3**. Hydrogen bonds are depicted by dashed lines.

Table 4
Experimental details.

	(3)	(2)
Crystal data		
Chemical formula	$C_{32}H_{22}N_2O_2$	$C_{17}H_{13}NO$
M_r	466.51	247.28
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/c$
Temperature (K)	120	100
a, b, c (Å)	12.272 (3), 18.720 (4), 11.425 (2)	14.859 (3), 17.747 (4), 9.995 (2)
β (°)	115.850 (3)	101.06 (3)
V (Å^3)	2362.0 (8)	2586.7 (9)
Z	4	8
Radiation type	Mo $K\alpha$	Synchrotron, λ = 0.80248 Å
μ (mm^{-1})	0.08	0.10
Crystal size (mm)	0.46 × 0.28 × 0.17	0.02 × 0.02 × 0.01
Data collection		
Diffractometer	Bruker SMART APEX CCD area detector	Mar CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)	Multi-scan (*SCALA*; Evans, 2006)
T_{min}, T_{max}	0.848, 0.903	0.997, 0.999
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	24543, 7095, 4255	23024, 5645, 4453
R_{int}	0.079	0.077
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.714	0.640
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.056, 0.130, 0.99	0.060, 0.157, 1.02
No. of reflections	7095	5645
No. of parameters	325	346
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.33, −0.31	0.23, −0.21

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *Marccd* (Doyle, 2011), *iMosflm* (Battye *et al.*, 2011), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

that only one of two independent molecules in **2** forms head-to-tail chains *via* a pair of intermolecular C—H···O and C—N···N bonds. None of the previously reported pyridine-2-yl-containing chalcones nor **3** forms such associates. Instead, the nitrogen atoms interact with the hydrogen atoms of the alkyl and aryl groups. For example, in the crystal of **3**, the hydrogen atoms of a pyridine-2-yl ring take part in C—H···N interactions [Fig. 3, r(C···N) = 3.445 (3)–3.665 (2) Å]. Oxygen atoms take part in C—H···O bonding with hydrogen atoms of the phenyl and pyridin-2-yl rings. In addition, in **2** and **3**, numerous hydrophobic interactions can be found.

4. Synthesis and crystallization

The 5-phenyl-1-(pyridin-2-yl)pent-2-en-4-yn-1-one, **1**, and 5-(4-methylphenyl)-1-(pyridin-2-yl)pent-2-en-4-yn-1-one, **2**, were synthesized according to the previously described method (Golovanov *et al.*, 2013). Single crystals of **3** were grown from solution of **1** in ethylene glycol. The ^{1}H NMR spectrum indicates the presence of a mixture of reaction products and unreacted **1**. Powder XRD indicated that the solid sample of the recrystallized ketone consisted of both **1** and **3**, and thus solid **3** could not be characterized by other physicochemical methods. Recrystallization of **2** from ethylene glycol afforded **2** as obtained from XRD data.

For **1**: yellowish needles, yield 61%, m.p. 348–351 K (from a mixture of water and ethanol). ^{1}H NMR (300 MHz, $CDCl_3$), δ, ppm: 8.48 *s* (1C, C_{Ar}, C_{Py}), 8.09–8.16 *m* (2C, C_{Ar}, C_{Py}, C^2), 7.84–7.79 *m* (2C, C_{Ar}, C_{Py}), 7.20–7.52 *m* (6C, C_{Ar}, C^3). ^{13}C NMR (75 MHz, $CDCl_3$), δ, ppm: 188.5 (C1), 152.6, 149.0, 132.2, 131.9, 129.5, 128.6, 128.2, 128.0, 127.1, 122.1, 99.6 (C^5), 88.9 (C^4). Found, %: C 82.44; H 5.41. $C_{16}H_{11}NO$. Calculated, %: C 82.38; H 4.75.

For **2**: yellowish needles, yield 34%, m.p. 373–374 K (from a mixture of water–ethanol. IR Spectra, ν, cm^{-1}: 2191 (C≡C), 1649 (C=O). Found, %: C 82.44; H 5.33. $C_{17}H_{13}NO$. Calculated, %: C 82.57; H 5.30.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. Intensity data for **2** were collected at the K4.4 'Belok' beamline of the Kurchatov Synchrotron Radiation Source (NRC 'Kurchatov Institute', Moscow, Russia) at a wavelength of 0.80248 Å using a Rayonix CCD 165 detector. Image integration was performed using *iMosflm* software (Battye *et al.*, 2011). Hydrogen atoms were placed in calculated positions (0.95–1.00 Å) and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

Acknowledgements

This study was supported by the Russian Science Foundation (grant No 17–13-01442). The X-ray diffraction experiments of **2** and **3** were performed using, respectively, the K4.4 'Belok' beamline of the Kurchatov Synchrotron Radiation Source and equipment of the Centre for Molecular Studies of INEOS RAS.

Funding information

Funding for this research was provided by: Russian Science Foundation (grant No. 17-13-01442 to Anna V. Vologzhanina).

References

Albaladejo, M. J., González-Soria, M. J. & Alonso, F. (2018). *Green Chem.* **20**, 701–712.
Battye, T. G. G., Kontogiannis, L., Johnson, O., Powell, H. R. & Leslie, A. G. W. (2011). *Acta Cryst.* D**67**, 271–281.
Brennan, C., Housecroft, C. E., Constable, E. C., Neuburger, M. & Prescimone, A. (2018). CSD Communication (refcode CIBYIY). CCDC, Cambridge, England. https://doi.org/10.5517/ccdc.csd.cc205qfc
Bruker (2014). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chantrapromma, S., Suwunwong, T., Boonnak, N. & Fun, H.-K. (2013). *Acta Cryst.* E**69**, o1076–o1077.
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
Doyle, R. A. (2011). *Marccd software manual*. Rayonix LLC, Evanston, USA.
Dudek, M., Clegg, J. K., Glasson, C. R. K., Kelly, N., Gloe, K., Gloe, K., Kelling, A., Buschmann, H., Jolliffe, K. A., Lindoy, L. F. & Meehan, G. V. (2011). *Cryst. Growth Des.* **11**, 1697–1704.
Evans, P. (2006). *Acta Cryst.* D**62**, 72–82.
Fan, C.-B. & Wang, X.-M. (2012). *Acta Cryst.* E**68**, o417.
Fun, H.-K., Chantrapromma, S. & Suwunwong, T. (2011*b*). *Acta Cryst.* E**67**, o2789–o2790.
Fun, H.-K., Suwunwong, T. & Chantrapromma, S. (2011*a*). *Acta Cryst.* E**67**, o2406–o2407.
Golovanov, A. A., Latypova, D. R., Bekin, V. V., Pisareva, V. S., Vologzhanina, A. V. & Dokichev, V. A. (2013). *Russ. J. Org. Chem.* **49**, 1264–1269.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.
Hopkin, S. E., Muir, M. & Theocharis, Ch. R. (1991). *J. Chem. Soc., Dalton Trans. 2.* pp. 1131–1135.
Jasinski, J. P., Butcher, R. J., Samshuddin, S., Narayana, B. & Yathirajan, H. S. (2011). *Acta Cryst.* E**67**, o352–o353.
Jayarama, A., Ravindra, H. J., Menezes, A. P., Dharmaprakash, S. M. & Ng, S. W. (2013). *J. Mol. Struct.* **1051**, 285–291.
Lee, S.-L., Tan, A. L., Young, D. J., Jotani, M. M. & Tiekink, E. R. T. (2016). *Acta Cryst.* E**72**, 849–853.
Li, X. (2010). *Acta Cryst.* E**66**, o1613.
Lin, S., Jia, R., Zhang, X., Wang, Z. & Yuan, Y. (2009). *Acta Cryst.* E**65**, o1161.
Nagarathinam, M., Peedikakkal, A. M. P. & Vittal, J. J. (2008). *Chem. Commun.* pp. 5277.
Prajapati, R., Mishra, L., Grabowski, S. J., Govil, G. & Dubey, S. K. (2008). *J. Mol. Struct.* **879**, 1–6.
Qian, Z., Li, D., Xie, T., Zhang, X., He, Y., Ai, Y. & Zhang, G. (2017). *CrystEngComm*, **19**, 2283–2287.
Rout, K. C. & Mondal, B. (2015). *Inorg. Chim. Acta*, **437**, 54–58.
Schmidt, G. M. J. (1971). *Pure. Apll. Chem.* **27**, 647–678.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Tan, J., Zhang, Y., Zhang, M., Tian, X., Wang, Y., Li, S., Wang, C., Zhou, H., Yang, J., Tian, Y. & Wu, J. (2016). *J. Mater. Chem. C.* **4**, 3256–3267.
Vatsadze, S. Z., Manaenkova, M. A., Sviridenkova, N. V., Zyk, N. V., Krut'ko, D. P., Churakov, A. V., Antipin, M. Yu., Howard, J. A. K. & Lang, H. (2006). *Russ. Chem. Bull.* **55**, 1184–1194.
Vologzhanina, A. V., Golovanov, A. A., Gusev, D. M., Odin, I. S., Apreyan, R. A. & Suponitsky, K. Yu. (2014). *Cryst. Growth Des.* **14**, 4402–4410.
Voronova, E. D., Golovanov, A. A., Odin, I. S., Anisimov, M. A., Dorovatovskii, P. V., Zubavichus, Y. V. & Vologzhanina, A. V. (2018). *Acta Cryst.* C**74**, 1674–1683.
Voronova, E. D., Golovanov, A. A., Suponitsky, K. Yu., Fedyanin, I. V. & Vologzhanina, A. V. (2016). *Cryst. Growth Des.* **16**, 3859–3868.
Wang, P., Moorefield, G. R. & Newkome, G. R. (2004). *Org. Lett.* **6**, 1197–1200.

supporting information

Acta Cryst. (2020). E**76**, 192-196 [https://doi.org/10.1107/S2056989020000055]

Crystal structures of (*E*)-5-(4-methylphenyl)-1-(pyridin-2-yl)pent-2-en-4-yn-1-one and [3,4-bis(phenylethynyl)cyclobutane-1,2-diyl]bis(pyridin-2-yl-methanone)

Ivan E. Ushakov, Ivan S. Odin, Pavel A. Gloukhov, Alexander A. Golovanov, Pavel V. Dorovatovskii and Anna V. Vologzhanina

Computing details

Data collection: *SAINT* (Bruker, 2014) for (3). Cell refinement: *APEX2* (Bruker, 2014) for (3); *Marccd* (Doyle, 2011) for (2). Data reduction: *SAINT*(Bruker, 2014) for (3); *iMosflm* (Battye *et al.*, 2011) for (2). For both structures, program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

[3,4-Bis(phenylethynyl)cyclobutane-1,2-diyl]bis(pyridin-2-ylmethanone) (3)

Crystal data

$C_{32}H_{22}N_2O_2$
M_r = 466.51
Monoclinic, $P2_1/c$
a = 12.272 (3) Å
b = 18.720 (4) Å
c = 11.425 (2) Å
β = 115.850 (3)°
V = 2362.0 (8) Å^3
Z = 4
$F(000)$ = 976
D_x = 1.312 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4566 reflections
θ = 2.2–30.0°
μ = 0.08 mm^{-1}
T = 120 K
Prism, orange
0.46 × 0.28 × 0.17 mm

Data collection

Bruker SMART APEX CCD area detector diffractometer
Radiation source: sealed tube
Graphite monochromator
Detector resolution: 8 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2014)
T_{min} = 0.848, T_{max} = 0.903
24543 measured reflections
7095 independent reflections
4255 reflections with $I > 2\sigma(I)$
R_{int} = 0.079
θ_{max} = 30.5°, θ_{min} = 1.8°
h = −15→17
k = −26→26
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.130
S = 0.99
7095 reflections
325 parameters
0 restraints

Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0557P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.14655 (11)	0.50117 (6)	0.61894 (10)	0.0224 (3)
O2	0.44626 (12)	0.53397 (6)	0.81027 (11)	0.0249 (3)
N1	0.03170 (13)	0.66755 (7)	0.47517 (13)	0.0215 (3)
N2	0.26664 (14)	0.68759 (7)	0.76527 (14)	0.0236 (3)
C1	0.14015 (15)	0.55793 (8)	0.56522 (14)	0.0167 (3)
C2	0.23152 (15)	0.57818 (8)	0.51555 (15)	0.0168 (3)
H2	0.1954	0.6114	0.4396	0.020*
C3	0.30105 (15)	0.51652 (8)	0.48769 (15)	0.0173 (3)
H3	0.3031	0.4743	0.5421	0.021*
C4	0.25950 (16)	0.49385 (8)	0.35333 (15)	0.0188 (3)
C5	0.22108 (16)	0.47470 (8)	0.24269 (16)	0.0202 (4)
C6	0.38544 (15)	0.58614 (8)	0.75844 (15)	0.0177 (3)
C7	0.35663 (15)	0.60721 (8)	0.62014 (15)	0.0175 (3)
H7	0.3676	0.6595	0.6106	0.021*
C8	0.42148 (16)	0.55980 (8)	0.55808 (15)	0.0179 (3)
H8	0.4875	0.5304	0.6245	0.021*
C9	0.46337 (16)	0.59998 (8)	0.47593 (15)	0.0198 (3)
C10	0.50391 (16)	0.63388 (8)	0.41551 (15)	0.0200 (4)
C11	0.04630 (15)	0.61189 (8)	0.55421 (15)	0.0171 (3)
C12	−0.01798 (17)	0.60341 (8)	0.62740 (17)	0.0241 (4)
H12	−0.0064	0.5624	0.6805	0.029*
C13	−0.09935 (18)	0.65600 (9)	0.62137 (19)	0.0303 (4)
H13	−0.1441	0.6521	0.6711	0.036*
C14	−0.11435 (18)	0.71411 (9)	0.54191 (18)	0.0302 (4)
H14	−0.1687	0.7514	0.5367	0.036*
C15	−0.04852 (17)	0.71700 (9)	0.46982 (17)	0.0270 (4)
H15	−0.0612	0.7565	0.4132	0.032*
C16	0.17317 (16)	0.45234 (8)	0.10882 (15)	0.0208 (4)
C17	0.06057 (18)	0.42003 (8)	0.04773 (17)	0.0282 (4)
H17	0.0149	0.4108	0.0952	0.034*
C18	0.0143 (2)	0.40106 (9)	−0.08242 (17)	0.0364 (5)
H18	−0.0629	0.3789	−0.1238	0.044*
C19	0.0804 (2)	0.41430 (10)	−0.15217 (18)	0.0406 (6)
H19	0.0486	0.4013	−0.2413	0.049*

C20	0.1925 (2)	0.44636 (10)	−0.09211 (18)	0.0370 (5)
H20	0.2375	0.4558	−0.1402	0.044*
C21	0.23975 (19)	0.46490 (9)	0.03804 (16)	0.0274 (4)
H21	0.3177	0.4862	0.0794	0.033*
C22	0.33415 (15)	0.63164 (8)	0.83025 (15)	0.0187 (3)
C23	0.35862 (17)	0.61505 (9)	0.95735 (16)	0.0244 (4)
H23	0.4081	0.5753	1.0000	0.029*
C24	0.30935 (18)	0.65777 (10)	1.02106 (18)	0.0313 (4)
H24	0.3238	0.6477	1.1081	0.038*
C25	0.23927 (18)	0.71490 (9)	0.95603 (18)	0.0309 (4)
H25	0.2044	0.7452	0.9972	0.037*
C26	0.22022 (18)	0.72774 (9)	0.82891 (18)	0.0290 (4)
H26	0.1713	0.7674	0.7847	0.035*
C27	0.55587 (15)	0.67731 (8)	0.34887 (15)	0.0187 (3)
C28	0.56084 (16)	0.75148 (8)	0.36530 (16)	0.0224 (4)
H28	0.5309	0.7727	0.4210	0.027*
C29	0.60909 (17)	0.79383 (9)	0.30078 (18)	0.0279 (4)
H29	0.6126	0.8442	0.3126	0.033*
C30	0.65229 (18)	0.76346 (9)	0.21905 (18)	0.0292 (4)
H30	0.6837	0.7930	0.1733	0.035*
C31	0.64973 (18)	0.69037 (9)	0.20388 (17)	0.0289 (4)
H31	0.6810	0.6696	0.1490	0.035*
C32	0.60178 (17)	0.64693 (8)	0.26814 (16)	0.0227 (4)
H32	0.6002	0.5966	0.2572	0.027*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0294 (8)	0.0176 (5)	0.0243 (6)	0.0001 (5)	0.0154 (6)	0.0028 (4)
O2	0.0310 (8)	0.0202 (6)	0.0250 (6)	0.0042 (5)	0.0136 (6)	0.0002 (5)
N1	0.0219 (9)	0.0185 (7)	0.0245 (7)	0.0011 (6)	0.0103 (7)	0.0008 (5)
N2	0.0242 (9)	0.0190 (7)	0.0269 (7)	−0.0005 (6)	0.0105 (7)	−0.0052 (6)
C1	0.0200 (10)	0.0153 (7)	0.0154 (7)	−0.0033 (6)	0.0084 (7)	−0.0035 (6)
C2	0.0211 (10)	0.0136 (7)	0.0182 (7)	0.0017 (6)	0.0109 (7)	0.0010 (6)
C3	0.0228 (10)	0.0126 (7)	0.0203 (8)	0.0007 (6)	0.0130 (7)	−0.0006 (6)
C4	0.0223 (10)	0.0140 (7)	0.0239 (8)	−0.0001 (6)	0.0136 (7)	0.0006 (6)
C5	0.0233 (10)	0.0159 (7)	0.0245 (8)	0.0015 (6)	0.0133 (8)	0.0007 (6)
C6	0.0206 (10)	0.0142 (7)	0.0198 (7)	−0.0042 (6)	0.0102 (7)	−0.0039 (6)
C7	0.0216 (10)	0.0125 (7)	0.0218 (8)	−0.0005 (6)	0.0125 (7)	−0.0015 (6)
C8	0.0218 (10)	0.0150 (7)	0.0205 (8)	0.0005 (6)	0.0126 (7)	0.0000 (6)
C9	0.0215 (10)	0.0164 (7)	0.0231 (8)	0.0004 (6)	0.0114 (7)	−0.0030 (6)
C10	0.0217 (10)	0.0173 (7)	0.0212 (8)	0.0010 (6)	0.0096 (7)	−0.0025 (6)
C11	0.0177 (10)	0.0141 (7)	0.0196 (8)	−0.0034 (6)	0.0083 (7)	−0.0034 (6)
C12	0.0290 (11)	0.0183 (8)	0.0311 (9)	−0.0010 (7)	0.0189 (8)	−0.0010 (7)
C13	0.0284 (12)	0.0279 (9)	0.0449 (11)	−0.0024 (8)	0.0255 (10)	−0.0047 (8)
C14	0.0235 (12)	0.0241 (9)	0.0448 (11)	0.0035 (7)	0.0167 (9)	−0.0025 (8)
C15	0.0268 (12)	0.0201 (8)	0.0324 (10)	0.0024 (7)	0.0115 (8)	0.0041 (7)
C16	0.0316 (12)	0.0130 (7)	0.0191 (8)	0.0045 (6)	0.0122 (8)	0.0008 (6)

C17	0.0356 (13)	0.0203 (8)	0.0275 (9)	−0.0002 (7)	0.0127 (9)	−0.0025 (7)
C18	0.0452 (15)	0.0226 (9)	0.0277 (10)	0.0036 (8)	0.0032 (9)	−0.0061 (7)
C19	0.0704 (18)	0.0258 (10)	0.0180 (9)	0.0156 (10)	0.0121 (10)	−0.0015 (7)
C20	0.0578 (16)	0.0339 (10)	0.0260 (9)	0.0149 (10)	0.0246 (10)	0.0060 (8)
C21	0.0369 (13)	0.0244 (9)	0.0240 (9)	0.0076 (8)	0.0162 (9)	0.0051 (7)
C22	0.0206 (10)	0.0155 (7)	0.0224 (8)	−0.0058 (6)	0.0115 (7)	−0.0071 (6)
C23	0.0282 (12)	0.0238 (8)	0.0251 (9)	−0.0019 (7)	0.0152 (8)	−0.0044 (7)
C24	0.0352 (13)	0.0381 (10)	0.0260 (9)	−0.0031 (8)	0.0184 (9)	−0.0091 (8)
C25	0.0293 (12)	0.0304 (9)	0.0374 (10)	−0.0038 (8)	0.0187 (9)	−0.0169 (8)
C26	0.0275 (12)	0.0219 (8)	0.0378 (10)	0.0016 (7)	0.0144 (9)	−0.0079 (7)
C27	0.0174 (10)	0.0189 (7)	0.0198 (8)	0.0005 (6)	0.0083 (7)	0.0017 (6)
C28	0.0209 (11)	0.0198 (8)	0.0248 (9)	0.0022 (7)	0.0084 (8)	−0.0008 (6)
C29	0.0249 (11)	0.0150 (8)	0.0378 (10)	0.0012 (7)	0.0081 (9)	0.0057 (7)
C30	0.0258 (12)	0.0293 (9)	0.0354 (10)	0.0011 (7)	0.0161 (9)	0.0136 (8)
C31	0.0308 (12)	0.0337 (10)	0.0288 (9)	0.0061 (8)	0.0192 (9)	0.0060 (8)
C32	0.0285 (11)	0.0178 (8)	0.0249 (9)	0.0033 (7)	0.0146 (8)	0.0020 (6)

Geometric parameters (Å, º)

O1—C1	1.2123 (18)	C15—H15	0.9500
O2—C6	1.2148 (19)	C16—C17	1.386 (3)
N1—C11	1.3387 (19)	C16—C21	1.397 (2)
N1—C15	1.333 (2)	C17—H17	0.9500
N2—C22	1.341 (2)	C17—C18	1.387 (2)
N2—C26	1.334 (2)	C18—H18	0.9500
C1—C2	1.509 (2)	C18—C19	1.385 (3)
C1—C11	1.495 (2)	C19—H19	0.9500
C2—H2	1.0000	C19—C20	1.378 (3)
C2—C3	1.549 (2)	C20—H20	0.9500
C2—C7	1.575 (2)	C20—C21	1.384 (3)
C3—H3	1.0000	C21—H21	0.9500
C3—C4	1.454 (2)	C22—C23	1.384 (2)
C3—C8	1.566 (2)	C23—H23	0.9500
C4—C5	1.195 (2)	C23—C24	1.385 (2)
C5—C16	1.441 (2)	C24—H24	0.9500
C6—C7	1.513 (2)	C24—C25	1.370 (3)
C6—C22	1.498 (2)	C25—H25	0.9500
C7—H7	1.0000	C25—C26	1.388 (3)
C7—C8	1.554 (2)	C26—H26	0.9500
C8—H8	1.0000	C27—C28	1.399 (2)
C8—C9	1.460 (2)	C27—C32	1.395 (2)
C9—C10	1.194 (2)	C28—H28	0.9500
C10—C27	1.439 (2)	C28—C29	1.380 (2)
C11—C12	1.386 (2)	C29—H29	0.9500
C12—H12	0.9500	C29—C30	1.381 (3)
C12—C13	1.382 (2)	C30—H30	0.9500
C13—H13	0.9500	C30—C31	1.378 (2)
C13—C14	1.377 (3)	C31—H31	0.9500

C14—H14	0.9500	C31—C32	1.387 (2)
C14—C15	1.384 (2)	C32—H32	0.9500
C15—N1—C11	116.77 (14)	C17—C16—C5	121.14 (15)
C26—N2—C22	116.48 (15)	C17—C16—C21	119.21 (16)
O1—C1—C2	121.04 (14)	C21—C16—C5	119.63 (17)
O1—C1—C11	120.81 (14)	C16—C17—H17	119.9
C11—C1—C2	118.03 (13)	C16—C17—C18	120.23 (18)
C1—C2—H2	111.2	C18—C17—H17	119.9
C1—C2—C3	117.15 (12)	C17—C18—H18	119.9
C1—C2—C7	116.08 (12)	C19—C18—C17	120.2 (2)
C3—C2—H2	111.2	C19—C18—H18	119.9
C3—C2—C7	88.33 (12)	C18—C19—H19	120.0
C7—C2—H2	111.2	C20—C19—C18	119.94 (17)
C2—C3—H3	109.2	C20—C19—H19	120.0
C2—C3—C8	89.43 (11)	C19—C20—H20	119.9
C4—C3—C2	117.61 (14)	C19—C20—C21	120.24 (19)
C4—C3—H3	109.2	C21—C20—H20	119.9
C4—C3—C8	120.76 (13)	C16—C21—H21	119.9
C8—C3—H3	109.2	C20—C21—C16	120.2 (2)
C5—C4—C3	177.49 (18)	C20—C21—H21	119.9
C4—C5—C16	179.10 (19)	N2—C22—C6	116.52 (14)
O2—C6—C7	122.18 (14)	N2—C22—C23	123.73 (14)
O2—C6—C22	120.37 (14)	C23—C22—C6	119.75 (15)
C22—C6—C7	117.44 (13)	C22—C23—H23	120.7
C2—C7—H7	112.8	C22—C23—C24	118.50 (16)
C6—C7—C2	114.14 (13)	C24—C23—H23	120.7
C6—C7—H7	112.8	C23—C24—H24	120.7
C6—C7—C8	113.24 (13)	C25—C24—C23	118.70 (16)
C8—C7—C2	88.89 (11)	C25—C24—H24	120.7
C8—C7—H7	112.8	C24—C25—H25	120.6
C3—C8—H8	112.1	C24—C25—C26	118.82 (16)
C7—C8—C3	88.48 (12)	C26—C25—H25	120.6
C7—C8—H8	112.1	N2—C26—C25	123.76 (17)
C9—C8—C3	117.03 (13)	N2—C26—H26	118.1
C9—C8—C7	113.14 (12)	C25—C26—H26	118.1
C9—C8—H8	112.1	C28—C27—C10	119.56 (14)
C10—C9—C8	175.96 (18)	C32—C27—C10	121.30 (14)
C9—C10—C27	176.81 (17)	C32—C27—C28	119.14 (15)
N1—C11—C1	117.01 (14)	C27—C28—H28	119.9
N1—C11—C12	123.50 (15)	C29—C28—C27	120.17 (16)
C12—C11—C1	119.46 (14)	C29—C28—H28	119.9
C11—C12—H12	120.8	C28—C29—H29	119.8
C13—C12—C11	118.50 (15)	C28—C29—C30	120.35 (15)
C13—C12—H12	120.8	C30—C29—H29	119.8
C12—C13—H13	120.6	C29—C30—H30	120.0
C14—C13—C12	118.78 (16)	C31—C30—C29	119.96 (16)
C14—C13—H13	120.6	C31—C30—H30	120.0

C13—C14—H14	120.7	C30—C31—H31	119.8
C13—C14—C15	118.58 (16)	C30—C31—C32	120.49 (16)
C15—C14—H14	120.7	C32—C31—H31	119.8
N1—C15—C14	123.84 (16)	C27—C32—H32	120.1
N1—C15—H15	118.1	C31—C32—C27	119.87 (15)
C14—C15—H15	118.1	C31—C32—H32	120.1

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···N1	1.00	2.45	2.835 (3)	102
C3—H3···O1	1.00	2.49	2.899 (2)	104
C8—H8···O2	1.00	2.39	2.804 (2)	104
C25—H25···N1^{i}	0.95	2.60	3.445 (3)	148
C19—H19···N1^{ii}	0.95	2.73	3.665 (2)	167
C20—H20···O1^{iii}	0.95	2.62	3.263 (2)	125
C32—H32···O2^{iv}	0.95	2.55	3.487 (2)	168

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

(*E*)-5-(4-Methylphenyl)-1-(pyridin-2-yl)pent-2-en-4-yn-1-one (2)

Crystal data

$C_{17}H_{13}NO$
$M_r = 247.28$
Monoclinic, $P2_1/c$
$a = 14.859$ (3) Å
$b = 17.747$ (4) Å
$c = 9.995$ (2) Å
$\beta = 101.06$ (3)°
$V = 2586.7$ (9) Å^3
$Z = 8$
$F(000) = 1040$
$D_x = 1.270$ Mg m^{-3}
Melting point: 373 K
Synchrotron radiation, $\lambda = 0.80248$ Å
Cell parameters from 148 reflections
$\theta = 3.5$–25.6°
$\mu = 0.10$ mm^{-1}
$T = 100$ K
Plate, yellow
0.02 × 0.02 × 0.01 mm

Data collection

Mar CCD diffractometer
phi scans
Absorption correction: multi-scan (*SCALA*; Evans, 2006)
$T_{min} = 0.997$, $T_{max} = 0.999$
23024 measured reflections
5645 independent reflections
4453 reflections with $I > 2\sigma(I)$
$R_{int} = 0.077$
$\theta_{max} = 30.9°$, $\theta_{min} = 3.3°$
$h = -18\rightarrow18$
$k = -22\rightarrow22$
$l = -12\rightarrow12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.157$
$S = 1.02$
5645 reflections
346 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.057P)^2 + 1.189P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.051 (4)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.08252 (11)	0.41331 (9)	0.08638 (13)	0.0493 (4)
N1	0.19600 (11)	0.39239 (9)	0.42804 (15)	0.0383 (4)
C1	0.11482 (13)	0.43098 (12)	0.20438 (18)	0.0379 (4)
C2	0.12405 (13)	0.51037 (11)	0.24820 (19)	0.0380 (4)
H2	0.1567	0.5228	0.3367	0.046*
C3	0.08643 (14)	0.56483 (12)	0.1634 (2)	0.0409 (5)
H3	0.0526	0.5493	0.0773	0.049*
C4	0.09180 (14)	0.64305 (12)	0.1887 (2)	0.0420 (5)
C5	0.09537 (14)	0.71075 (12)	0.1993 (2)	0.0413 (5)
C6	0.10213 (14)	0.79075 (12)	0.20949 (19)	0.0394 (4)
C7	0.07036 (14)	0.83675 (12)	0.0969 (2)	0.0418 (5)
H7	0.0431	0.8147	0.0122	0.050*
C8	0.07839 (14)	0.91450 (12)	0.1084 (2)	0.0414 (5)
H8	0.0561	0.9450	0.0311	0.050*
C9	0.11833 (14)	0.94863 (12)	0.2305 (2)	0.0412 (5)
C10	0.14997 (15)	0.90211 (13)	0.3424 (2)	0.0467 (5)
H10	0.1776	0.9242	0.4269	0.056*
C11	0.14194 (15)	0.82508 (13)	0.3329 (2)	0.0458 (5)
H11	0.1636	0.7948	0.4108	0.055*
C12	0.12884 (16)	1.03274 (12)	0.2434 (2)	0.0494 (5)
H12A	0.1932	1.0464	0.2476	0.074*
H12B	0.1094	1.0496	0.3268	0.074*
H12C	0.0908	1.0571	0.1643	0.074*
C13	0.14687 (13)	0.37043 (11)	0.30695 (18)	0.0353 (4)
C14	0.12604 (14)	0.29623 (12)	0.2729 (2)	0.0408 (5)
H14	0.0903	0.2835	0.1865	0.049*
C15	0.15839 (15)	0.24046 (12)	0.3676 (2)	0.0449 (5)
H15	0.1445	0.1889	0.3479	0.054*
C16	0.21129 (15)	0.26184 (12)	0.4913 (2)	0.0431 (5)
H16	0.2359	0.2251	0.5575	0.052*
C17	0.22753 (15)	0.33754 (12)	0.51642 (19)	0.0426 (5)
H17	0.2634	0.3516	0.6020	0.051*
O2	0.45764 (10)	0.31523 (8)	0.50791 (12)	0.0395 (3)
N2	0.35093 (11)	0.37903 (9)	0.18087 (14)	0.0342 (4)
C18	0.42385 (13)	0.31321 (11)	0.38578 (17)	0.0327 (4)
C19	0.41012 (13)	0.24154 (10)	0.30956 (17)	0.0331 (4)
H19	0.3996	0.2414	0.2128	0.040*
C20	0.41275 (13)	0.17693 (10)	0.37887 (18)	0.0342 (4)
H20	0.4235	0.1806	0.4755	0.041*

C21	0.40113 (13)	0.10337 (10)	0.32236 (17)	0.0339 (4)
C22	0.39020 (13)	0.03862 (11)	0.28721 (17)	0.0343 (4)
C23	0.37756 (13)	−0.03916 (10)	0.25127 (17)	0.0328 (4)
C24	0.41627 (13)	−0.09465 (10)	0.34423 (17)	0.0343 (4)
H24	0.4510	−0.0804	0.4304	0.041*
C25	0.40418 (13)	−0.17024 (11)	0.31134 (18)	0.0362 (4)
H25	0.4313	−0.2073	0.3751	0.043*
C26	0.35279 (13)	−0.19286 (11)	0.18589 (18)	0.0354 (4)
C27	0.31461 (13)	−0.13723 (11)	0.09343 (18)	0.0369 (4)
H27	0.2800	−0.1516	0.0072	0.044*
C28	0.32623 (13)	−0.06173 (11)	0.12495 (17)	0.0354 (4)
H28	0.2993	−0.0248	0.0607	0.042*
C29	0.33893 (15)	−0.27549 (11)	0.1537 (2)	0.0435 (5)
H29A	0.3925	−0.3039	0.2002	0.065*
H29B	0.3313	−0.2831	0.0551	0.065*
H29C	0.2840	−0.2933	0.1849	0.065*
C30	0.39586 (12)	0.38512 (10)	0.31068 (16)	0.0312 (4)
C31	0.41532 (13)	0.45334 (11)	0.37716 (18)	0.0355 (4)
H31	0.4450	0.4547	0.4702	0.043*
C32	0.39093 (14)	0.51947 (11)	0.30610 (19)	0.0386 (4)
H32	0.4052	0.5671	0.3482	0.046*
C33	0.34521 (14)	0.51431 (11)	0.17197 (19)	0.0380 (4)
H33	0.3273	0.5585	0.1200	0.046*
C34	0.32614 (13)	0.44366 (11)	0.11495 (18)	0.0368 (4)
H34	0.2935	0.4409	0.0235	0.044*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0589 (9)	0.0542 (9)	0.0279 (7)	0.0043 (7)	−0.0087 (6)	−0.0013 (6)
N1	0.0397 (9)	0.0449 (9)	0.0267 (8)	0.0005 (7)	−0.0026 (6)	−0.0014 (6)
C1	0.0361 (10)	0.0482 (11)	0.0266 (9)	0.0012 (8)	−0.0009 (7)	−0.0001 (7)
C2	0.0393 (10)	0.0440 (11)	0.0280 (9)	0.0010 (8)	−0.0005 (7)	0.0017 (7)
C3	0.0403 (11)	0.0461 (11)	0.0337 (10)	−0.0007 (9)	0.0005 (8)	0.0029 (8)
C4	0.0401 (11)	0.0479 (12)	0.0352 (10)	0.0009 (9)	0.0000 (8)	0.0059 (8)
C5	0.0391 (10)	0.0472 (12)	0.0352 (10)	0.0002 (9)	0.0012 (8)	0.0050 (8)
C6	0.0374 (10)	0.0442 (11)	0.0354 (10)	−0.0003 (8)	0.0037 (8)	0.0025 (8)
C7	0.0420 (11)	0.0485 (12)	0.0317 (9)	−0.0002 (9)	−0.0015 (8)	0.0006 (8)
C8	0.0400 (11)	0.0465 (11)	0.0350 (10)	0.0017 (9)	0.0010 (8)	0.0052 (8)
C9	0.0354 (10)	0.0480 (12)	0.0396 (10)	−0.0015 (9)	0.0060 (8)	−0.0007 (8)
C10	0.0486 (12)	0.0568 (13)	0.0318 (10)	−0.0047 (10)	0.0003 (8)	−0.0014 (9)
C11	0.0475 (12)	0.0549 (13)	0.0319 (10)	−0.0025 (10)	−0.0007 (8)	0.0056 (9)
C12	0.0462 (12)	0.0478 (12)	0.0528 (13)	−0.0038 (10)	0.0064 (10)	−0.0031 (10)
C13	0.0343 (9)	0.0441 (10)	0.0260 (9)	−0.0002 (8)	0.0015 (7)	−0.0005 (7)
C14	0.0398 (11)	0.0451 (11)	0.0349 (10)	0.0007 (9)	0.0005 (8)	−0.0049 (8)
C15	0.0483 (12)	0.0402 (11)	0.0445 (11)	0.0016 (9)	0.0048 (9)	−0.0016 (8)
C16	0.0470 (12)	0.0460 (11)	0.0352 (10)	0.0058 (9)	0.0049 (8)	0.0051 (8)
C17	0.0474 (11)	0.0478 (11)	0.0284 (9)	0.0054 (9)	−0.0029 (8)	0.0010 (8)

O2	0.0535 (8)	0.0405 (7)	0.0211 (6)	−0.0012 (6)	−0.0019 (5)	−0.0009 (5)
N2	0.0402 (9)	0.0382 (8)	0.0221 (7)	−0.0011 (7)	0.0007 (6)	−0.0018 (6)
C18	0.0361 (9)	0.0384 (10)	0.0226 (8)	−0.0007 (8)	0.0028 (7)	−0.0010 (7)
C19	0.0392 (10)	0.0357 (10)	0.0222 (8)	−0.0007 (8)	0.0008 (7)	−0.0007 (7)
C20	0.0379 (10)	0.0372 (10)	0.0253 (8)	−0.0006 (8)	0.0001 (7)	−0.0003 (7)
C21	0.0374 (10)	0.0366 (10)	0.0249 (8)	0.0011 (8)	−0.0013 (7)	0.0039 (7)
C22	0.0354 (9)	0.0396 (10)	0.0256 (8)	0.0016 (8)	0.0004 (7)	0.0037 (7)
C23	0.0361 (9)	0.0350 (9)	0.0260 (8)	0.0002 (8)	0.0026 (7)	−0.0001 (7)
C24	0.0367 (10)	0.0383 (10)	0.0251 (8)	0.0004 (8)	−0.0009 (7)	0.0008 (7)
C25	0.0392 (10)	0.0366 (10)	0.0306 (9)	0.0029 (8)	0.0009 (7)	0.0029 (7)
C26	0.0359 (10)	0.0377 (10)	0.0322 (9)	−0.0022 (8)	0.0051 (7)	−0.0033 (7)
C27	0.0395 (10)	0.0432 (11)	0.0258 (8)	−0.0019 (8)	0.0011 (7)	−0.0045 (7)
C28	0.0391 (10)	0.0397 (10)	0.0246 (8)	0.0006 (8)	−0.0008 (7)	0.0023 (7)
C29	0.0457 (11)	0.0401 (11)	0.0435 (11)	−0.0023 (9)	0.0056 (9)	−0.0064 (8)
C30	0.0348 (9)	0.0359 (10)	0.0215 (8)	−0.0012 (7)	0.0018 (7)	−0.0014 (6)
C31	0.0407 (10)	0.0375 (10)	0.0256 (8)	−0.0021 (8)	0.0000 (7)	−0.0031 (7)
C32	0.0458 (11)	0.0354 (10)	0.0327 (9)	−0.0022 (8)	0.0024 (8)	−0.0047 (7)
C33	0.0438 (11)	0.0376 (10)	0.0309 (9)	0.0010 (8)	0.0031 (8)	0.0049 (7)
C34	0.0436 (11)	0.0404 (10)	0.0239 (8)	0.0029 (8)	−0.0001 (7)	0.0003 (7)

Geometric parameters (Å, º)

O1—C1	1.226 (2)	O2—C18	1.228 (2)
N1—C13	1.346 (2)	N2—C30	1.345 (2)
N1—C17	1.337 (2)	N2—C34	1.339 (2)
C1—C2	1.474 (3)	C18—C19	1.476 (2)
C1—C13	1.498 (3)	C18—C30	1.498 (2)
C2—H2	0.9500	C19—H19	0.9500
C2—C3	1.335 (3)	C19—C20	1.336 (3)
C3—H3	0.9500	C20—H20	0.9500
C3—C4	1.411 (3)	C20—C21	1.420 (3)
C4—C5	1.206 (3)	C21—C22	1.203 (3)
C5—C6	1.426 (3)	C22—C23	1.430 (3)
C6—C7	1.397 (3)	C23—C24	1.400 (2)
C6—C11	1.401 (3)	C23—C28	1.402 (2)
C7—H7	0.9500	C24—H24	0.9500
C7—C8	1.388 (3)	C24—C25	1.385 (3)
C8—H8	0.9500	C25—H25	0.9500
C8—C9	1.390 (3)	C25—C26	1.396 (3)
C9—C10	1.397 (3)	C26—C27	1.396 (3)
C9—C12	1.504 (3)	C26—C29	1.507 (3)
C10—H10	0.9500	C27—H27	0.9500
C10—C11	1.374 (3)	C27—C28	1.380 (3)
C11—H11	0.9500	C28—H28	0.9500
C12—H12A	0.9800	C29—H29A	0.9800
C12—H12B	0.9800	C29—H29B	0.9800
C12—H12C	0.9800	C29—H29C	0.9800
C13—C14	1.380 (3)	C30—C31	1.385 (2)

C14—H14	0.9500	C31—H31	0.9500
C14—C15	1.390 (3)	C31—C32	1.384 (3)
C15—H15	0.9500	C32—H32	0.9500
C15—C16	1.385 (3)	C32—C33	1.385 (3)
C16—H16	0.9500	C33—H33	0.9500
C16—C17	1.379 (3)	C33—C34	1.384 (3)
C17—H17	0.9500	C34—H34	0.9500
C17—N1—C13	116.36 (17)	C34—N2—C30	116.40 (16)
O1—C1—C2	121.82 (18)	O2—C18—C19	121.85 (16)
O1—C1—C13	119.30 (19)	O2—C18—C30	119.62 (16)
C2—C1—C13	118.88 (15)	C19—C18—C30	118.53 (14)
C1—C2—H2	120.0	C18—C19—H19	120.5
C3—C2—C1	119.97 (17)	C20—C19—C18	118.97 (16)
C3—C2—H2	120.0	C20—C19—H19	120.5
C2—C3—H3	116.7	C19—C20—H20	116.8
C2—C3—C4	126.59 (19)	C19—C20—C21	126.41 (17)
C4—C3—H3	116.7	C21—C20—H20	116.8
C5—C4—C3	174.8 (2)	C22—C21—C20	173.42 (19)
C4—C5—C6	178.1 (2)	C21—C22—C23	177.61 (18)
C7—C6—C5	121.09 (18)	C24—C23—C22	119.68 (16)
C7—C6—C11	118.38 (19)	C24—C23—C28	118.69 (17)
C11—C6—C5	120.53 (18)	C28—C23—C22	121.63 (16)
C6—C7—H7	119.9	C23—C24—H24	119.8
C8—C7—C6	120.28 (18)	C25—C24—C23	120.40 (16)
C8—C7—H7	119.9	C25—C24—H24	119.8
C7—C8—H8	119.3	C24—C25—H25	119.5
C7—C8—C9	121.42 (18)	C24—C25—C26	121.03 (17)
C9—C8—H8	119.3	C26—C25—H25	119.5
C8—C9—C10	117.8 (2)	C25—C26—C27	118.29 (17)
C8—C9—C12	121.87 (19)	C25—C26—C29	120.02 (17)
C10—C9—C12	120.29 (19)	C27—C26—C29	121.68 (17)
C9—C10—H10	119.3	C26—C27—H27	119.4
C11—C10—C9	121.41 (19)	C28—C27—C26	121.22 (17)
C11—C10—H10	119.3	C28—C27—H27	119.4
C6—C11—H11	119.7	C23—C28—H28	119.8
C10—C11—C6	120.67 (19)	C27—C28—C23	120.36 (17)
C10—C11—H11	119.7	C27—C28—H28	119.8
C9—C12—H12A	109.5	C26—C29—H29A	109.5
C9—C12—H12B	109.5	C26—C29—H29B	109.5
C9—C12—H12C	109.5	C26—C29—H29C	109.5
H12A—C12—H12B	109.5	H29A—C29—H29B	109.5
H12A—C12—H12C	109.5	H29A—C29—H29C	109.5
H12B—C12—H12C	109.5	H29B—C29—H29C	109.5
N1—C13—C1	117.00 (17)	N2—C30—C18	116.97 (15)
N1—C13—C14	123.73 (18)	N2—C30—C31	123.55 (16)
C14—C13—C1	119.26 (16)	C31—C30—C18	119.47 (15)
C13—C14—H14	120.7	C30—C31—H31	120.5

C13—C14—C15	118.67 (18)	C32—C31—C30	119.03 (17)
C15—C14—H14	120.7	C32—C31—H31	120.5
C14—C15—H15	120.8	C31—C32—H32	120.9
C16—C15—C14	118.4 (2)	C31—C32—C33	118.19 (17)
C16—C15—H15	120.8	C33—C32—H32	120.9
C15—C16—H16	120.7	C32—C33—H33	120.6
C17—C16—C15	118.59 (19)	C34—C33—C32	118.83 (17)
C17—C16—H16	120.7	C34—C33—H33	120.6
N1—C17—C16	124.19 (18)	N2—C34—C33	123.94 (16)
N1—C17—H17	117.9	N2—C34—H34	118.0
C16—C17—H17	117.9	C33—C34—H34	118.0

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···N1	0.95	2.52	2.832 (3)	100
C16—H16···N2^{i}	0.95	2.66	3.555 (3)	158
C20—H20···N2^{i}	0.95	2.71	3.465 (3)	136
C3—H3···O1^{ii}	0.95	2.43	3.206 (3)	139
C19—H19···O2^{iii}	0.95	2.57	3.379 (2)	143
C25—H25···O2^{iv}	0.95	2.65	3.561 (2)	161

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