IUCrData

ISSN 2414-3146

9-(3,5-Dimethylbenzylidene)-8-(3,5-dimethylphenyl)-1,3,5,7-tetraphenyl-2,4,7,8-tetrahydro-4,7-methanoazulene acetonitrile disolvate

Sonya K. Adas, Andrew J. Peloquin, Scott T. Iacono and Gary J. Balaich*

Department of Chemistry & Chemistry Research Center, United States Air Force Academy, Colorado Springs, CO 80840, USA. *Correspondence e-mail: gary.balaich@usafa.edu

Received 23 March 2018
Accepted 9 April 2018

Edited by J. Simpson, University of Otago, New Zealand

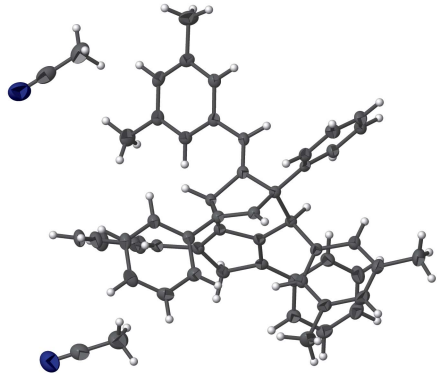
Keywords: cystal structure; azulene; benzylidene; crystal structure.

CCDC reference: 1835896

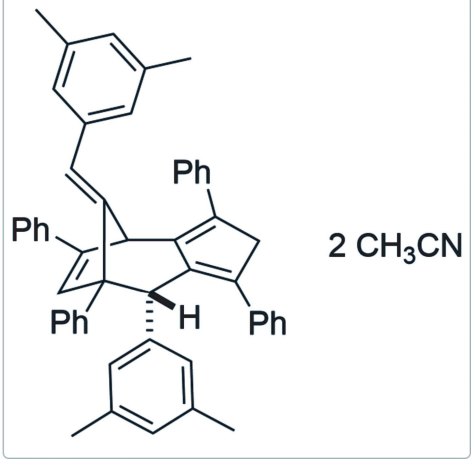
Structural data: full structural data are available from iucrdata.iucr.org

The title Diels–Alder product, $C_{52}H_{44}\cdot 2CH_3CN$, was obtained in trace quantity as the '*endo*' isomer during the synthesis of 1,3-diphenyl-6-(3,5-dimethylphenyl)fulvene. One of the two co-crystallized acetonitrile molecules is linked to the main molecule by a weak C—H···N hydrogen bond.

3D view



Chemical scheme



Structure description

The title compound is formed by Diels–Alder reaction of the fulvene component of one molecule of 1,3-diphenyl-6-(3,5-dimethylphenyl)fulvene and the exocyclic double bond of a second fulvene molecule, resulting in an '*endo*' product. The molecular structure is shown in Fig. 1. The bond lengths are typical of those observed in related fulvenes (Peloquin *et al.*, 2012). A weak C34—H34···N1 hydrogen bond (Table 1) links the main molecule to one of the two acetonitrile solvent molecules.

Synthesis and crystallization

To a vigorously stirred suspension of 1,3-diphenylcyclopentadiene (1.36 g, 6.21 mmol) in absolute EtOH (50 ml) under N_2 was added 3,5-dimethylbenzaldehyde (1.00 g, 7.45 mmol) and pyrrolidine (0.82 ml, 9.94 mmol), and the reaction mixture was maintained at room temperature for 8 h. The reaction mixture was cooled to −5°C for 12 h, the resulting precipitate isolated by vacuum filtration, and dried *in vacuo* to yield a red solid (0.215 g, 10%). The solvent was removed from the filtrate *in vacuo*. The resulting residue was dissolved in Et_2O (20 ml), washed with saturated $NaHSO_3$, dried over $MgSO_4$, and cooled to −5°C for 48 hrs. An additional crop of red solid material was isolated by vacuum filtration (0.339 g, total combined yield 0.554 g, 26%). Crystals

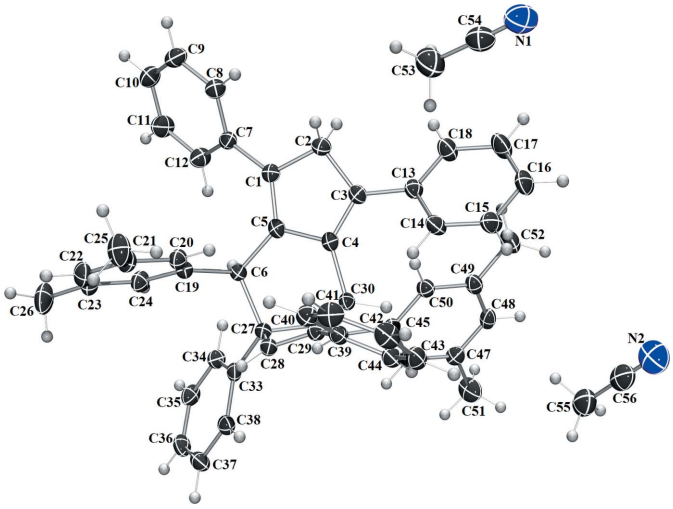


Figure 1
The asymmetric unit of of the title compound. Displacement ellipsoids are shown at the 50% probability level.

suitable for single-crystal X-ray diffraction were obtained by slow evaporation of an acetonitrile solution.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Funding information

Funding for this research was provided by: Defense Threat Reduction Agency (DTRA) - Joint Science and Technology Transfer Office for Chemical and Biological Defense (MIPR No. HDTRA13964) ; Air Force Office of Scientific Research (AFOSR) .

References

Bruker, (2017). *APEX2*, *SAINT*, and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.

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

D—H···A	D—H	H···A	D···A	D—H···A
C34—H34···N1^{i}	0.95	2.63	3.417 (3)	140

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{52}H_{44}{\cdot}2C_2H_3N$
M_r	750.98
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	100
a, b, c (Å)	10.4026 (3), 13.6115 (4), 16.5769 (6)
α, β, γ (°)	105.502 (2), 105.796 (2), 94.038 (2)
V (Å^3)	2150.30 (12)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.07
Crystal size (mm)	0.19 × 0.18 × 0.18
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2017)
T_{min}, T_{max}	0.804, 0.914
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	32078, 8992, 5894
R_{int}	0.059
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.634
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.053, 0.140, 1.02
No. of reflections	8992
No. of parameters	529
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.24

Computer programs: *APEX2* and *SAINT* (Bruker, 2017), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2016* (Sheldrick, 2015*b*), *ORTEP-3* for Windows (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008) and *OLEX2* (Dolomanov *et al.*, 2009).

Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Peloquin, A. J., Stone, R. S., Avila, S. E., Rudico, E. R., Horn, C. B., Gardner, K. A., Ball, D. W., Johnson, J. E. B., Iacono, S. T. & Balaich, G. J. (2012). *J. Org. Chem.* **77**, 6371–6376.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

full crystallographic data

IUCrData (2018). **3**, x180553 [https://doi.org/10.1107/S2414314618005539]

9-(3,5-Dimethylbenzylidene)-8-(3,5-dimethylphenyl)-1,3,5,7-tetraphenyl-2,4,7,8-tetrahydro-4,7-methanoazulene acetonitrile disolvate

Sonya K. Adas, Andrew J. Peloquin, Scott T. Iacono and Gary J. Balaich

9-(3,5-Dimethylbenzylidene)-8-(3,5-dimethylphenyl)-1,3,5,7-tetraphenyl-2,4,7,8-tetrahydro-4,7-methanoazulene acetonitrile disolvate

Crystal data

$C_{52}H_{44}·2C_2H_3N$
M_r = 750.98
Triclinic, $P\overline{1}$
a = 10.4026 (3) Å
b = 13.6115 (4) Å
c = 16.5769 (6) Å
α = 105.502 (2)°
β = 105.796 (2)°
γ = 94.038 (2)°
V = 2150.30 (12) Å^3
Z = 2
$F(000)$ = 800
D_x = 1.160 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3666 reflections
θ = 2.7–23.0°
μ = 0.07 mm^{-1}
T = 100 K
Prism, dark red
0.19 × 0.18 × 0.18 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2017)
T_{min} = 0.804, T_{max} = 0.914
32078 measured reflections
8992 independent reflections
5894 reflections with $I > 2\sigma(I)$
R_{int} = 0.059
θ_{max} = 26.8°, θ_{min} = 1.7°
h = −13→13
k = −17→17
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.140
S = 1.02
8992 reflections
529 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0524P)^2 + 0.8994P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 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}
C1	0.62899 (18)	0.47941 (15)	0.14895 (12)	0.0208 (4)
C2	0.6900 (2)	0.59098 (15)	0.17214 (13)	0.0233 (4)
H2A	0.643654	0.622662	0.127142	0.028*
H2B	0.787475	0.597427	0.177324	0.028*
C3	0.66858 (18)	0.64125 (15)	0.25942 (13)	0.0216 (4)
C4	0.60350 (18)	0.56787 (14)	0.28198 (12)	0.0195 (4)
C5	0.57835 (18)	0.46750 (14)	0.21387 (12)	0.0203 (4)
C6	0.51239 (18)	0.36835 (14)	0.22266 (12)	0.0194 (4)
H6	0.580932	0.320384	0.224780	0.023*
C7	0.63983 (19)	0.40180 (15)	0.07072 (12)	0.0221 (4)
C8	0.6316 (2)	0.42727 (16)	−0.00655 (13)	0.0262 (5)
H8	0.617660	0.494949	−0.008448	0.031*
C9	0.6433 (2)	0.35588 (17)	−0.08068 (14)	0.0318 (5)
H9	0.634451	0.374417	−0.133041	0.038*
C10	0.6677 (2)	0.25814 (17)	−0.07852 (14)	0.0351 (5)
H10	0.677284	0.209527	−0.128810	0.042*
C11	0.6780 (2)	0.23184 (17)	−0.00221 (15)	0.0377 (6)
H11	0.695009	0.164728	−0.000227	0.045*
C12	0.6638 (2)	0.30217 (16)	0.07139 (14)	0.0293 (5)
H12	0.670427	0.282428	0.123007	0.035*
C13	0.71457 (19)	0.75077 (15)	0.30998 (13)	0.0228 (4)
C14	0.8398 (2)	0.80224 (16)	0.31707 (15)	0.0315 (5)
H14	0.896653	0.766764	0.287011	0.038*
C15	0.8825 (2)	0.90480 (17)	0.36757 (16)	0.0374 (6)
H15	0.967772	0.938845	0.371234	0.045*
C16	0.8021 (2)	0.95767 (16)	0.41248 (15)	0.0335 (5)
H16	0.832662	1.027197	0.448174	0.040*
C17	0.6769 (2)	0.90832 (16)	0.40489 (14)	0.0311 (5)
H17	0.620503	0.944311	0.434985	0.037*
C18	0.6332 (2)	0.80677 (15)	0.35372 (14)	0.0270 (5)
H18	0.546002	0.774372	0.348177	0.032*
C19	0.38835 (19)	0.31274 (15)	0.14540 (12)	0.0216 (4)
C20	0.29397 (19)	0.36707 (15)	0.10607 (12)	0.0238 (4)
H20	0.311035	0.440359	0.124881	0.029*
C21	0.1756 (2)	0.31651 (16)	0.04003 (13)	0.0279 (5)
C22	0.1519 (2)	0.20943 (17)	0.01294 (14)	0.0332 (5)
H22	0.070889	0.174148	−0.031758	0.040*
C23	0.2445 (2)	0.15275 (16)	0.05000 (14)	0.0312 (5)
C24	0.3626 (2)	0.20562 (15)	0.11579 (13)	0.0269 (5)
H24	0.426966	0.167545	0.140923	0.032*
C25	0.0735 (2)	0.37751 (18)	0.00006 (16)	0.0411 (6)
H25A	0.093454	0.449349	0.037407	0.062*
H25B	−0.017614	0.347532	−0.004506	0.062*
H25C	0.078555	0.375134	−0.058587	0.062*
C26	0.2178 (3)	0.03637 (17)	0.02230 (16)	0.0471 (7)

H26A	0.199817	0.013230	0.069850	0.071*
H26B	0.297117	0.009100	0.009420	0.071*
H26C	0.139204	0.011188	−0.030373	0.071*
C27	0.47673 (18)	0.39042 (14)	0.31296 (12)	0.0198 (4)
C28	0.36308 (19)	0.45486 (14)	0.30875 (12)	0.0204 (4)
H28	0.269753	0.426725	0.287361	0.024*
C29	0.40990 (19)	0.55538 (15)	0.33861 (12)	0.0208 (4)
C30	0.56498 (18)	0.57118 (14)	0.36412 (12)	0.0194 (4)
H30	0.610396	0.633564	0.415039	0.023*
C31	0.59450 (18)	0.46958 (14)	0.38120 (12)	0.0193 (4)
C32	0.69876 (18)	0.45072 (15)	0.43931 (12)	0.0204 (4)
H32	0.697236	0.380820	0.439198	0.025*
C33	0.44909 (19)	0.29195 (14)	0.33668 (12)	0.0209 (4)
C34	0.5261 (2)	0.21256 (15)	0.32410 (13)	0.0249 (4)
H34	0.598925	0.220035	0.300825	0.030*
C35	0.4982 (2)	0.12266 (16)	0.34500 (13)	0.0290 (5)
H35	0.550963	0.069071	0.335040	0.035*
C36	0.3940 (2)	0.11082 (16)	0.38019 (14)	0.0306 (5)
H36	0.373663	0.048802	0.393312	0.037*
C37	0.3198 (2)	0.19009 (16)	0.39607 (14)	0.0292 (5)
H37	0.249673	0.183301	0.421778	0.035*
C38	0.34690 (19)	0.27988 (15)	0.37467 (13)	0.0238 (4)
H38	0.295060	0.333805	0.386105	0.029*
C39	0.33417 (19)	0.64335 (15)	0.33960 (13)	0.0216 (4)
C40	0.2347 (2)	0.64701 (16)	0.26484 (13)	0.0265 (5)
H40	0.209202	0.589441	0.213367	0.032*
C41	0.1732 (2)	0.73391 (17)	0.26525 (15)	0.0318 (5)
H41	0.107113	0.736006	0.213577	0.038*
C42	0.2068 (2)	0.81772 (17)	0.33996 (15)	0.0332 (5)
H42	0.165791	0.877817	0.339330	0.040*
C43	0.3008 (2)	0.81329 (16)	0.41577 (14)	0.0287 (5)
H43	0.321562	0.869367	0.468090	0.034*
C44	0.3647 (2)	0.72731 (15)	0.41550 (13)	0.0243 (4)
H44	0.429979	0.725404	0.467562	0.029*
C45	0.81598 (18)	0.52355 (14)	0.50365 (12)	0.0209 (4)
C46	0.87823 (19)	0.50031 (15)	0.58076 (13)	0.0236 (4)
H46	0.845059	0.437801	0.588632	0.028*
C47	0.98684 (19)	0.56552 (16)	0.64614 (13)	0.0262 (5)
C48	1.03670 (19)	0.65565 (16)	0.63328 (13)	0.0259 (5)
H48	1.110829	0.701187	0.677859	0.031*
C49	0.98033 (19)	0.68044 (15)	0.55664 (13)	0.0256 (5)
C50	0.87020 (19)	0.61401 (15)	0.49205 (13)	0.0236 (4)
H50	0.831347	0.630440	0.439310	0.028*
C51	1.0514 (2)	0.5376 (2)	0.72843 (15)	0.0426 (6)
H51A	0.997946	0.475463	0.728623	0.064*
H51B	1.054786	0.594710	0.779978	0.064*
H51C	1.143378	0.524448	0.730204	0.064*
C52	1.0402 (2)	0.77656 (17)	0.54269 (16)	0.0389 (6)

H52A	1.090664	0.825899	0.599695	0.058*
H52B	0.967505	0.807697	0.512110	0.058*
H52C	1.101452	0.758471	0.507218	0.058*
C53	0.6664 (3)	0.9128 (2)	0.15696 (19)	0.0587 (8)
H53A	0.627272	0.895627	0.199880	0.088*
H53B	0.752146	0.885960	0.160801	0.088*
H53C	0.603605	0.881801	0.097701	0.088*
C54	0.6906 (2)	1.0242 (2)	0.17549 (16)	0.0403 (6)
C55	0.9502 (3)	0.8449 (2)	0.80174 (18)	0.0521 (7)
H55A	1.026712	0.842034	0.850591	0.078*
H55B	0.954771	0.797311	0.746906	0.078*
H55C	0.865336	0.824845	0.812274	0.078*
C56	0.9560 (2)	0.9494 (2)	0.79496 (16)	0.0466 (7)
N1	0.7109 (2)	1.11149 (19)	0.18894 (16)	0.0535 (6)
N2	0.9628 (2)	1.0317 (2)	0.79074 (16)	0.0636 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0183 (10)	0.0239 (11)	0.0204 (10)	0.0052 (8)	0.0035 (8)	0.0088 (8)
C2	0.0233 (10)	0.0245 (11)	0.0243 (10)	0.0039 (8)	0.0076 (8)	0.0103 (9)
C3	0.0178 (10)	0.0224 (11)	0.0247 (10)	0.0034 (8)	0.0055 (8)	0.0083 (8)
C4	0.0154 (9)	0.0203 (10)	0.0219 (10)	0.0039 (8)	0.0035 (8)	0.0066 (8)
C5	0.0164 (9)	0.0206 (10)	0.0230 (10)	0.0034 (8)	0.0030 (8)	0.0077 (8)
C6	0.0185 (10)	0.0195 (10)	0.0205 (10)	0.0037 (8)	0.0062 (8)	0.0063 (8)
C7	0.0188 (10)	0.0255 (11)	0.0218 (10)	0.0023 (8)	0.0073 (8)	0.0058 (8)
C8	0.0267 (11)	0.0283 (12)	0.0257 (11)	0.0061 (9)	0.0100 (9)	0.0089 (9)
C9	0.0337 (12)	0.0395 (14)	0.0243 (11)	0.0066 (10)	0.0115 (9)	0.0096 (10)
C10	0.0426 (14)	0.0324 (13)	0.0294 (12)	0.0035 (11)	0.0181 (10)	0.0008 (10)
C11	0.0533 (15)	0.0248 (12)	0.0415 (14)	0.0106 (11)	0.0251 (12)	0.0083 (10)
C12	0.0354 (12)	0.0283 (12)	0.0280 (11)	0.0091 (10)	0.0128 (9)	0.0102 (9)
C13	0.0248 (10)	0.0198 (11)	0.0245 (10)	0.0033 (8)	0.0071 (8)	0.0079 (8)
C14	0.0271 (11)	0.0241 (12)	0.0438 (13)	0.0026 (9)	0.0157 (10)	0.0063 (10)
C15	0.0284 (12)	0.0291 (13)	0.0522 (15)	−0.0043 (10)	0.0158 (11)	0.0064 (11)
C16	0.0401 (13)	0.0189 (11)	0.0397 (13)	0.0001 (10)	0.0133 (10)	0.0054 (9)
C17	0.0402 (13)	0.0217 (12)	0.0371 (12)	0.0065 (10)	0.0203 (10)	0.0090 (10)
C18	0.0274 (11)	0.0221 (11)	0.0353 (12)	0.0023 (9)	0.0141 (9)	0.0104 (9)
C19	0.0223 (10)	0.0219 (11)	0.0200 (10)	0.0010 (8)	0.0073 (8)	0.0050 (8)
C20	0.0267 (11)	0.0206 (11)	0.0224 (10)	0.0029 (8)	0.0069 (8)	0.0044 (8)
C21	0.0251 (11)	0.0309 (12)	0.0241 (11)	0.0017 (9)	0.0029 (9)	0.0078 (9)
C22	0.0301 (12)	0.0338 (13)	0.0251 (11)	−0.0050 (10)	−0.0011 (9)	0.0037 (10)
C23	0.0379 (13)	0.0245 (12)	0.0253 (11)	−0.0016 (10)	0.0064 (9)	0.0026 (9)
C24	0.0324 (12)	0.0219 (11)	0.0244 (11)	0.0053 (9)	0.0077 (9)	0.0047 (9)
C25	0.0334 (13)	0.0383 (14)	0.0409 (14)	0.0052 (11)	−0.0047 (11)	0.0105 (11)
C26	0.0613 (17)	0.0261 (13)	0.0385 (14)	−0.0019 (12)	0.0006 (12)	0.0012 (11)
C27	0.0194 (10)	0.0199 (10)	0.0199 (10)	0.0023 (8)	0.0066 (8)	0.0052 (8)
C28	0.0188 (10)	0.0216 (11)	0.0203 (10)	0.0020 (8)	0.0070 (8)	0.0047 (8)
C29	0.0193 (10)	0.0227 (11)	0.0206 (10)	0.0032 (8)	0.0068 (8)	0.0063 (8)

C30	0.0199 (10)	0.0169 (10)	0.0211 (10)	0.0021 (8)	0.0070 (8)	0.0047 (8)
C31	0.0199 (10)	0.0199 (10)	0.0204 (10)	0.0025 (8)	0.0110 (8)	0.0050 (8)
C32	0.0211 (10)	0.0171 (10)	0.0232 (10)	0.0027 (8)	0.0084 (8)	0.0047 (8)
C33	0.0219 (10)	0.0181 (10)	0.0185 (9)	−0.0012 (8)	0.0025 (8)	0.0032 (8)
C34	0.0281 (11)	0.0217 (11)	0.0256 (11)	0.0039 (9)	0.0093 (9)	0.0069 (9)
C35	0.0395 (13)	0.0215 (11)	0.0257 (11)	0.0081 (9)	0.0090 (9)	0.0068 (9)
C36	0.0370 (13)	0.0216 (11)	0.0296 (12)	−0.0030 (10)	0.0047 (10)	0.0091 (9)
C37	0.0262 (11)	0.0305 (12)	0.0312 (12)	−0.0020 (9)	0.0078 (9)	0.0119 (10)
C38	0.0213 (10)	0.0232 (11)	0.0248 (10)	0.0018 (8)	0.0058 (8)	0.0056 (9)
C39	0.0192 (10)	0.0205 (10)	0.0273 (11)	0.0015 (8)	0.0105 (8)	0.0076 (8)
C40	0.0242 (11)	0.0287 (12)	0.0267 (11)	0.0055 (9)	0.0087 (9)	0.0072 (9)
C41	0.0250 (11)	0.0403 (14)	0.0340 (12)	0.0123 (10)	0.0074 (9)	0.0171 (11)
C42	0.0304 (12)	0.0266 (12)	0.0488 (14)	0.0121 (10)	0.0171 (11)	0.0142 (11)
C43	0.0288 (11)	0.0218 (11)	0.0356 (12)	0.0038 (9)	0.0149 (10)	0.0036 (9)
C44	0.0240 (10)	0.0219 (11)	0.0279 (11)	0.0038 (9)	0.0102 (9)	0.0065 (9)
C45	0.0188 (10)	0.0210 (11)	0.0230 (10)	0.0038 (8)	0.0089 (8)	0.0039 (8)
C46	0.0205 (10)	0.0231 (11)	0.0280 (11)	0.0037 (8)	0.0087 (8)	0.0071 (9)
C47	0.0194 (10)	0.0327 (12)	0.0257 (11)	0.0064 (9)	0.0061 (8)	0.0074 (9)
C48	0.0167 (10)	0.0274 (12)	0.0270 (11)	0.0012 (8)	0.0054 (8)	−0.0008 (9)
C49	0.0205 (10)	0.0233 (11)	0.0315 (11)	0.0019 (8)	0.0094 (9)	0.0048 (9)
C50	0.0213 (10)	0.0248 (11)	0.0247 (10)	0.0029 (8)	0.0071 (8)	0.0074 (9)
C51	0.0296 (13)	0.0536 (16)	0.0386 (14)	−0.0004 (11)	−0.0031 (10)	0.0195 (12)
C52	0.0347 (13)	0.0340 (13)	0.0420 (14)	−0.0084 (10)	0.0069 (10)	0.0099 (11)
C53	0.070 (2)	0.0522 (18)	0.0521 (17)	−0.0051 (15)	0.0122 (15)	0.0229 (14)
C54	0.0357 (13)	0.0501 (17)	0.0417 (14)	0.0102 (12)	0.0161 (11)	0.0192 (13)
C55	0.0455 (16)	0.0554 (18)	0.0453 (16)	−0.0020 (13)	0.0127 (12)	0.0019 (13)
C56	0.0338 (14)	0.061 (2)	0.0344 (14)	−0.0010 (13)	0.0034 (11)	0.0066 (13)
N1	0.0609 (15)	0.0514 (15)	0.0639 (15)	0.0208 (12)	0.0363 (12)	0.0217 (12)
N2	0.0529 (15)	0.0726 (19)	0.0565 (16)	0.0047 (14)	0.0003 (12)	0.0230 (14)

Geometric parameters (Å, º)

C1—C2	1.509 (3)	C28—C29	1.329 (3)
C1—C5	1.361 (3)	C29—C30	1.536 (3)
C1—C7	1.475 (3)	C29—C39	1.478 (3)
C2—H2A	0.9900	C30—H30	1.0000
C2—H2B	0.9900	C30—C31	1.519 (2)
C2—C3	1.509 (3)	C31—C32	1.331 (3)
C3—C4	1.356 (2)	C32—H32	0.9500
C3—C13	1.472 (3)	C32—C45	1.471 (3)
C4—C5	1.474 (3)	C33—C34	1.395 (3)
C4—C30	1.511 (3)	C33—C38	1.396 (3)
C5—C6	1.531 (2)	C34—H34	0.9500
C6—H6	1.0000	C34—C35	1.390 (3)
C6—C19	1.524 (3)	C35—H35	0.9500
C6—C27	1.596 (3)	C35—C36	1.382 (3)
C7—C8	1.396 (3)	C36—H36	0.9500
C7—C12	1.398 (3)	C36—C37	1.380 (3)

C8—H8	0.9500	C37—H37	0.9500
C8—C9	1.389 (3)	C37—C38	1.392 (3)
C9—H9	0.9500	C38—H38	0.9500
C9—C10	1.380 (3)	C39—C40	1.397 (3)
C10—H10	0.9500	C39—C44	1.397 (3)
C10—C11	1.383 (3)	C40—H40	0.9500
C11—H11	0.9500	C40—C41	1.383 (3)
C11—C12	1.386 (3)	C41—H41	0.9500
C12—H12	0.9500	C41—C42	1.382 (3)
C13—C14	1.395 (3)	C42—H42	0.9500
C13—C18	1.397 (3)	C42—C43	1.385 (3)
C14—H14	0.9500	C43—H43	0.9500
C14—C15	1.390 (3)	C43—C44	1.385 (3)
C15—H15	0.9500	C44—H44	0.9500
C15—C16	1.382 (3)	C45—C46	1.396 (3)
C16—H16	0.9500	C45—C50	1.400 (3)
C16—C17	1.381 (3)	C46—H46	0.9500
C17—H17	0.9500	C46—C47	1.384 (3)
C17—C18	1.383 (3)	C47—C48	1.389 (3)
C18—H18	0.9500	C47—C51	1.507 (3)
C19—C20	1.395 (3)	C48—H48	0.9500
C19—C24	1.389 (3)	C48—C49	1.387 (3)
C20—H20	0.9500	C49—C50	1.395 (3)
C20—C21	1.390 (3)	C49—C52	1.509 (3)
C21—C22	1.388 (3)	C50—H50	0.9500
C21—C25	1.514 (3)	C51—H51A	0.9800
C22—H22	0.9500	C51—H51B	0.9800
C22—C23	1.392 (3)	C51—H51C	0.9800
C23—C24	1.394 (3)	C52—H52A	0.9800
C23—C26	1.509 (3)	C52—H52B	0.9800
C24—H24	0.9500	C52—H52C	0.9800
C25—H25A	0.9800	C53—H53A	0.9800
C25—H25B	0.9800	C53—H53B	0.9800
C25—H25C	0.9800	C53—H53C	0.9800
C26—H26A	0.9800	C53—C54	1.452 (4)
C26—H26B	0.9800	C54—N1	1.142 (3)
C26—H26C	0.9800	C55—H55A	0.9800
C27—C28	1.519 (3)	C55—H55B	0.9800
C27—C31	1.533 (3)	C55—H55C	0.9800
C27—C33	1.524 (2)	C55—C56	1.455 (4)
C28—H28	0.9500	C56—N2	1.140 (3)

C5—C1—C2	108.57 (17)	C29—C28—C27	111.86 (16)
C5—C1—C7	130.12 (18)	C29—C28—H28	124.1
C7—C1—C2	121.10 (16)	C28—C29—C30	109.16 (17)
C1—C2—H2A	110.9	C28—C29—C39	129.03 (17)
C1—C2—H2B	110.9	C39—C29—C30	121.42 (16)
C1—C2—C3	104.33 (15)	C4—C30—C29	107.43 (15)

H2A—C2—H2B	108.9	C4—C30—H30	114.2
C3—C2—H2A	110.9	C4—C30—C31	104.56 (15)
C3—C2—H2B	110.9	C29—C30—H30	114.2
C4—C3—C2	107.99 (17)	C31—C30—C29	101.13 (14)
C4—C3—C13	127.47 (18)	C31—C30—H30	114.2
C13—C3—C2	124.52 (16)	C30—C31—C27	103.66 (15)
C3—C4—C5	110.18 (17)	C32—C31—C27	126.94 (17)
C3—C4—C30	131.60 (18)	C32—C31—C30	129.37 (17)
C5—C4—C30	118.04 (16)	C31—C32—H32	115.6
C1—C5—C4	108.93 (16)	C31—C32—C45	128.72 (18)
C1—C5—C6	128.46 (18)	C45—C32—H32	115.6
C4—C5—C6	122.53 (16)	C34—C33—C27	122.01 (17)
C5—C6—H6	107.1	C34—C33—C38	117.59 (17)
C5—C6—C27	110.84 (15)	C38—C33—C27	120.38 (18)
C19—C6—C5	113.78 (15)	C33—C34—H34	119.4
C19—C6—H6	107.1	C35—C34—C33	121.18 (19)
C19—C6—C27	110.62 (15)	C35—C34—H34	119.4
C27—C6—H6	107.1	C34—C35—H35	119.8
C8—C7—C1	120.45 (18)	C36—C35—C34	120.4 (2)
C8—C7—C12	117.38 (18)	C36—C35—H35	119.8
C12—C7—C1	122.12 (17)	C35—C36—H36	120.4
C7—C8—H8	119.3	C37—C36—C35	119.28 (19)
C9—C8—C7	121.49 (19)	C37—C36—H36	120.4
C9—C8—H8	119.3	C36—C37—H37	119.8
C8—C9—H9	119.9	C36—C37—C38	120.5 (2)
C10—C9—C8	120.2 (2)	C38—C37—H37	119.8
C10—C9—H9	119.9	C33—C38—H38	119.5
C9—C10—H10	120.4	C37—C38—C33	121.0 (2)
C9—C10—C11	119.2 (2)	C37—C38—H38	119.5
C11—C10—H10	120.4	C40—C39—C29	121.53 (18)
C10—C11—H11	119.6	C40—C39—C44	118.30 (18)
C10—C11—C12	120.8 (2)	C44—C39—C29	120.13 (17)
C12—C11—H11	119.6	C39—C40—H40	119.8
C7—C12—H12	119.5	C41—C40—C39	120.4 (2)
C11—C12—C7	120.91 (19)	C41—C40—H40	119.8
C11—C12—H12	119.5	C40—C41—H41	119.6
C14—C13—C3	122.04 (18)	C42—C41—C40	120.8 (2)
C14—C13—C18	117.55 (18)	C42—C41—H41	119.6
C18—C13—C3	120.41 (17)	C41—C42—H42	120.3
C13—C14—H14	119.6	C41—C42—C43	119.4 (2)
C15—C14—C13	120.8 (2)	C43—C42—H42	120.3
C15—C14—H14	119.6	C42—C43—H43	119.9
C14—C15—H15	119.7	C42—C43—C44	120.2 (2)
C16—C15—C14	120.6 (2)	C44—C43—H43	119.9
C16—C15—H15	119.7	C39—C44—H44	119.6
C15—C16—H16	120.4	C43—C44—C39	120.82 (19)
C17—C16—C15	119.2 (2)	C43—C44—H44	119.6
C17—C16—H16	120.4	C46—C45—C32	117.90 (17)

C16—C17—H17	119.8	C46—C45—C50	117.86 (17)
C16—C17—C18	120.3 (2)	C50—C45—C32	124.23 (17)
C18—C17—H17	119.8	C45—C46—H46	119.0
C13—C18—H18	119.3	C47—C46—C45	122.04 (18)
C17—C18—C13	121.41 (19)	C47—C46—H46	119.0
C17—C18—H18	119.3	C46—C47—C48	118.63 (18)
C20—C19—C6	121.36 (17)	C46—C47—C51	120.43 (19)
C24—C19—C6	120.25 (17)	C48—C47—C51	120.92 (18)
C24—C19—C20	118.28 (18)	C47—C48—H48	119.4
C19—C20—H20	119.2	C49—C48—C47	121.27 (18)
C21—C20—C19	121.58 (18)	C49—C48—H48	119.4
C21—C20—H20	119.2	C48—C49—C50	119.05 (18)
C20—C21—C25	120.40 (19)	C48—C49—C52	120.19 (18)
C22—C21—C20	118.70 (19)	C50—C49—C52	120.74 (18)
C22—C21—C25	120.89 (18)	C45—C50—H50	119.5
C21—C22—H22	119.4	C49—C50—C45	121.09 (18)
C21—C22—C23	121.27 (19)	C49—C50—H50	119.5
C23—C22—H22	119.4	C47—C51—H51A	109.5
C22—C23—C24	118.70 (19)	C47—C51—H51B	109.5
C22—C23—C26	121.51 (19)	C47—C51—H51C	109.5
C24—C23—C26	119.8 (2)	H51A—C51—H51B	109.5
C19—C24—C23	121.45 (19)	H51A—C51—H51C	109.5
C19—C24—H24	119.3	H51B—C51—H51C	109.5
C23—C24—H24	119.3	C49—C52—H52A	109.5
C21—C25—H25A	109.5	C49—C52—H52B	109.5
C21—C25—H25B	109.5	C49—C52—H52C	109.5
C21—C25—H25C	109.5	H52A—C52—H52B	109.5
H25A—C25—H25B	109.5	H52A—C52—H52C	109.5
H25A—C25—H25C	109.5	H52B—C52—H52C	109.5
H25B—C25—H25C	109.5	H53A—C53—H53B	109.5
C23—C26—H26A	109.5	H53A—C53—H53C	109.5
C23—C26—H26B	109.5	H53B—C53—H53C	109.5
C23—C26—H26C	109.5	C54—C53—H53A	109.5
H26A—C26—H26B	109.5	C54—C53—H53B	109.5
H26A—C26—H26C	109.5	C54—C53—H53C	109.5
H26B—C26—H26C	109.5	N1—C54—C53	178.7 (3)
C28—C27—C6	108.65 (14)	H55A—C55—H55B	109.5
C28—C27—C31	100.54 (15)	H55A—C55—H55C	109.5
C28—C27—C33	114.32 (15)	H55B—C55—H55C	109.5
C31—C27—C6	105.60 (14)	C56—C55—H55A	109.5
C33—C27—C6	112.36 (15)	C56—C55—H55B	109.5
C33—C27—C31	114.40 (15)	C56—C55—H55C	109.5
C27—C28—H28	124.1	N2—C56—C55	178.8 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

C34—H34···N1^{i}	0.95	2.63	3.417 (3)	140

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